DOI: https://doi.org/10.4038/ta.v167i4.98 *Tropical Agriculturist, Vol.167 (4), 2019*

Occurrence of pesticide residues in imported and locally-grown crops as analyzed by gas chromatography mass spectrometry

P.W.Y. Lakshani, J.A. Sumith, M.K.L.K. Rajapaksha, W.P.A.P.Dhanapala and W.R.W.Wimalasena

Office of the Registrar of Pesticide, Kandy Road, Getambe, Sri Lanka

Abstract

Food safety is gaining prominence as a global issue. Consumers are frequently notified of health concerns associated with imported foods as well as specific fruits and vegetables. Pesticide residues in 111 random samples, including 6 different crops as 28 samples of potatoes, 22 samples of oranges, 34 samples of onions, 13 samples of chilies, 12 samples of apples and 3 samples of grapes from selected growing areas and imported consignments at Orugodawatta, Sri Lanka were evaluated against 12 pesticides Samples were extracted according to the quick, easy, cheap, efficient, rugged, safe (QuEChERS) method as per Association of Official Analytical Chemist (AOAC) official method 2007.01. Concentrated samples were achieved by Gas Chromatography Mass Spectrometry (GC-MS) in Selective Ion Mode (SIM). Recovery studies at three spiking concentration levels as 1 Limit of Quantification (LOQ), 5 LOQ and 10 LOQ, varied from 70 % to 110 %, with a Relative Standard Deviation (RSD) below 20%. Among the imported samples that were positive for pesticide residues, Diazinon, Chlorphyrifos, Fipronil, Oxyfluorfen, Tebuconazole were detected in 5.56%, 1.85%, 14.81%, 16.66 %, 12.96 % samples, respectively and among the local samples that were positive for pesticide residues, Pendimethalin, Pretilachlor, Bifenthrin, , Chlorphyrifos, Fipronil, , Oxyfluorfen, Tebuconazole were detected in 2.77%, 2.77%, 22.22%, 1.75%, 12.28%, 17.54%, 7.01% respectively. The results revealed the presence of pesticide residues in both imported and local samples between the range of 0.044 ppm to 6.57 ppm. Continuous monitoring of pesticide residue in imported crops and those grown in Sri Lanka need to be conducted to obtain sufficient evidence to make conclusive policy decisions to ensure consumer safety.

Key words: Leafy Vegetables, LOQ, Pesticide residues, QuEChERS

Introduction

During the past two decades international trade in fruits and vegetables (F&V) has increased rapidly than other agricultural commodities. So far, trade in F&V has well globalized and a large volume of F&V is being traded around the world Perera *et al*, 2015. Sri Lanka had imported 3,441 mt of fresh fruits during the month of April 2018 worth Rs. 640 Mn. Of the imported quantity, 77% were apples, 13% were grapes and 10% were oranges. The main importing countries of apple were China, South Africa, New Zealand and the United States. Grapes had been imported from Australia, India and South Africa while oranges were imported from Egypt. During the month of April 2018, a quantity of 26,044 mt of big onions was imported from India (99%) and Pakistan (1%) while a quantity of 11,645 mt of potatoes had been imported from Pakistan and India (HARTI Food Information bulletin, 2018). Sri Lanka produces around 710,000 metric tons of vegetables and around 540,000 metric tons of fruits, annually (Esham., 2006).

Pesticide contamination is a worldwide public health concern and one of the crucial issues in international trade. Pesticide residues are specified as substances in food, agricultural commodities or animal feed resulting from the use of pesticides (Chandra *et al*., 2014). They include any derivative of a pesticide, such as conversion products, metabolites, reaction products and impurities of toxicological significance (Denis *et al*., 2004). This study reported analysis of the residues of twelve pesticides in six commodities including potato, oranges, onion, apple, grapes and dry chillies in selected growing areas and import consignments in Sri Lanka aiming at implementation of continuous monitoring programs ensuring access of high quality food stuff to consumers.

Materials and methods

Selection of samples

Details of the crops selected areas in Sri Lanka and the sample size are shown in the Table 1 and the details of the crops and the imported countries and the sample size for imported crops are shown in Table 2.

