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**RESEARCH ARTICLE :**

# Monitoring of pesticide residues in okra (*Abelmoschus esculentus* L.)

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**SUMMARY :** The okra samples were collected from three local vegetable markets *viz.*, Rythu bazar, Tower circle and Ramnagar of Karimnagar during 2014-15 and analyzed for residues following the validated QuChERS method. The results revealed that, monitored samples of okra contained 10 pesticide residues namely acetamiprid, flubendiamide,chlorpyriphos, profenophos, fipronil, spinosad, lambda cyhalothrin, monocrotophos, acephate and imidacloprid and the commonly detected pesticides in the three market samples were flubendiamide, lambda cyhalothrin, fipronil, profenophos, chlorpyriphos and acetamiprid. The insecticide residues detected in the okra samples collected from Rythu bazar vegetable market were flubendiamide (0.142 mg/kg), profenophos (0.042 mg/kg), acephate (0.193 mg/kg), fipronil (0.028 mg/kg), chlorpyrifos (0.275 mg/kg), lambda-cyhalothrin (0.241 mg/kg), acetamiprid (0.167 mg/kg), monocrotophos (0.011 mg/kg), and imidacloprid (0.032 mg/kg) while spinosad was not detected. The insecticide residues detected in the okra samples collected from Tower circle vegetable market were flubendiamide(0.183 mg/kg),chlorpyriphos (0.123 mg/kg), profenophos (0.065 mg/kg), acetamiprid (0.242 mg/kg), acephate (0.009 mg/kg),fipronil(0.013 mg/kg),spinosad (0.128 mg/kg),lambda cyhalothrin (0.201 mg/kg) and monocrotophos (0.022 mg/kg) while imidacloprid was not detected.The insecticide residues detected in the okra samples collected from Ramnagar vegetable marketwere lambda cyhalothrin (0.041 mg/kg), flubendiamide (0.168 mg/kg), chlorpyriphos (0.289 mg/kg), acetamiprid (0.142 mg/kg), profenophos (0.052 mg/kg), fipronil (0.042 mg/kg), imidacloprid (0.062 mg/kg) and acephate (0.184 mg/kg)while monocrotophos was not detected. Assessment on the permissibility of the three market samples of okra for safe consumption was also analysed. Among the samples analyzed for different insecticide residues, two of the insecticides *i.e.* chlorpyriphos and acetamiprid have shown above MRL value established by Codex, while the remaining insecticide residues detected in the samples were below the MRL.The chlorpyriphos residues exceeding the MRL were recorded in three markets *i.e.*, Rythu bazar (0.275 mg/kg), Tower circle (0.346 mg/kg) and Ramnagar (0.289 mg/kg). The acetamiprid residues exceeding the MRL were recorded in two markets *i.e.*, Rythu bazar (0.340 mg/kg) and Tower circle (0.242 mg/kg).

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## BACKGROUND AND OBJECTIVES

Okra (*Abelmoschus esculentus* L.) is an important vegetable crop grown throughout the year in India. The productivity of our country is low compared to other countries due to yield losses caused by insect pests, diseases and nematodes. The crop is attacked by more than 72 insect pests and infestation is observed from seedling to harvest stage. Farmers cannot tolerate any loss to the vegetables either by insects or diseases and resort to chemical control.Pesticide use has increased rapidly over the last two decades at the rate of 12 % per year (Thacker *et al*., 2005). About 13% of total pesticides used in agriculture are consumed by vegetable crops which cover only 3% of total cropped area. The wide spread use of pesticides resulted in the presence of their toxic residues in various environmental components/ commodities (Kumari *et al*., 2002, 2008 and Srivastava *et al*., 2011 and Wang *et al*., 2011).

Indiscriminate and improper use of pesticides on vegetables and negligence to follow proper waiting periods make marketed vegetables very often contaminated with pesticides (Lakshminarayana and Menon, 1975). Many farm gate vegetable samples showed presence of insecticide residues (Singh *et al*., 1999). Literature reveals that vegetables which contain the residues of pesticides above their respective maximum residue limit MRL may pose health hazards to consumers (Elliion *et al*., 2000 and Mukherjee and Gopal, 2003). Thus, contamination of vegetable crops is sometimes more than the prescribed tolerance limits.

Monitoring of pesticides is conducted globally to assess the environmental load of their residues. These pesticide residues find their way into the human body through food, water, and environment. Thus, analysis of pesticide residues in food and other environmental commodities like soil, water, fruits, vegetables, and total diet have become essential requirement for consumers, producers, and food quality control authorities.

## RESOURCES AND METHODS

The Okra samples were collected from three different vegetable markets of Karimnagar *viz.,* Rhythu Bazar, Tower circle and Ramnagar. The sample size collected from each market was five kgs of okra. The samples were collected from different vendors in the market to get a representative sample.

