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Research Article

OCCURRENCE OF PESTICIDE RESIDUES IN RANDOM BROILER CHICKEN MEAT SAMPLES

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ABSTRACT

The analysis of organochlorine (OC), pyrethroids and organophosphorous (OP) pesticide residues in broiler chicken meat samples collected from poultry farms of seven districts of Haryana and two districts of Rajasthan was carried out. Instrumental analysis was carried out on a gas chromatograph equipped with an electron capture detector (GC-ECD) for OC and pyrethroids and nitrogen phosphorous detector (GC–NPD) for OP's. Out of 50 samples collected, twelve samples (24%) revealed the presence of OC pesticide residues,10 samples (20%) were found to possess OP pesticide residues, however, all the three pyrethroids tested were not detected in any of the meat samples. Among the 12 samples found positive for OC pesticides, most prevalent OC was HCH which was detected in 9 samples followed by endosulfan in 5 samples. Among different isomers of HCH, highest occurrence was of δ-HCH (14%), followed by γ-HCH (8%), α-HCH (4%) and β-HCH (2%). Whereas for endosulfan, all the three isomers (α, β and endosulfan sulphate) were equally found at rate of 6% each. In case of OP pesticides, monocrotophos was detected in maximum number of samples (8, 16%), followed by chlorpyriphos (5, 10%), dichlorovas and malathion (3 each, 6%) and quinalphos (1, 2%). On comparison with MRL's, 7 samples out of 12 positive for OC's were found to violate MRL's and 5 samples out of 10 positives for OP's were found to violate MRL set by one or the other agency used for comparison.

Key words: Broiler, Meat, Pesticide, Residues

The usage of Organochlorine pesticides (OCPs) is indispensable in the agricultural production technology and control of vector borne diseases in the developing countries. The lipophilic nature, hydrophobicity and low chemical and biological degradation rates of OCPs have led to their widespread accumulation in food chain (John *et al.*, 2001; Bedi *et al.*, 2005; Aulakh *et al.*, 2006) and subsequent magnification of concentrations in human, a final link in the food chain (Surendernath *et al.*, 2000). Pyrethroids are used all over the world to control a wide range of insects in agricultural fields, in greenhouses and in post-harvesting storage; some of them are used in veterinary medicine against some common domestic and farm animal parasites. Organophosphorus (OP) compounds constitute a heterogeneous category of chemicals specifically designed for the control of pests, weeds or plant diseases. Their application is still the most effective and accepted means for the protection of plants from pests, and has contributed significantly to enhanced agricultural productivity and crop yields (Bolognesi, 2003).

Pesticides along with certain environment chemicals are known to cause endocrine disruption by mimicking or antagonizing natural hormones in the body and it has been postulated that their long term, low dose exposure is linked to animal and human health effects such as immunosuppression, hormone disruption, diminished intelligence, reproductive abnormalities and cancer. (Crisp *et al.*, 1998; Hurley *et al.*, 1998; Khurana and Chauhan, 2005).

The facts and figures mentioned above underlines the concern of pesticide residues in poultry meat and its products marketed in India. In this connection, monitoring pesticide residues in poultry meat is one of the most important aspects in minimizing potential hazards to human health (Bagchi *et al.*, 2008). In India especially Haryana, there is paucity of reports related to occurrence of pesticide residues in poultry meat in different districts of Haryana. Therefore, the present investigation was planned with the objective to standardize the gas liquid chromatography (GLC) technique for detection and quantification of pesticides residues of Organochlorines (endosulfan, HCH), Pyrethroids (cyhalothrin, cypermethrin and deltamethrin) and OPs (dichlorovas, monocrotophos, pirimiphos methyl, fenitrothion, malathiopn, chlorpyriphos, quinalphos and edifenphos) as well as their estimation in random poultry meat samples from some parts of Haryana.

MATERIALS AND METHODS

Collection of samples: The present work was carried out in the Department of Veterinary Public Health and Epidemiology, LUVAS, Hisar. The poultry meat of around 250g per birdwas collected from 4-5 different birds of same farm brought for post mortem. They were pooled, minced, mixed well and a representative sample of 250g was taken for analysis of organochlorine, pyrethroid and organophosphate residues. A total of 50 samples were collected from seven districts of Haryana (Hisar, Bhiwani, Fatehabad, Rohtak Sirsa, Sonipat and Jind) and two districts of Rajasthan (Jhunjhunu and Churu). Meat samples collected in transparent polyethylene bags were stored in a deep freezer at -20°C analysis.