Table 1. Selected study areas for sampling

Crop	District	Area	Total No. of Sample
Potato	Nuwara Eliya	Nuwara Eliya	16
Oranges	Ampara	Bibile	20
Onion	Matale	Dambulla	21

Table 2. Details of the tested important crops and respective country

Crop	Import Country	No. of Sample	Total No. of Sample
Potato	India	4	-
	China	3	11
	Pakistan	4	-
Oranges	South Africa	1	2
	Australia	1	-
Onion	India	12	-
	Bangladesh	1	13
Dry Chilie	India	12	13
	Pakistan	1	-
Apple	China	10	12
	France	2	-
Grapes	China	1	3
	USA	2	-

Sampling procedure adopted to sample F&V (Figure 1) was mainly based on the recommendation by the Codex Alimentarius Commission of the Food and Agriculture Organization (FAO) of United Nations (CODEX, 2017). Field Samples and samples from consignments were collected according to the "Recommended Methods of Sampling For the Determination of Pesticide Residues for Compliance With MRL's CAC/Gl 33-1999" issued by FAO CODEX (Cac/Gl,1999). The crops were then divided into four parts and each part was mixed well. The fruits from each part were divided into two parts and one part was kept at the refrigerator at 4-8 °C (AOAC, 2011) and the other was used for the analysis. While harvesting, handling, packaging, transportation and preparation, necessary precautions were taken. The samples were stored in large zip lock polyethylene bags or foil-lined paper bags at 10 °C. Each sample was properly labelled (ISO 874:1980).

Selection of pesticides

Eleven pesticides were selected for the analysis due to their world-wide usage, known toxicological effects and the availability of analytical facilities. Details of the pesticides selected for the study is shown in Table 3.

Table 3. Details of pesticides selected for the study

Pesticide	CAS No	Chemical type	Main use	ADI (mg/kg body weight)	AI Hazard Class	AI LD50 (rat oral, mg/kg body weight)
Pirimiphos-methyl	29232-93-7	Organophosphate	I	0.03	IV	1,667
Dicofol	115-32-2	Organochlorine	AC	0.002	IV	675
Pendimethalin	40487-42-1	Dinitroaniline	H	0.125	IV	1,050
Quinalphos	13593-03-8	Organophosphate	I	0.01	III	62
Pretilachlor	51218-49-6	Pyrethroids	H	0.018	V	6,100
Bifenthrin	82657-04-3	Pyrethroid	I	0.02	III	55
Diazinon	333-41-5	Organophosphate	I	0.005	II	300
Chlorpyrifos	2921-88-2	Organophosphate	I	0.0002	II	135
Prothiofos	34643-46-4	Organophosphate	I	0.005	II	925
Oxyfluorfen	42874-03-3	Diphenyl ether	H	0.01	IV	>5,000
Tebuconazole	107534-98-3	Triazole	F	0.03	II	1,700

CAS: Chemical Abstract No., AC: Acaricide, ADI: Acceptable Daily Intake, F: Fungicide, I: Insecticide, AI: Active Ingredient; LD50: Lethal Dosage; Hazard Class (*WHO,* 2009)

Certified reference materials (CRM) were procured from Sigma Aldrich® for all the pesticides used in the study and purity were range 98.5%-99.8%.

Instrument details and operating parameters

Agilent 6890/5975B GC-MSD (Mass Selective Detector) equipped with DB 35 MS fused silica capillary column (Agilent J & W GC® column, 5% phenylated methyl siloxane, 30m length, 0.25mm internal diameter and 0.25µm film thickness) was used for the analysis of pesticides. Concentrated samples were analyzed by GC/MS in selective

ion mode. Presence of pesticides was confirmed with the Retention Time (RT) and Mass Spectrum (MS) (Hussain and Maqbool *et al*., 2014). Matching RT and MS data of the sample peak with that of the CRM gave unambiguous identification of the pesticides presented in the sample analysis was carried out using temperature programming of the initial temperature 80°C for 1 min, a ramp rate of 10 °C/min up to a temperature of 160 °C with a holding time of 1 min, followed by 6 °C min ramp to a temperature of 250 °C with a holding time of 1 min, and by 10 °C min ramp to a temperature of 300 °C with a holding time of 5 min. The injector was operated in splitless mode at 280 ^{o}C. The interface, ion source and quadrapole temperatures were set at 280 ^{o}C, 250 ^{o}C and 150 ^{o}C, respectively. The instrument was operated at Electron Impact (EI) mode with electron energy 400 eV. Helium was used as carrier gas at a flow rate of 1 mL/min. Solvent delay time was given as 6.5 min. (Lehotay *et al*., 2005).

