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Considering post harvest period of Chlorpyrifos insecticide on greenhouse tomatoes with the solid phase extraction method and with the help of the GC, GC/MS

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ABSTRACT

This study was conducted to measure post harvest period (php) of Chlorpyrifos . Tomato samples cultivated in 2 greenhouses with the design pattern of randomized complete block with 3 replicates. Every treatment sprayed with studied insecticide 2 times. Within different days after spraying sampling was performed. Samples in laboratory were extracted by using SPE(Solid Phase Extraction) and with the help of N method were concentrated and extracts were injected for measurement into GC(Gas Chromatograph),GC/MS(GC/Mass Spectrometer) devices. Data were compared with WHO/FAO MRL and Recovery results were 101% also results showed that Chlorpyrifos has the post harvest period near 15 days in greenhouse condition has been studied.

Key words: Insecticide residue, MRL, post harvest period, Recovery test.

INTRODUCTION

According to an economic perspective the tomato is considered the second most valuable agricultural after potato, and also in terms of per capita consumption is then. Because the tomato is one of the products to be consumed fresh and raw, there remains the possibility that pesticides can be very disturbing (torres *et al* 1996). Pesticide residues in agricultural products, particularly because of the greenhouse-coated culture are important. Depending on the environment because farmers are faced with various pests and frequent use of different pesticides that regard to glass or plastic used in roof and walls of the greenhouse effect and the wavelength of light in the greenhouse pesticides is not into the environment (Sadlo *et al*., 2000). Since most of these compounds are relatively toxic and persistent pesticides are in the environment that able to adverse effects on human health and the environment. Pesticide is absorbed into the body through food far more than is absorbed by water and air that it remains to determine the significance of pesticides in crops can be fixed. So today, many studies are doing on pesticide residues measuring in Iran and the world. (Wong *et a*l.,2004). Due to the high per capita consumption of tomatoes contaminated samples can cause large amounts of toxins in the body and cause acute and chronic poisoning.... This study is important in terms of health.

MATERIALS AND METHODS

In this study Tomato samples cultivated in 2 greenhouses with the design pattern of randomized complete block with 3 replicates. Every treatment sprayed with studied insecticide 2 times. Within one hour and 1, 3, 5,7,10,15,20,30 days after spraying sampling was performed. Tomato samples randomly collected and each sample was 2 kg .Collected samples carried out to laboratory and each of them divided to five small parts and blended with a laboratory blender, then 200 gr separated for extraction procedure. After mixing and homogenize them with a proper homogenizer, 20 gr separated for sample preparation and pesticide residue measuring. Each of these seprated samples mixed by 10 ml acetonitryl,10 ml HPLC grade water and 10 ml methanol and shaked them for 15 minutes then transferred samples to a sonicator for ten minutes .Mixed materials separated by a centrifuge and supernatant used for next procedure. The supernatant liquid first filtered, controlled their PH and adjusted between 3-7/5 these extract applied to SPE cartridges.The next step for the separation of analytic extraction and purification of the solid phase cartridges were used in the study of chemical properties of C18 solid phase cartridges.A separate cartridge for each sample was initially considered in the first 10 ml hexane and 5 ml water and finally 5 ml of methanol deionizer slowly passed through the column when the level of methanol in the column to the absorber surface extract that was prepared to enter columns and it was passed through 5 ml of ethyl acetate and then poured into the column and collect the output of liquid extract or an extract of the above-named machines for injection Gas Chromatography, Gas Chromatography with Mass Spectrometers were kept in the fridge for an extra step in isolating the toxin in 5 ml of the hexane extract was passed through the cartridge outlet and there were some tests to control the collection and was named No. 2.

Gas chromatography conditions:
HP-5 column Capillary - NPD detector type - Injector temperature 200˚c - 260˚c and the detector temperature, injection volume of 10 micro liters.

Temperature programming gas chromatography:
Starting80˚c with the slope at 10 ˚c per minute to 160 ˚c and with the slopes 5 ˚c/ Min to 260 ˚c and 5 min hold at 260 ˚c.

Gas chromatography with mass spectrometers:
Capillary HP-5 column 30 m long - Injector temperature 220˚c temperature detectors 160 ˚c, 1 micro liters injection volume.