The collected okra samples were tested for different pesticide residues to find out the frequently used pesticides. The sample was homogenized using Robot Coupe Blixer (High volume homogenizer), homogenized sample of each 15 g was taken into 50 ml centrifuge tubes, 30±0.1 mlacetonitrile was added to the tube and then it was silent crushed (to get fine sample).Later,3gms of sodium chloride was added, centrifuged for 3 minutes, the supernatant of 16ml was taken into 50 ml centrifuge tubes, into this added 9 g of anhydrous sodium sulphate, from this 8ml was taken into 15 ml centrifuge tubes, into which 1.2 g magnesium sulphate and 0.4 g of PSA was already added, kept on vertex for 30 seconds, centrifuged for 5 minutes and 2ml of it was transferred into the vials. These samples were used in GC and LC analysis of chemicals. These fortification levels are selected to know the suitability of the method to detect and quantify pesticides in okra below Maximum Residue Limits (MRLs) of Codex Alimentarius Commission (CAC).

The AOAC official method 2007.01 (Pesticide Residues of Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulphate) was slightly modified to suit to the facilities available at the laboratory and the same was validated for estimation of LOQ (Limit of Quantification).The final extract of the sample *i.e.* 2 ml equal to 1 g of the sample was evaporated using turbovap and made upto 1 ml (equal to 1 g sample) using suitable solvent for analysis on GC, while for LC analysis, filtered 1 ml final extract (equal to 0.5 g sample) was directly injected in LC and the residues of pesticides recovered from fortified samples were calculatedusing the following formula.

$$\text{Residues (mg/kg)} = \frac{\text{Sample peak area x conc. of std. (ppm) x µl of std. injected x Final volume of the sample}}{\text{Standard peak area x Weight of sample x µl of sample injected}}$$

$$\text{Weight of the sample analysed} = \frac{\text{Sample weight (15 g) x aliquot taken}}{\text{Volume of accetonitrile (30 ml)}}$$

## OBSERVATIONS AND ANALYSIS

The okra samples were collected from three different vegetable markets *viz.,* vegetable market at Rythu bazar, vegetable market at Tower circle and the local vegetable market, Ramnagar of Karimnagar and analyzed for insecticide residues following the validated QuEChERS method. The residue results for each market are presented below.

**Rythu bazar, Karimnagar:**

The okra samples collected from Rythu bazar vegetable market were contaminated with various insecticidesat detectable levels, and presented in Table 1. The insecticide residues detected were flubendiamide (0.142 mg/kg), profenophos (0.042 mg/kg), acephate (0.193 mg/kg), fipronil (0.028 mg/kg), chlorpyrifos (0.275 mg/kg), lambda-cyhalothrin (0.241 mg/kg), acetamiprid (0.167 mg/kg), monocrotophos (0.011 mg/kg), and imidacloprid (0.032 mg/kg), while spinosad was not detected.

**Vegetable market, Tower circle :**

The okra samples collected from Tower circle vegetable market were contaminated with insecticide residuesat detectable levels and presented in Table 1. The insecticide residues detected were flubendiamide (0.183 mg/kg),chlorpyriphos (0.123 mg/kg), profenophos (0.065 mg/kg), acetamiprid (0.242 mg/kg), acephate (0.009 mg/kg),fipronil(0.013 mg/kg), spinosad (0.128 mg/kg),lambda cyhalothrin (0.201 mg/kg) and monocrotophos (0.022 mg/kg) while imidacloprid was not detected.

**Vegetable market, Ramnagar:**

The okra samples collected from Ramnagar vegetable market were contaminated with insecticide residuesat detectable levels and presented in Table 1. The detected insecticide residues were lambda cyhalothrin (0.041 mg/kg), flubendiamide (0.168 mg/kg), chlorpyriphos (0.289 mg/kg), acetamiprid (0.142 mg/kg), profenophos (0.052 mg/kg), fipronil (0.042 mg/kg), imidacloprid (0.062 mg/kg) and acephate (0.184 mg/kg)while monocrotophos was not detected.

In the present study, it was found that except acephate, spinosad, monocrotophos and imidacloprid, all other insecticides like profenophos, acetamiprid, fipronil, flubendiamide, lambda cyhalothrin, chlorpyriphoswere detected in all the three markets.The insecticides that were found in all the three markets were profenophos, acetamiprid, fipronil, flubendiamide, lambda cyhalothrin and chlorpyrifos

The results found are in accordance with the findings of Ananda Gowda and Somashekar (2012) who reported 58% of okra samples collected from different vegetable markets of Karnataka were highly contaminated with acephate residues. Charan *et al*. (2010) monitored samples of okra, cauliflower, brinjal, tomato, cabbage and potato and reported that 35.62% of samples exceeded the maximum residual limit (MRL) values. Subhash Chandra *et al*. (2014) reported that samples of brinjal, capsicum, cauliflower and okra from local market contained detectable levels of chlorpyriphos, cypermethrin and monocrotophos residues but below the maximum residue limits (MRL). Beena Kumari (2007)

