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Research Article

STANDARDIZATION OF GAS CHROMATOGRAPHY METHOD FOR DETECTION OF DELTAMETHRIN RESIDUES IN CHICKEN MEAT

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ABSTRACT: A Gas chromatography-electron capture detector-based analytical technique for the detection of deltamethrin residues in chicken meat samples was standardized and validated in the present study. Deltamethrin was extracted from the chicken meat matrix as per QuEChERS protocol. The method standardized was sensitive, reliable, accurate, and precise and allows the detection of deltamethrin residues in chicken meat > 0.08 µg/g. The standardized method was applied for the detection of deltamethrin residues in 33 chicken meat samples collected from Tamil Nadu, India. Two samples of chicken meat were positive for deltamethrin residues and the percent of samples positive for deltamethrin was 0.06%. However, the levels were less than MRL values of deletamethrin as indicated by FSSAI, India. Monitoring studies with a higher number of samples are required to assess the actual status of pesticide residues in animal food products. Hence, regular monitoring programs and creating awareness among livestock farmers training are also required to control pesticide residues in animal food products.

Keywords: Gas chromatography, Deltamethrin, Pyrethroids, Residue, Chicken meat.

INTRODUCTION

Pesticide residues may be defined as the presence of pesticide or its metabolite in milk, meat, and egg. With the advancement of technology and intensification of agricultural practices and livestock production, the use of pesticides for eradication of pests and external parasites is inevitable and thus causing higher incidence of pesticide residues in these products. Consumption of meat and meat products contaminated with pesticide residue has been associated with increased incidence of tumor, kidney diseases, immune-suppression, reproductive disorders, and hormonal disorders, etc. in man [1, 2]. The presence of pesticide residue in these products is a major public health concern and also will have a great impact on the trade of poultry products and the economy.

The residues in these products may arise out of the direct use of pesticides to control external parasites or indirectly through feed and water. The widespread use of these compounds in farming can lead to contamination of the environment with pesticides which could cause exposure of farm animals to these compounds [3, 4]. In the agricultural sector Pyrethroid pesticides which include deltamethrin, cypermethrin, permethrin, and flumethrin are widely used for the control of pests and also during post-harvest storage. In veterinary medicine, it is used for the control of external parasites in farm animals [5].

The status of chemical contaminants in animal food products can be assessed by surveillance or monitoring programs but they are relatively a neglected area until the last decade. Of late, consumers are highly knowledgeable and readily express concerns about food safety. This can also affect the export of these products on quality issues. Hence, the present investigation was undertaken with the objective of standardization of analytical methods for the detection of deltamethrin residues and analyzing the chicken meat samples collected at four districts of Tamil Nadu, India for the presence of deltamethrin residues.

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MATERIALS AND METHODS

Chemicals and reagents

Deltamethrin standard, QuEChERS acetate extraction tubes (SupelTMQuE 55234-U), and SPE clean-up tubes (SupelTMQuE Z-Sep+ 55486-U) were purchased from M/s Sigma Aldrich Corporation, Bangalore. Chromatography grade Acetic acid, acetonitilre, ethyl acetate and methanol were purchased from M/s Thermo Fisher Scientific (Mumbai, India).

Preparation of pesticide standard

Deltamethrin was dissolved in ethyl acetate to prepare a stock solution of 100 µg/ml. The working standards were also prepared in ethyl acetate. Both stock solution and working standards were stored at 4°C.

Sample collection

A total of 33 samples of chicken meat from retail broiler outlets in Chennai, Chengalpattu, Kanchipuram, and Tiruvallur districts of Tamil Nadu were collected and stored at deep freezer (-20°C).

Instrumentation and chromatographic conditions

The instrument consisted of Gas chromatography (model 7890B GC, Agilent, USA) equipped with an electron capture detector, and autosampler (G4513) with splitless injection mode.