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Chemicals and Reagents: All the individual pesticide reference standards of purity >98.0%, aluminium oxide, ethyl acetate, methanol, acetonitrile, calcium silicate, celite 545, acetone and HPLC grade n-hexane were procured from Sigma Aldrich, U.S.A. Petroleum ether (60°C) and anhydrous sodium sulphate were procured from M/s Sisco Research Laboratories.

Preparation of reagents: Reagents were prepared and calibrated as per the instructions given in Pesticide Residues Manual (AMPRF, 1996). Stock solution of each pesticide (primary standard solution) was prepared in n-hexane. For preparation of working standard solutions, the maximum residue limits (MRLs) prescribed by Food Safety and Standard Authority of India (FSSAI, 2011), European Union (EU, 2006), Codex Alimentarius Commission of WHO (Codex, 2006) for all pesticides were considered depending upon their existence. Based on the MRLs, a linearity range (50, 100, 150, 200 and 250 ng/ml) was selected to cover the lowest MRL prescribed. Then appropriate dilutions of secondary standard solution in n-hexane were made to produce a required dilution of working solution.

Analytical Procedure:

Sample extraction and cleanup: Laboratory method for detection of pesticide residues in meat was standardized as per Pesticide Residue Manual (AMPRF, 1996).The poultry meat (10g) samples spiked with pesticide standards or blank samples (unknown meat samples) were mixed and then added with 5 g of sand and 10-20 g of sodium sulphate in a mortar and triturated to obtain free flowing powder. This powder was wrapped in Whatman filter paper No.1 and loaded into especially prepared glass thimbles of extractor of Socs Plus® apparatus and extracted with 150 ml petroleum ether for 2 hours. The extract was then evaporated to dryness in a rotary vacuum evaporator at a bath temperature of 65°C. The amount of extracted fat was determined by gravimetric method and recorded. The residue was then dissolved again in petroleum ether so that the solution contains 50 mg of fat per ml for further cleanup process.

For sample cleanup, 2 ml petroleum ether solution was loaded on the basic aluminium oxide column pre-rinsed with petroleum ether. The column was then eluted using 25 ml petroleum ether. The elute was evaporated to dryness, reconstituted with 10 ml n-hexane and 1 µL of this was injected in the GLC column for chromatographic analysis.

In case of OP, the blank as well as spiked poultry meat (10 g) samples were thoroughly mixed individually and minced for uniform distribution of analytes. Minced sample was mixed with sodium sulphate in a mortar until a free-flowing powder was obtained. To this powder, 5 g celite 545, 10 g calcium silicate, 13 ml acetone and 125 ml acetonitrile were added and blended for 2-3 minutes. The extract was filtered through Whitman's filter paper No.1 (100 mm diameter) over Buchner filter by applying partial vacuum. Filtrate was evaporated to dryness in a rotary vacuum evaporator at a bath temperature of 60-65° C.

The evaporated residue was dissolved in 5 ml acetonitrile saturated beforehand with n-hexane and transferred to separating funnel (60 ml capacity) containing 5 ml n-hexane saturated beforehand with acetonitrile. The funnel was vigorously shaken for two minutes and phases were allowed to separate. The lower (acetonitrile) portion was collected, evaporated to dryness, reconstituted with the 10 ml ethyl acetate and then subjected to gas chromatographic analysis.

Chromatographic Analysis:

Organochlorines, Pyrethroids and Organophosphate pesticides: A Shimadzu gas chromatograph model GC 2010 Plus equipped with autosampler AOC-20i mounted on a split /split less injector port connected to ^{63}Ni electron capture detector through Equity® 5 capillary column (30 m x 0.25 mm I.D.) was used in the study for analysis of both OC and pyrethroid pesticides with following instrumental conditions:

Initial temperature of column was adjusted to 60° C and held for 0.5 min., then temp was raised at the rate of 20° C/min to 204° C, 2° C/min to 208° C, 0.5° C/min to 210° C then 20° C/min to 300° C held for 5 min. The total run time was 23.20 min. Split ratio used was 1:47 with column flow at the rate of 4.0 ml/min. Injector and detector temperature was set to 200° C & 320° C, respectively.