Preparation of working solutions

Working standards of 0.2, 0.5, 1.0, 1.5 and 2.0 mg L^{-1} were prepared from the primary working standard kept at 4-6 °C. Calibration curves were prepared at these five concentrations for each of the pesticides. Pesticides that showed analytical results within the lowest and the highest calibration standards, with linear correlation more than 0.99 were accepted.

Procedure for pesticide residue analysis

Samples were cut coarsely with a stainless steel knife and ground separately by using a mechanical blender. The blender was cleaned thoroughly before being used for the next sample to avoid cross contamination. The blended samples were placed separately in clean plastic containers, labelled and frozen overnight (AOAC, 2011). The low temperature storage was expected to reduce the deteriorative reactions and contamination from the environment. Further, low temperature storage was required as the temperature of the mixture could increase rapidly after addition of the reagents thus, changing or destroying the pesticide residues in the samples.

Each sample that was homogenized and frozen overnight was weighed into 15 g portions, 10 g ± 0.1 g of sample was then transferred into 50 mL Teflon tube and 6 g of anhydrous magnesium sulphate, 1.5 g of anhydrous sodium acetate or 1.5 g of sodium

chloride were added (Michelangelo *et al*., 2003). Ten mL of 1% acetic acid in acetonitrile was added into the sample tube, vortexed for 1min and centrifuged at 3,000 rpm for 3min. An aliquot from the supernatant layer was transferred into the dispersive 15 mL SPE tube containing 400mg PSA, and 1,200 mg magnesium sulphate. The Solid Phase Extraction (SPE) tubes were also vortexed for 1min and centrifuged for 3 min. at 3,000 rpm. The supernatant layer was transferred into a rotary evaporator and the content was evaporated at a temperature below 40 °C, concentrated to near dryness, and dissolved in 1 mL of acetone. The solution was passed through a 0.45 µm HDPE filter and the extract was injected to GC/MS for quantitative analysis (AOAC, 2011). Concentrations of pesticides in samples were determined using calibration curves.

Linearity

All calibration curves were prepared with multi-pesticide standard solutions, including the blank standard solution. The correlation coefficient (r) values were calculated for each curve. The analytical calibration extended over a range appropriate to the lowest and the highest concentration of the analyte, and the linearity criteria of $r^2 \geq 0.99$ had to be achieved.

Determination of recovery percentage

Replicates of high-spiked and low-spiked samples were analyzed using the candidate method and recovery percentage was estimated. The mean recoveries for each level were in the range of 70-110%, with an acceptability RSD ≤ 20%.

Maximum Residue Level (MRL)

The maximum residue level (MRL) is the maximum concentration of pesticide residues (mg kg^{-1}) to be legally permitted in or on food commodities and animal feeds recommended by the CODEX (CODEX Alimentarius, 2015) or National Regulatory Authority (e.g. Gazette Extraordinary, 16.07.2017). Different MRL's data available for selected pesticides are shown in Table 4.

Table 4. Different MRL data for the selected crops

Pesticide	MRL's	Crop					
		Apple ppm	Grape ppm	Orange ppm	Onion ppm	Chilli ppm	Potato ppm
Bifenthrin	CODEX	-	0.300	-	-	-	-
	EU	0.010	0.300	0.050	0.010	-	0.050
Chlorpyrifos	CODEX	-	0.500	-	0.20	20.000	2.000
	EU	0.010	-	1.500	0.20	-	0.010
Diazinon	CODEX	-	-	-	0.050	-	0.010
	EU	0.010	0.010	0.010	0.050	-	0.010
Dicofol	CODEX	0.100	0.100	0.100	-	-	0.020
	EU	0.020	-	0.020	0.050	-	0.050
Fipronil	CODEX	-	-	-	-	-	0.020
	EU	0.005	0.005	0.020	-	-	0.005
Quinalphos	CODEX	-	-	-	-	-	-
	EU	0.010	0.010	0.010	-	-	-
Pendimethalin	CODEX	-	-	-	0.050	-	-
	EU	0.050	0.050	0.050	0.050	-	0.050
Pirimiphos methyl	CODEX	-	-	-	-	-	-
	EU	0.010	0.010	0.010	0.010	-	0.010
Pretilachlor	CODEX	-	-	-	-	-	-
	EU	-	-	-	-	-	-
Prothiophos	CODEX	-	-	-	-	-	-
	EU	-	-	-	-	-	-
Oxyfluorfen	CODEX	0.010	-	-	-	-	-
	EU	-	-	0.050	0.050	-	0.050
Tebucanazole	CODEX	1.000	6.000	-	0.150	-	-
	EU	-	-	0.090	0.150	10.000	0.020

- Not available

The limit of detection (LOD) for pesticide residue analysis in crops was 40 ppb. The LOD values of the converted matrix with uncertinity for pesticide residues are 0.040±0.006 mg kg^{-1} for all pesticides.