Nitrogen was used as a carrier gas at 1.2 ml/min at a pressure of 8.5231 psi. The oven was set at an initial temperature of 150°C with a heating rate of 20°C min-1 up to 250°C and then at the rate of 10°C min-1 up to 290°C and held for 5 min. The run time was 14 min. The detector and injector temperatures were 300°C and 280°C, respectively. The column used was a Phenyl Methyl Siloxane capillary column (30 m length X 320 µm diameter X 0.25 µm film thickness).

Sample extraction and clean up

Deltamethrin residues were extracted from chicken meat as per the QuEChERS protocol. To 15 g of homogenized meat samples 10 ml of acetonitrile with 1 % acetic acid was added and vortexed for 1 min. To this, the components of the acetate extraction tube (6.0 g magnesium sulfate +1.5 g sodium acetate) were added, vortexed, and centrifuged at 1000 rpm at 4°C for 15 minutes. Three ml of supernatant acetonitrile layer was transferred into Z-Sep+ dispersive clean up tube, vortexed for 1 min and then centrifuged at 6000 rpm for 10 min. The supernatant was filtered using a 0.4 micron nylon membrane filter and placed in autosampler. The injection volume was 0.2 µl.

RESULTS AND DISCUSSION

The occurrence of chemical contaminants in animal food products is highly possible due to the widespread use of chemicals such as drugs and pesticides in livestock and agricultural practices. The information on the levels of pesticide residues occurring in food commodities is essential and can be obtained through regular monitoring and/or surveillance. Monitoring studies will reveal the status of contamination in the food products and enable us to identify the source of origin and implement control as well as preventive measures.

Many methods including colorimetric and chromatographic methods have been used for the detection of pesticides in animal products. The application of colorimetric and TLC method are limited due to their low sensitivity and mostly they are employed for qualitative screening. Gas chromatography or GC-MS is commonly used for the determination of thermo-labile compounds such as synthetic pyrethroids and N-methyl carbamate pesticides [6]. GC-ECD is the most commonly used detection technique for pyrethroids analyses at low detection limits [7].

In this study, the analytical method for the detection of deltamethrin using Gas Chromatography with an Electron Capture Detector (ECD) was standardized and validated.

We followed QuEChERs protocol for the extraction of deltamethrin from the chicken meat. It involves extraction using solvents followed by clean-up procedures and it can be employed for the extraction of a wide range of pesticides with high extraction efficiency. It is cost-effective, less time-consuming, and more effective compared to time-consuming, laborious, and more complex conventional extraction techniques. The analytical method standardized was validated by studying the limit of detection (LOD), limit of quantification (LOQ), repeatability, and recovery of the method.

The chromatograms of chicken meat blank and chicken meat samples spiked with deltamethrin were compared. It was observed that there were no interfering peaks at the retention time of deltamethrin indicating that the method was highly specific. The peak of deltamethrin was observed at 12.2 min (retention time) (Fig. 1 and Fig. 2).

The method was found to be linear in the range of 0.05 - 0.5 µg/ml and 0.04 to 1.0 µg/g with a correlation coefficient of 0.995 and 0.999 in standard and spiked chicken meat samples, respectively (Fig. 3).

Table 1. Recovery of deltamethrin.

Spiked quantity (µg/g)	Recovery (Mean) (µg/g)	Recovery (%)
0.04	0.03	81.83
0.25	0.24	97.84
0.5	0.48	96.70

Table 2. Precision of deltamethrin assay in the method.

Concentration spiked (µg/g)	Intra-day assay		Inter-day assay	
	RSD	CV (%)	RSD	CV (%)
0.04	0.091	9.103	0.036	3.635
0.25	0.089	8.906	0.013	1.278
0.5	0.070	7.004	0.006	0.561