Whereas, a programmed temperature vaporizer (PTV) injection port (manual injection) connected to nitrogen-phosphorous detector through Equity 1 capillary column (30 m x 0.25 mm I.D.) was used for analysis of OP pesticides with instrumental conditions as- initial temperature of column was 100°C with hold for 1 min., and then raised 10°C/min to 200°C without hold, 20° C/min to 260° C with hold for 2 min. The total run time was 16 min, pressure 107.9 kPa, split ratio 1:14, total flow 25 ml/min, column flow 1.47 ml/min, detector temperature was 300°C and injector temperature was 280° C.

RESULTS AND DISCUSSION

Standardization and validation studies of Gas Chromatography technique: GC-ECD and GC-NPD technique was standardized for the extraction of residues of OC/pyrethroidand OP pesticides, respectively from

poultry meat as per the method described by Dutch Ministry of Public Health, Welfare and Sports, the Netherlands (AMPRF, 1996) with slight modifications. Standard mixtures of selected OC, pyrethroid and OP pesticides were prepared separately and validated using following parameters:

i) **System Precision:** The system precision was evaluated by studying the reproducibility of the instrumental response with respect to retention time and area of an analyte. The percent Relative Standard Deviation (% RSD) for all OC, pyrethroids and OP's was found to be less than 0.02 per cent for area, whereas it was 0.009 percent and 0.34 per cent for retention time for OC/pyrethroid and OP's, respectively.

ii) **Specificity:** It was evaluated by visual observation of chromatograms of blank sample matrix and sample matrix spiked with standard mixture. It was found that the chromatographic signals at the retention times of pesticides were absent in blank sample matrix (meat).

iii) **Linearity:** The standard calibration curves of the analysed OC, pyrethroid and pesticides presented a good regression line (r2>0.99 for OC & pyrethroids and r2>0.98 for OP) in the range of explored concentrations i.e. from 50 to 250 hg/ml. The graphs showing calibration curve of these pesticides revealed that all concentrations of the OC, pyrethroid and OP pesticides under study were collinear and thus calibration curves were further employed for the detection of analytes under study.

iv) **Limit of detection and Limit of quantitation:** Table 1 and 2 summaries the LOD and LOQ obtained for each analyte in OC, pyrethroid and OP pesticide group. Perusal of tables clearly indicates that the LOD and LOQ for individual analytes were well below their respective MRLs indicating that the method was able to detect the given pesticide at sufficiently low level.

Table 1
Method performance parameters for detection of residues of OC & Pyrethroid pesticides in poultry meat

Analyte	Limit of Detection (mg/kg)	Limit of Quanti-tation (mg/kg)	Accuracy (Average recovery %)	Precision (Average CV %)
α- HCH	0.002	0.005	80.83	6.76
β- HCH	0.005	0.012	87.04	11.97
γ- HCH	0.002	0.004	90.61	14.76
δ- HCH	0.015	0.041	82.80	6.06
α- Endosulfan	0.005	0.014	84.26	6.74
β- Endosulfan	0.003	0.009	88.90	12.61
Endosulfan sulphate	0.015	0.037	80.19	4.73
λ- Cyhalothrin	0.005	0.015	78.27	7.01
Cypermethrin	0.015	0.041	72.06	8.06
Deltamethrin	0.004	0.012	80.74	6.27

Table 2
Method performance parameters for detection of residues of OP pesticides in poultry meat

Analyte	Limit of Detection (mg/kg)	Limit of Quanti-tation (mg/kg)	Accuracy (Average recovery %)	Precision (Average CV %)
Dichlorovas	0.0029	0.0074	82.90	5.60
Monocrotophos	0.0017	0.0040	84.49	9.65
Pirimiphos Methyl	0.0007	0.0016	91.43	8.43
Fenitrothion	0.0006	0.0015	77.39	6.16
Malathion	0.0016	0.0041	86.14	11.44
Chlorpyriphos	0.0012	0.0030	86.24	4.27
Quinalphos	0.0005	0.0011	83.05	8.32
Edifenphos	0.0011	0.0027	85.71	7.97

v) **Accuracy:** The accuracy in terms of percent recovery of each pesticides in all OC, pyrethroid and OP pesticide group at five different fortification levels (50, 100, 150, 200 and 250 mg/kg) were evaluated and the results are presented in tables 1 & 2. Satisfactory results were found in almost all instances. The analyte-matrix combinations recoveries ranged between 76.56–84.71, 75.35– 81.47 and 81.08-89.98 percent for OC's, pyrethroids and OP's, respectively. However, in general, the pesticides gave acceptable recoveries within the mentioned validation interval as per EU legislation (EU, 2002) between 70 and 110 percent. The chromatographs of standard mixture of OC, pyrethroid and OP pesticides are shown in Fig.1.

vi) **Precision:** The precision was assessed, at five concentration levels (50, 100, 150, 200 and 250 mg/kg) of the recovery studies, by extraction and analysis. Repeatability and intermediate precision values, expressed as relative standard deviation (CV percent) were found <14.76 for OC, <8.06 for pyrethroids and <11.44 for OP (Tables 1 & 2).