Results and discussion

Quantification of selected pesticides

The quantitation ions, confirmation ion, calibration range and the retention times of the selected pesticides are presented in Table 5. The chromatograms for the selected pesticides are illustrated in Figure 1 and Figure 2. The calibration range is within 0.04-0.40 mg kg^{-1}

Table 5. Quantitation ion, conformation ion and calibration range of selected pesticides

Compound	Quantitation ions	Confirmation ion	Calibration range (mg kg^{-1})	Calibration range (mg kg^{-1})
Pirimiphosmethyl	290, 305, 276	290	0.04-0.40	0.04-0.40
Dicofol	139, 250, 141	139	0.04-0.40	0.04-0.40
Pendimethalin	252, 281, 253	252	0.04-0.40	0.04-0.40
Quinalphos	146, 157, 162	146	0.04-0.40	0.04-0.40
Pretilachlor	238, 162, 202	238	0.04-0.40	0.04-0.40
Bifenthrin	181, 182, 180	181	0.04-0.40	0.04-0.40
Quinalphos	146, 157, 162	146	0.04-0.40	0.04-0.40
Diazinon	304,179,199	304	0.04-0.40	0.04-0.40
Chlorpyriphos	314,197,199	314	0.04-0.40	0.04-0.40
Oxyfluorfen	252, 361, 300	252	0.04-0.40	0.04-0.40
Prothiophos	309,267,162	309	0.04-0.40	0.04-0.40
Tebuconazole	250,125,126	250	0.04-0.40	0.04-0.40

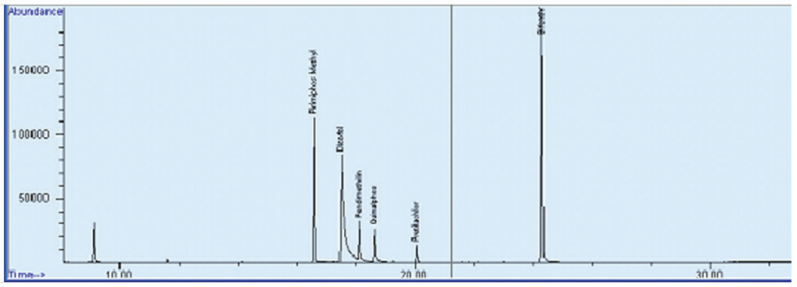

Figure 1. Chromatogram for twelve selected pesticides (group 1)

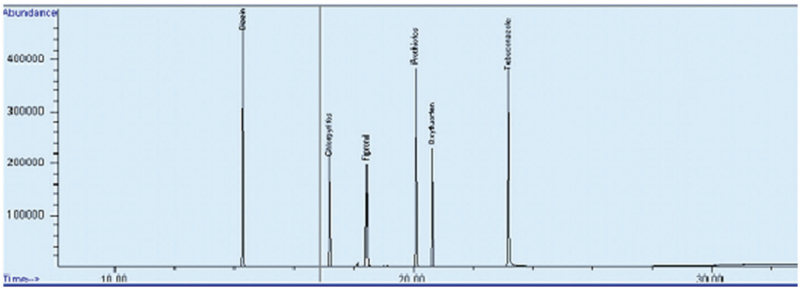

Figure 2. Chromatogram for six selected pesticides (group 2)

Linearity of standards

Calibration curve for selected pesticides are illustrated in Figure 3, Figure 4 respectively and correlation coefficient is >0.9900.