**Table 1 : Residue levels of various insecticides in okra samples collected from three vegetable markets**

| Sr. No. | Insecticides | Residues (mg/kg) | | |
|---|---|---|---|---|
| | | Rythu bazar | Tower circle | Ramnagar |
| 1. | Flubendiamide | 0.142 | 0.183 | 0.168 |
| 2. | Lambda cyhalothrin | 0.078 | 0.126 | 0.041 |
| 3. | Monocrotophos | 0.011 | 0.060 | ND |
| 4. | Acetamiprid | 0.340 | 0.242 | 0.142 |
| 5. | Imidacloprid | 0.032 | ND | 0.062 |
| 6. | Acephate | 0.10 | 0.009 | ND |
| 7. | Fipronil | 0.020 | 0.014 | 0.012 |
| 8. | Chlorpyrifos | 0.275 | 0.346 | 0.289 |
| 9. | Profenophos | 0.042 | 0.065 | 0.052 |
| 10. | Spinosad | ND | 0.019 | ND |

N.D- Not Detected

**Table 2 : Insecticide residues exceeding the MRL values in okra samples collected from three vegetable markets of Karimnagar**

| Sr. No. | Insecticides | Markets | Residues detected | MRL values of residues by codex |
|---|---|---|---|---|
| 1. | Chlorpyriphos | Rythu bazar | 0.275 mg/kg | 0.2 mg/kg |
| | | Tower circle | 0.346 mg/kg | 0.2 mg/kg |
| 2. | Acetamiprid | Rythu bazar | 0.340 mg/kg | 0.2 mg/kg |
| | | Tower circle | 0.242 mg/kg | 0.2 mg/kg |

monitored samples of brinjal, cabbage and cauliflower and reported the residues of cypermethrin, chlorpyriphos and permethrin, each in two samples. Chowdhury*et al*. (2013) monitored eight types of domestic vegetables and reported that most frequently detected pesticides were chlorpyriphos, carbofuran, diazinon, carbaryl, malathion, endosulfan, cypermethrin and dimethoate. Pallavi *et al*. (2014) analysed okra samples and found residues of malathion and profenophos.

**Assessment of permissibility of insecticide residues in market samples of okra for safe consumption:**

In order to assess the permissibility of the market samples for safe consumption, insecticide residue levels of okra samples collected from three vegetable markets of Karimnagar *viz.*, Rythu bazar vegetable market, Tower circle vegetable market and Ramnagar vegetable market were analyzed and compared with maximum residue limits (MRL) established by Codex Alimentarius Commission on pesticide residues .

Among the okra samples collected from 3 markets and analyzed for different insecticide residues, two of the insecticides i.e. chlorpyriphos and acetamiprid have shown residues above the MRL value established by Codex (Table 2), while the remaining insecticide residues detected in the samples were below the MRL. The chlorpyriphos residues exceeding the MRLs were recorded in three markets i.e., Rythu bazar (0.275 mg/ kg), Tower circle (0.346 mg/kg) and Ramnagar (0.289 mg/kg). The acetamiprid residues exceeding the MRL were recorded in two markets i.e., Rythu bazar (0.340 mg/kg) and Tower circle (0.242 mg/kg).

In the present study, it was found that except chlorpyriphos and acetamiprid, the other insecticides detected were below the MRL values and it is in line with the findings of Beena Kumari (2007) who assessed market samples of okra, brinjal, cabbage, cauliflower and found 4-100% contamination with low but measurable amounts of residues and reported that the residues of chlorpyriphos exceeded their respective MRL values. Bhanti and Taneja (2005) evaluated the residual concentration of selected organophosphorous pesticides (methyl parathion, chlorpyriphos and malathion) in vegetables and found that the concentration of the various pesticides was below the established tolerances but continuous consumption of such vegetables even with moderate contamination level can accumulate in the receptor's body and may prove fatal for human population in the long term. Ahuja *et al.* (2006) reported that lambda -cyhalothrin residues fell below detectable limits in aubergine fruits and hence lambda -cyhalothrin can be used at the recommended rate on aubergine. Ahmed *et al.* (2011) reported detectable levels of pesticide residues of malathion, ethion, profenophos, fenpropathrin and cypermethrin at concentration equal to or exceeding their European Union's maximum residue limits (EU-MRLs). Godfred and Akoto (2008) reported that health risks were found to be associated with dietary intake of organophosphorus pesticide residues through vegetables from Kumasi, Ghana. Gilden *et al*. (2010) reported that the presence of pesticide residues is of concern for consumers because pesticides are known to have potential harmful effects to other non-targeted organisms than pests and diseases. Samriti and Kumar (2011) reported that chlorpyriphos residues in okra samples reached BDL of 0.010 mg/ kg on 7th and 15th day in case of single and double dose after treatment, respectively. Chourasiya *et al*. (2015) reported thatmost of the OCP residues recorded in vegetable samples exceeded the maximum residue levels (MRLs) set by international and national regulatory agencies.

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