Overall the multiresidue method followed for detection and quantification of OC, pyrethroid and OP pesticide residues in chicken was subjected to rigorous validation parameters. The system precision values indicated a good consistency in response by the GC instrument used during present study. A good linearity was noted for standards and spiked tissue samples. Absence of interfering peaks in blank samples indicates good specificity of extraction and clean up method. Accuracy and precision of the method were in accepted range in

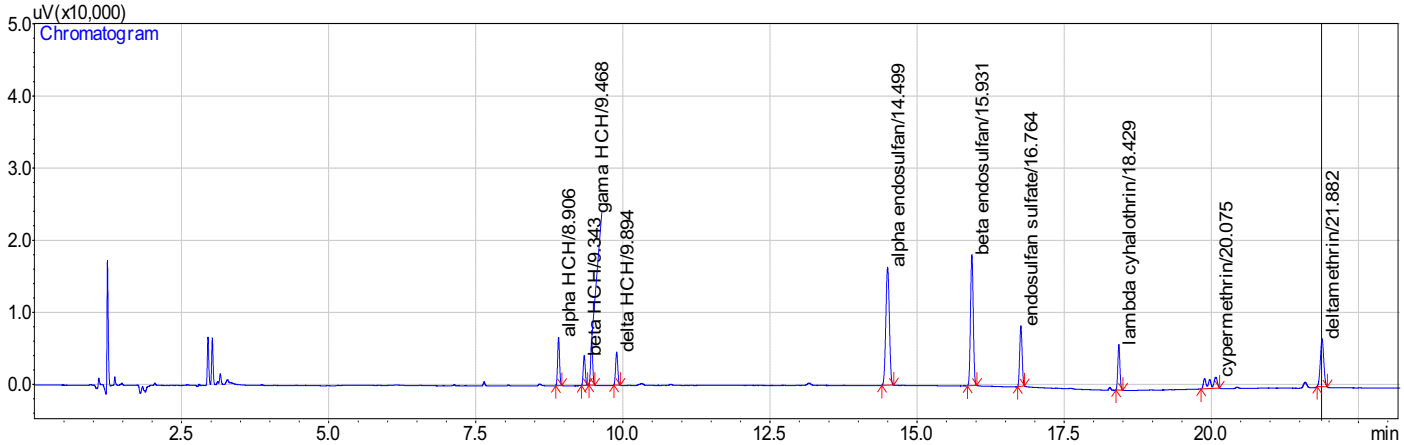

Fig. 1a. Standard mixture of seven OC and three Pyrethroid pesticides in the concentration of 100 hg/ml

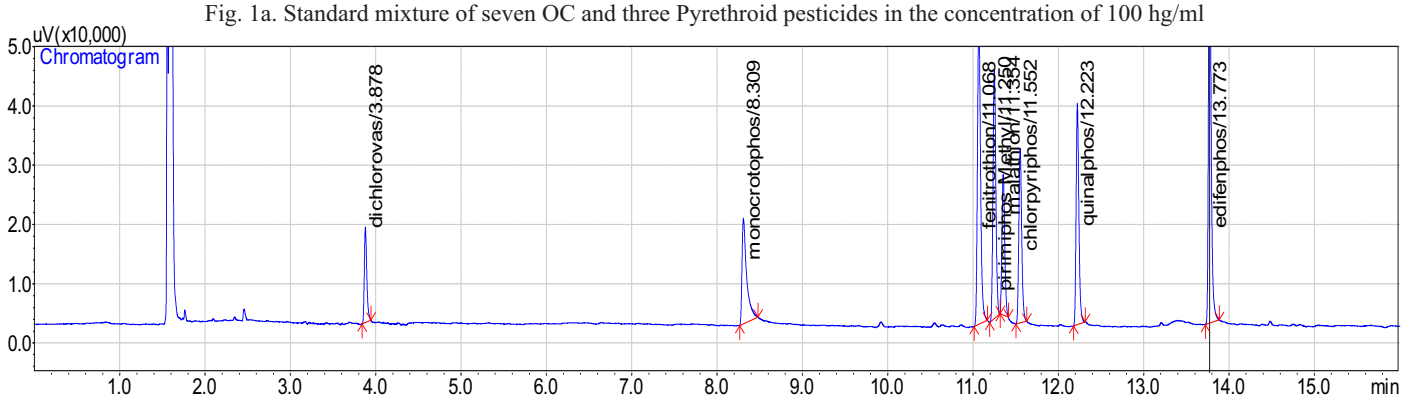

Fig. 1b. Standard mixture of eight OP pesticides in the concentration of 100 hg/ml