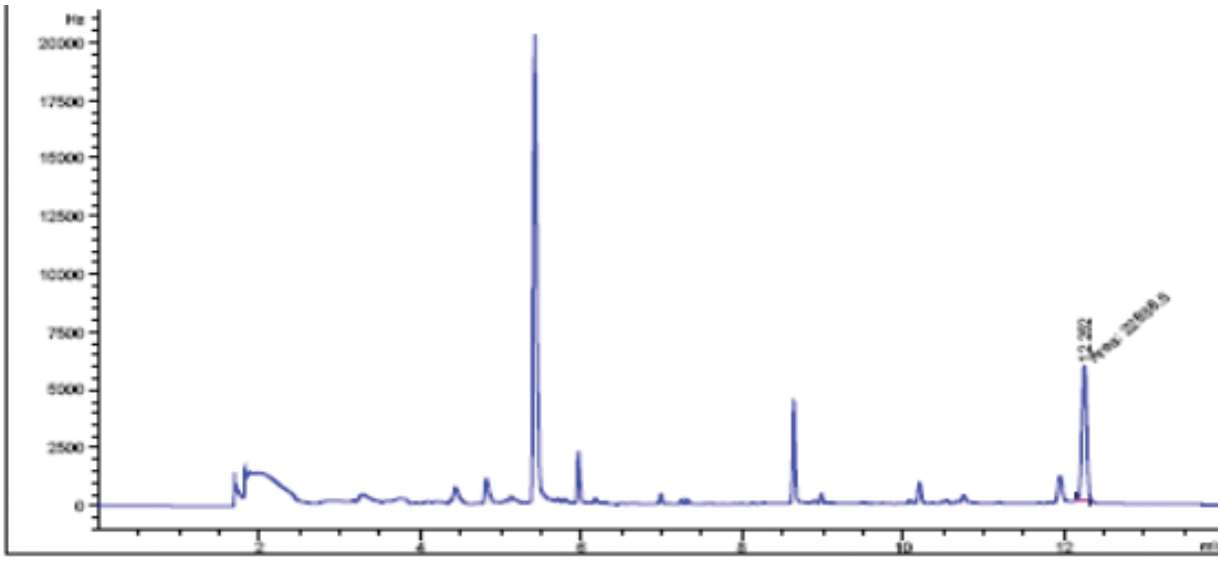

Fig. 1. Chromatogram of deltamethrin (0.5 µg/ml) in ethyl acetate.