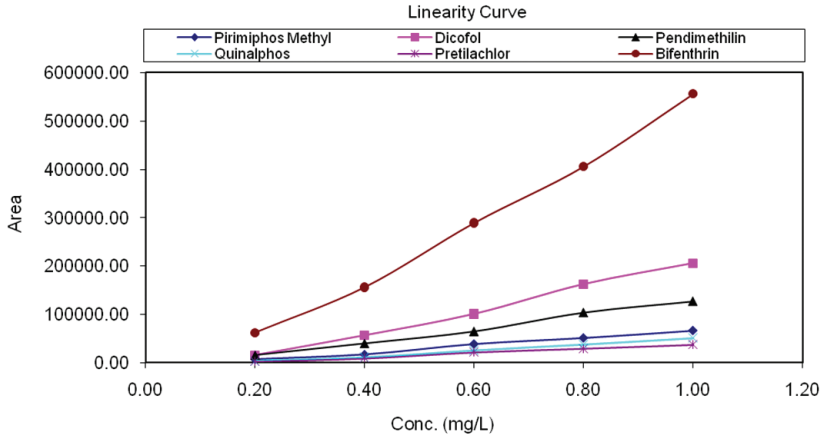

Figure 3. Calibration curve for selected pesticides (group 1)

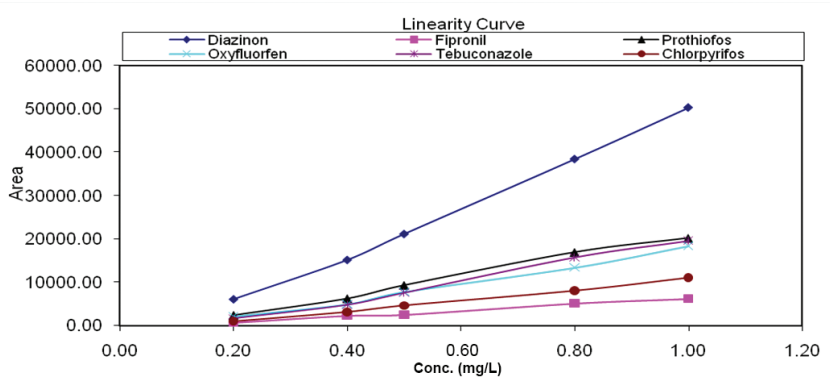

Figure 4. Calibration curve for selected pesticides (group 2)

Comparison of pesticide residues in imported and locally-produced commodities

Of 57 locally-produced commodity samples analyzed and the maximum pesticide detection of 22.2% was encountered with bifenthrin followed by oxyfluorfen (detection of 17.5%). Fipronil and tebuconazole were detected at 12.3% and 7%, respectively. The least detected pesticides were pendimethalin, pretilachlor and chlorpyriphos at or below 2.8%.

Of 55 samples of imported commodities analyzed and the, the maximum pesticide detection was encountered with oxyfluorfen (16.7%) while fipronil and tebuconazole were detected at the rate of 14.8% and 12.9%, respectively. Diazinon and chlorpyriphos were among the least detected pesticides at 5.6% and 1.9%, respectively.

The percentages of the detection between locally-produced and the imported commodities are illustrated in Figure 5. There were more detections for fipronil, tebuconazole and diazinon in imported commodity samples compared to locally-produced commodity samples. The detection of oxyfluorfen was marginally above in locally-produced commodity samples than imported samples. However, the detection of chlorpyriphos was comparable (Figure 5). Chlopyrifos, fipronil, oxyflurofen and tebuconazole detections in locally produced samples ranged between 0.04 ppm to 0.05 ppm, 0.044 ppm to 1.010 ppm, 0.052 ppm to 6.57 ppm and 0.048 ppm to 0.126 ppm, respectively while chlopyrifos, diazinon, fipronil, Oxyflurofen and tebuconazole

detection in imported samples ranged between 0.04 ppm to 0.058 ppm, 0.056 ppm to 0.070 ppm, 0.060 ppm to 0.300 ppm, 0.070 ppm to 4.620 ppm, respectively.

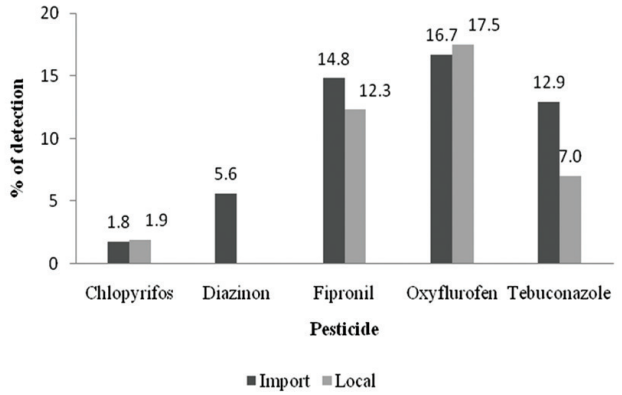

Figure 5. Precentage detection in imported and locally-produced commodity samples