Recovery test At Two concentrations and four replicates were run for each concentration. For testing some pure poison Spraying was not added to the sample concentration in the samples to separate the 0/2 and 2 ppm and then be extracted from these samples was similar to the unknown samples and the preparation of the final extract was measured to determine the percent recovered. Standards were prepared from different concentrations of toxins (0/01-0/1- 1-10-100-1000 ppm) and the injection system was studied and the calibration curve and the data were used to evaluate the detector response.

RESULTS

The average percent recovery was 101%. recycling rate in solid-phase extraction method was high, indicating that the application of this method was successful and the venom extracted from the reliable and accurate results in the removal, containing residual solvent to extract pesticides with gas chromatography systems, gas chromatographs and mass spectrometers were injected and pesticide residues, compared with the curve corresponding to each sample and standard curve was calculated. The amount of spraying the tomatoes have been different at different times and average remaining an hour after spraying was calculated 51 / 9 ppm, and one day after spraying 16 / 7 ppm. Pesticides residues pesticide residues compared with the limit set by the Codex Food (0 / 5 ppm for Chlorpyrifos) showed no significant difference in the average amount outstanding at 15. So Chlorpyrifos period about 15 days there. Data analysis using SPSS was 5% α level. The injection of various concentrations of standard curve was plotted and the equation Y = 5.065.56x was obtained Chlorpyrifos line.

The detection limit (LD) of the device were as follows:
Gas chromatograph: 0 / 001 ppm
Gas chromatography with mass spectrometer: 0 / 07 ppm

Table 1- Compare means chlorpyrifos residue with MRL (ppm)

Day after application of pesticide	The average of measured chlorpyrifos(ppm)	MRL	Explanations
0	9.51	0.5	significantly different
1	7.16	0.5	significantly different
3	4.60	″	″
5	2.30	″	″
7	1.63	″	″
10	0.70	″	″
15	0.029	″	″
20	ND	″	are not significantly different
30	ND	″	are not significantly different

* *ND (Not detect)*

Table 2- The average percent recovery test

Pesticide	Concentration of standard ppm	Recovery percent average	Total average
Chlopyrifos	0/2	104%	101%
	2	98%	

DISCUSSION

Post harvest period awareness of the toxins and compliance by the manufacturers of this period is important. Post harvest period a pesticide depends on many factors. The amount of pesticides that can be used, weather conditions, irrigation, types and varieties planted crop, sowing time and type of formulation used as pesticides (Lze-lyamu et al 2007). According to Iran's per capita consumption of 62.2 kilograms of tomatoes per year. Note that the remaining amount of the tomato product measured is very high and thinking of a solution to remove it because of recent epidemiological studies show that more than 80 percent of the remaining pesticides has serious risks in humans, the health consequences depending on the amount of toxin can cause both acute and chronic. Poisoning, abortion, skin and neurological symptoms, behavioral disorders and cancer are the most common adverse effects associated with pesticides. Chlorpyrifos contaminations of tomato products were analyzed. In this study of the pollution over the limit was close to 30 percent (Meloni et al 2001). Also studies about pesticides of Chlorpyrifos, Diazinon, Fozalon, Permethrin and some Chlorinated Lindin that show pollution levels exceeded (Dejonckheere et al 1996). In another study it was found that 53/33% of samples Cucumbers and tomatoes were infected to Chlorpyrifos, Andosolfan and Fozalon, pesticides (Fernande *et al* 2000). In another study conducted in Mazandaran on cucumber showed that most products are contaminated with toxins Benomil and Mankuzb pesticides (Shokrzade et al 2003). Studies on tomatoes for pesticide Chlorpyrifos and Dimitvat also indicates the amount of pollution in the product poisons (Aysel et al 2004). In another study in the province of Chahar Mahal and Bakhtiari shows that 100% of the samples exceeded the remaining cucumber and tomatoes are Oxi Dimeton Metil pesticide (Mardani et al 2008). Therefore, this issue should be taken seriously and the different ways to solve the problem.

Suggestions:
1 - Training to farmers and manufacturers on how to use pesticides
2 - Continuous and accurate monitoring of experts on pesticide spraying and various onsumer products
3 - Establish centers for the control of residues of pesticides in fruits and vegetables before consumption cycle
4 - Use of pesticides and toxins, rather than systemic and lasting durability.

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