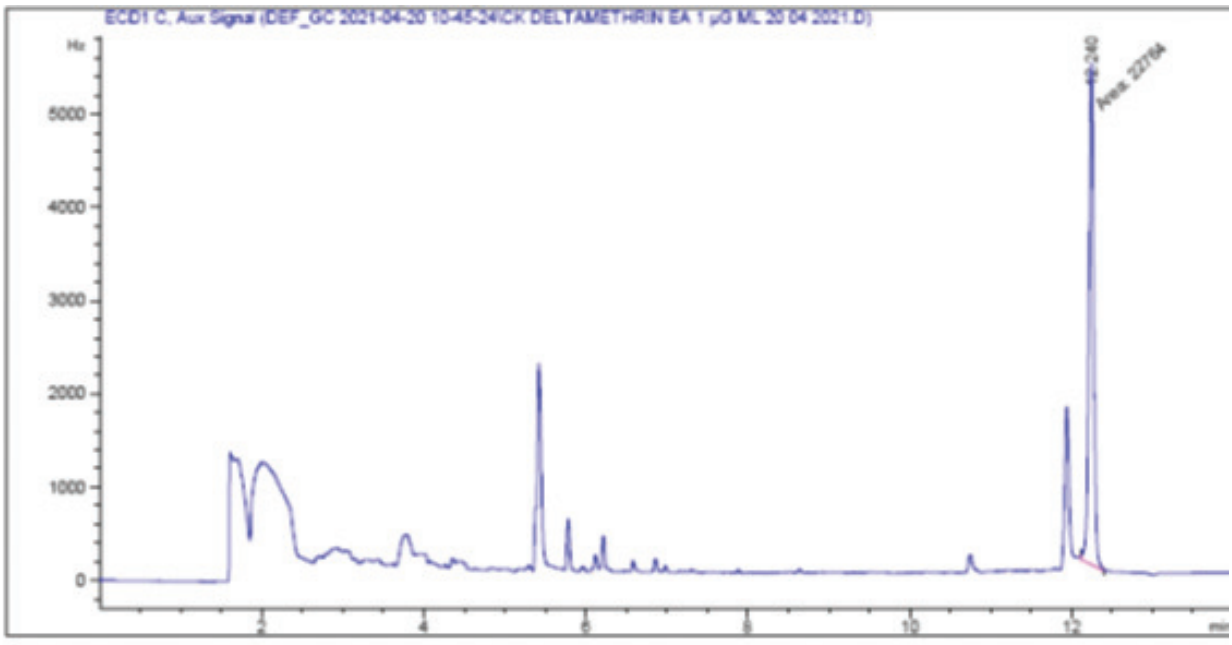

Fig. 2. Chromatogram of deltamethrin (0.5 µg/g) in chicken meat.

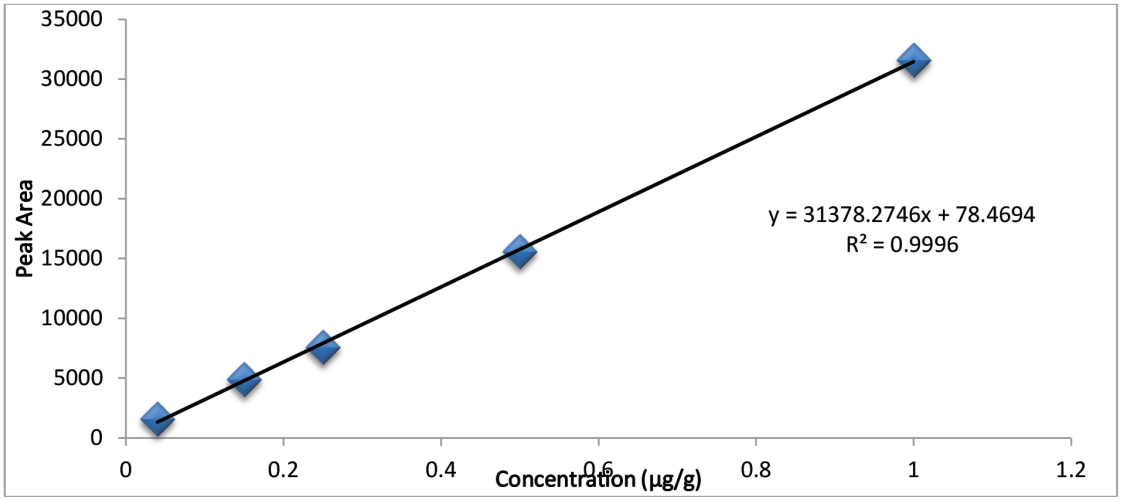

Fig. 3. Calibration curve of deltamethrin in chicken meat.