Pesticide residues in imported and locally-produced commodity samples by crop category

The percent detection of pesticides in imported commodities is illustrated in Figure 6. Detection of five pesticide residues on apples ranged within 0.050 ppm-0.450 ppm and detection of pesticides in chillies fluctuated between 0.068 ppm to 0.380 ppm while residues in grape detected between 0.050 ppm to 0.070 ppm. Furthermore, residue detection ranged between 0.050 ppm to 4.620 ppm and 0.058 ppm to 0.060 ppm for residues in oranges and potatoes in imported commodities, respectively. Out of 20 numbers of Locally grown Orange samples, only 9 samples were exceeding the MRL's and from the total of 16 samples of locally grown Potato samples, only 7 samples were exceeding the MRL's. Out of 20 samples of locally grown onions, eight samples of locally grown Onion samples were exceeding the MRL's and from total number of 20 samples of imported apples, 7 samples were contaminated with pesticides, thus only one sample is exceeding the MRL's. Out of 12 number of imported Onion samples only 4 samples were exceeding the MRL's.

The highest detection rate (16.7%) was recorded on orange followed by apples & grapes with equal opportunities (11.1%). In contrast, the tested vegetable samples showed less frequencies of detection in the range of 5.1% - 8.3% (Figure 6).

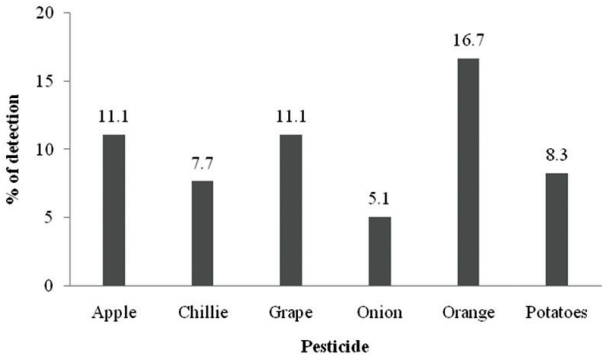

Figure 6. Precentage of detection on imported samples by crop

Meanwhile, the percentage of detection of pesticides in imported and locally-produced samples by selected crop categories is shown in Figure 7. The frequency of detection of residues in imported orange was almost three times compared to locally-produced orange. Similarly, the frequency of detection of residues in imported potato was more than double compared to locally-produced potato (Figure 7). Detection of the twelve pesticide residues in local potato ranged between 0.048 ppm to 6.57 ppm, while, residues in onion and orange ranged between 0.050 ppm to 1.010 ppm and 0.044 ppm to 0.156 ppm

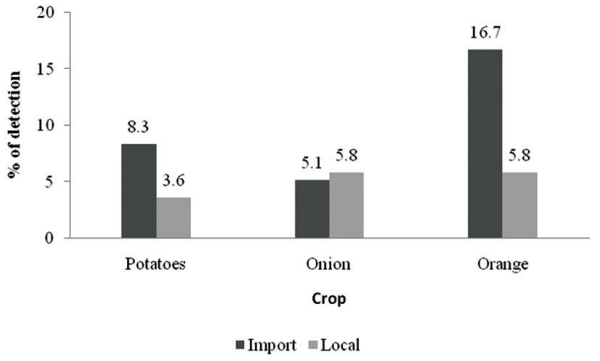

Figure 7. Precentage of detection on imported and locally-produced samples by crop categories

Pesticide residues in imported sample with country and crop category

The precentage of detection in imported samples according to the country and the crop category is shown in Figure 8. The detection rates were 13.3% and 12.5%, respectively. Meanwhie, the imported potatoes from India and China were less frequently contaminated with tested pesticides; the detection rates were 8.3 and 5.6%, respectively. Although less frequent, onion & dry chillies from India were also contaminated with detectable amounts of pesticides at 5.6% and 6.9%, respectively. From 20 samples imported from india reported with 11 detections as 2 detections on potato samples with 0.050 ppm and 4 detections in onion samples ranged between 0.050 ppm to 4.62 ppm and 5 detections for dry chillie samples ranged between 0.05 ppm to 0.160 ppm. Futhermore 14 samples of potato and apples imported from china were contaminated with 10 detections including 0.054 ppm detection in one potato sample and 9 detections on apple samples ranged between 0.05 ppm to 0.450 ppm.

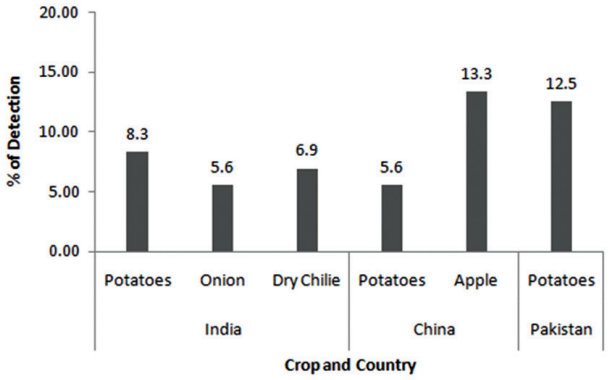

Figure 8. Precentage of Detection on imported samples by country and crop category

Pesticide residues in imported sample with country and type of pesticide

Percentage of detection in imported sample with country and pesticide are shown in Figure 9. The highest rate of detection was observed for contamination from tebucanazole for commodity samples imported from China. 20% of detection rates in Diazinon,Fipronil, and Oxyflurofen observed for commodities imported from India. Meanwhie, the imported commodities from pakistan was less frequently contaminated with tested pesticides. 20% of detection rates for chlopyrifos was detected with commodities imported from Pakistan. The samples imported from india showed 11 detections including one detection

with 0.050 ppm for diazinon and five detections for fipronil ranged between 0.060 ppm to 0.380 ppm. Meanwhile there were 4 detections reported for oxyflurofen reported with 0.050 ppm to 0.082 ppm and one sample was reported with 4.60 ppm for tebucanazole. Potatoes and apples imported from china reported with 10 detections as 0.076 ppm for diazinon and 0.15 ppm detection for fipronil while two detections for oxyflurofen reported between 0.050 ppm to 0.054 ppm and 6 contaminations for tebucanazole ranged with 0.070 ppm-0.450 ppm. From 5 samples imported from pakistan observed with two detections including chlopyrifos and oxyflurofen contained 0.058 ppm to 0.081 ppm.

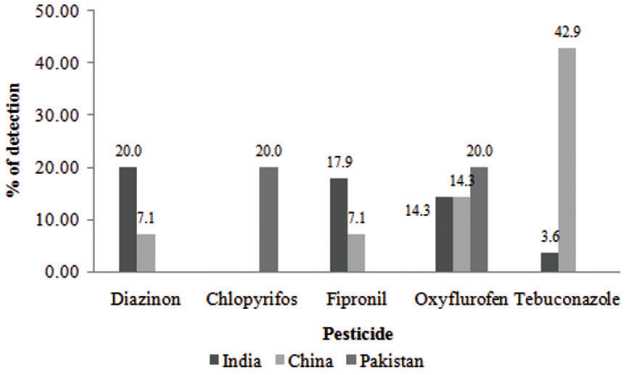

Figure 9. Precentage of Detection on imported samples by pesticide and country

Pesticide residues detections in local samples with crop categories

Percentage of detection in local samples according to pesticide and commodities are shown in Figure 10. The highest levels of detections were observed for the Oxyflurofen in onion and the Bifenthrin in orange. Detection rates were 33% and 30% respectively. From 16 local potato samples , only 07 detections reported as 2 detections for bifenthrin observed between 0.06 ppm to 0.07 ppm and 0.906 ppm detection for fipronil. Meanwhile, 3 detections for Oxyflurofen reported between 0.066 ppm to 0.570 ppm and 0.048 ppm detection was reported for tebuconazole. Furthermore from 20 local orange samples observed with 14 detections including 0.044 ppm detection for pendimethalin, 0.138 ppm detection for pretilachlor and 6 detections between 0.066 ppm to 0.152 ppm for bifenthrin, 3 detections between 0.044 ppm to 0.048 ppm for fipronil and 3 detections for tebuconazole ranged between 0.084 ppm to 0.126 ppm. Meanwhile from 21 local onion samples were reported with 11 detections including 0.050 ppm for chlopyrifos,

3 detections for fipronil reported between 0.058 ppm to 1.010 ppm and 7 detections for oxyflurofen between 0.052 ppm to 0.063 ppm.