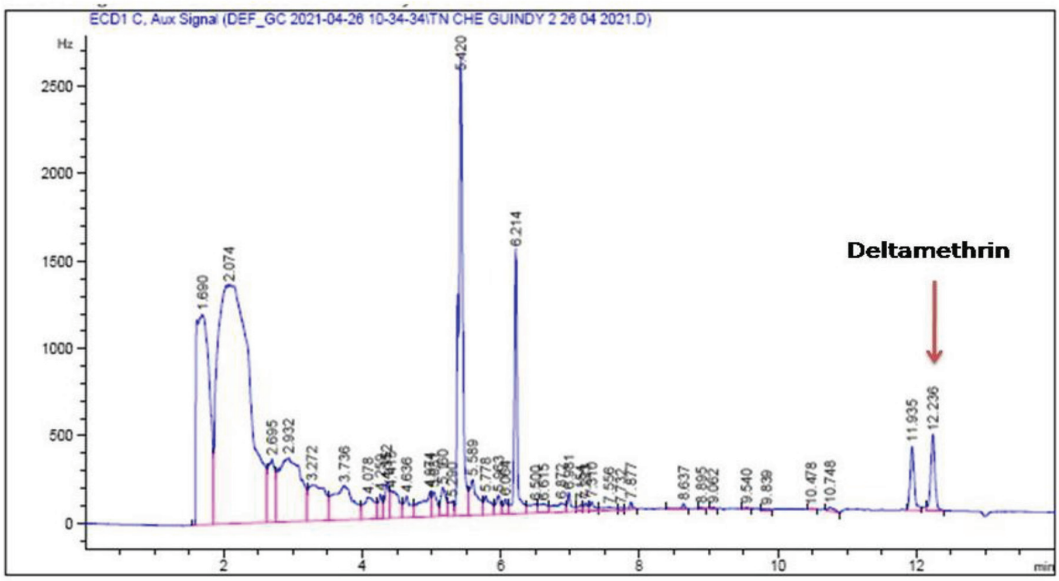

Fig. 4. Chicken meat sample showing deltamethrin residue.

The limit of detection (LOD) and Limit of quantitation (LOQ) were determined using the slope and standard deviation of the calibration curve and they were 0.036 and 0.082 µg/g, respectively. The LOQ of deletmethrin was lower than the MRL for chicken meat which is 0.1 mg/kg (Codex Alimentarius) and 0.5 mg/kg (FSSAI) [8]. It was evident that the LOD and LOQ of deltamethrin were well below the MRL.

After testing at three concentrations, the mean % recovery was found to be in the range of 81.83 - 97.84% (Table 1) and it is well within the acceptable limit (70-120%) of European Commission SANTE/11813/2017 [9].

The repeatability parameters *viz.* intra-day precision and inter-day precision were tested at three different concentrations (0.04, 0.25, and 0.5 µg/g). The method was highly precise with a lower coefficient of variation (less than 10%) (Table 2), This meets the acceptance criteria of CV ≤ 20% as per the European commission SANTE/11813/2017.

The accuracy, as well as precision of the method, was in the acceptable range as per the guidelines. This indicates that the standardized method can be routinely used for the analysis of deltamethrin residues in chicken meat samples.

The analytical method thus validated was used for the analysis of deltamethrin residues in thirty three samples of chicken meat from retail broiler shops in

Chennai, Chengalpattu, Kancheepuram, and Tiruvallur districts of Tamil Nadu. Out of the thirty three samples analyzed, two samples, one each from Kancheepuram and Chennai districts were found to be positive for deltamethrin residues and their concentrations were 0.109 and 0.056 mg/kg, respectively (Fig. 4). The percent of samples positive for deltamethrin residues is 0.06%. The concentration of deltamethrin in one sample was 0.109 mg/kg which was above the MRL level prescribed by Codex Alimentarius Commission (0.1 mg/kg) but it was well within the limits of MRL prescribed by FSSAI, India (0.5 mg/kg). Though the two positive samples contain deltamethrin residues well within the limits of MRL prescribed by FSSAI, India but still it is a matter of public concern.

Though there are strict regulations imposed in India due to public concerns about adverse environmental and human health impacts of pesticides, pesticides are still being used in the agriculture and veterinary sector. As a result of which, there is a high possibility of human exposure to pesticides through residues in food. There is a paucity of information regarding pesticide residues in meat, milk, and egg. Therefore, national wide monitoring programs for screening animal food products for pesticide residues have to be implemented to ascertain the actual residue status in animal food products which will help to locate the source of contamination, and possible violations and to establish legal enforcement to reduce their incidence.

It is important to check the level of pesticide contamination and prevent possible sources of exposure such as feeds and raw materials. Continuous monitoring programs and creating awareness among livestock farmers training are also required to control pesticide residues in animal food products.

CONCLUSION

A method for sensitive detection of for deltamethrin residues in chicken meat is described. The method can be useful for testing laboratories and can conform to the standards prescribed by regulatory authorities. Based on the method, analysis was carried out in thirty three chicken meat samples. The sample analysis revealed positive levels of deltamethrin in two out of thirty three samples.

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