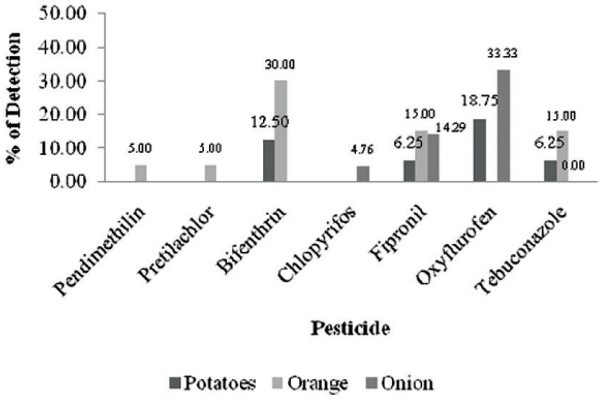

Figure 10. Percentage of detection in imported samples by pesticide and commodities

Conclusions

None of the samples were contaminated with Pirimiphos Methyl, Dicofol, Quinalphos and Prothiophos. One of the imported potato sample and local onion sample were contaminated with Chlopyrifos and not exceeding the MRL's. MRL's for Chilli has unavailable except Chlorpyrifos and Tebuconazole.

There are several violations of some pesticide residues in study samples and exceeding the MRLs declared by the European Union (EU) and FAO CODEX. . There were more opportunities for detection of fipronil, tebuconazole and diazinon in imported commodity samples compared to locally-produced commodity samples. The detection of oxyfluorfen was marginally above in locally-produced commodity samples than imported samples . The results revealed the presence of pesticide residues in both imported and local samples. The scope of residue analysis is only limited for twelve pesticides and other pesticide may be presence on this samples and results only revealed in only for studied pesticides.

This study was initiated to fill the research gap and to make an analysis for imported commodities. It is recommended to expand monitoring studies to other Pesticides and study cannot be generalizing for pesticide contamination in local and

Imported commodity samples. Such studies will serve as a basis for future policy about the standards and quality control for imported commodities.

References

AOAC International, (2011). AOAC Official Method 2007.01 Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate. Official Methods of Analysis of AOAC International 90(2):17-26.

Chandra, S., A.N. Mahindrakar and L.P. Shinde, (2014). Analysis of Pesticide Residue in Vegetables Local Market. International Journal of ChemTech Research 6(5): 2760–2768.

CODEX Alimentarius. http://www.fao.org/cordexalimentarius (Accessed 15th July 2017).

Denis H., A. Ambrus, D. Roland, F. Allan, H. Caroline, P. Barbara, R. Ken, G. Roberto, T. Keiji, E. Mike, R. Graham and B. Raj, (2004). Pesticide residues in food acute dietary exposure. Pest Management Science. 60(4): 311–339.

Esham, M. 2006. Procurement Behavior of the Fruit and Vegetable Industry in Sri Lanka. Journal of Rural Problems. 36–44.

Gazette Extraordinary, No 2023/34, Sri Lanka. Issued on 14.06.2017.

HARTI food information bulletin, (2018). http/hart.gov.lk/market information/2018/monthly/April 000208 (accessed on the May 20, 2018)

Hussain, S.Z. and K. Maqbool. (2014), GC-MS : Principle , Technique and its application in Food Science, International Journal of Current Science. 13: 116–126.

Lehotay, S.J., A. de Kok, M. Hiemstra, P. Van Bodegraven, (2005) Validation of a fast and easy method for the determination of residues from 229 pesticides in fruits and vegetables using gas and liquid chromatography and mass spectrometric detection. *Journal of AOAC International*. 88(2):595–614.

Michelangelo A., J.L. Steven, S. Darinka and J.S. Frank, (2003). Fast Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning, Dispersive Solid-Phase Extraction for the Determination of Pesticide Residues in Produce, Journal of AOAC International. 86.

Perera, S., M. Rambodagedara, R. Wijesinghe. (2015). Fruit and vegetable export growth, instability and diversification. Hector Kobbekaduwa Agrarian Research and Training Institute, Colombo, Sri Lanka

Recommended Methods of Sampling for The Determination of Pesticide Residues for Compliance with Mrls Cac/Gl 33-1999 pg 8, http://www.fao.org/fao-who-codexalimentarius. 8. (Accesed on 10.05.2019).

World Health Organization, (2002). Recommended classification of pesticides by hazard and guidelines to classification. World Health Organization, Geneva.