Fig. 1: Chromatographs of standard mixture of OC, pyrethroid and OP pesticides

Table 3

Concentration of organochlorine and pyrethroid pesticide residues in random broiler chicken meat samples collected from different poultry farms (in µg/kg)/ppb

Sr. No.	Samples i.d	Total HCH	α-HCH	β-HCH	γ-HCH	δ-HCH	Total Endos-ulfan	α-Endos-ulfan	β-Endos-ulfan	Endosulfan Sulphate	λ-Cyhal-othrin	Cyperm-ethrin	Deltam-ethrin
1	MS–8	+ve	1.396	1.572	1.565	1.447	-	-	-	-	-	-	-
2	MS–10	-	-	-	-	-	+ve	2.184	-	1.243	-	-	-
3	MS–11	+ve	-	-	2.310	-	+ve	-	-	0.721	-	-	-
4	MS–14	-	-	-	-	-	+ve	11.692	10.489	-	-	-	-
5	MS–16	-	-	-	-	-	+ve	2.154	2.245	-	-	-	-
6	MS–27	+ve	-	-	-	7.966	-	-	-	-	-	-	-
7	MS–30	+ve	-	-	-	5.014	-	-	-	-	-	-	-
8	MS–31	+ve	-	-	-	4.577	-	-	-	-	-	-	-
9	MS–35	+ve	-	-	-	1.658	-	-	-	-	-	-	-
10	MS–39	+ve	-	-	-	6.722	-	-	-	-	-	-	-
11	MS–43	+ve	7.816	-	8.195	7.457	-	-	-	-	-	-	-
12	MS–46	+ve	-	-	1.720	-	+ve	-	2.590	3.591	-	-	-
13-50	Rest of all the samples	-	-	-	-	-	-	-	-	-	-	-	-
Total Positive	12 (24.0%)	09 (18.0%)	2 (4.0%)	1 (2.0%)	4 (8.0%)	7 (14.0%)	05 (10.0%)	3 (6.0%)	3 (6.0%)	3 (6.0%)	Nil	Nil	Nil

Note: The mark "-" indicates that level of pesticides were below detectable limit

comparison with international guidelines. These results of validation study clearly demonstrated that the present method is suited for routine analysis of OC, pyrethroid and OP pesticides in poultry meat samples.

Determination of residues of OC, pyrethroids and OP in random meat samples: After successful standardization and validation of techniques for detection of OC, pyrethroid and OP pesticide residues, the extraction,

Table 4
Concentration of organophosphate pesticide residues in random broiler chicken meat samples collected from different poultry farms (in µg/kg)/ppb

Sr. No.	Sample i.d	Dichlorovas	Monocrotophos	Pirimiphos Methyl	Fenitrothion	Malathion	Chlorpyriphos	Quinalphos	Edifenphos
1	MS – 1	-	4.105	-	-	9.876	11.668	6.266	-
2	MS – 2	-	3.332	-	-	4.951	12.121	-	-
3	MS – 6	-	-	-	-	3.192	2.931	-	-
4	MS – 14	-	2.055	-	-	-	-	-	-
5	MS – 15	-	11.813	-	-	-	-	-	-
6	MS – 23	1.388	4.101	-	-	-	-	-	-
7	MS – 26	-	-	-	-	-	7.819	-	-
8	MS – 29	-	7.061	-	-	-	-	-	-
9	MS – 43	4.291	7.159	-	-	-	3.578	-	-
10	MS – 46	12.012	5.351	-	-	-	-	-	-
11	Rest of all the forty samples	-	-	-	-	-	-	-	-
Total Positive	10 (20.00%)	3 (6.0%)	8 (16.0%)	Nil	Nil	3 (6.0%)	5 (10.0%)	1 (2.0%)	Nil

Note: The mark "-" indicates that level of pesticides were below detectable limit

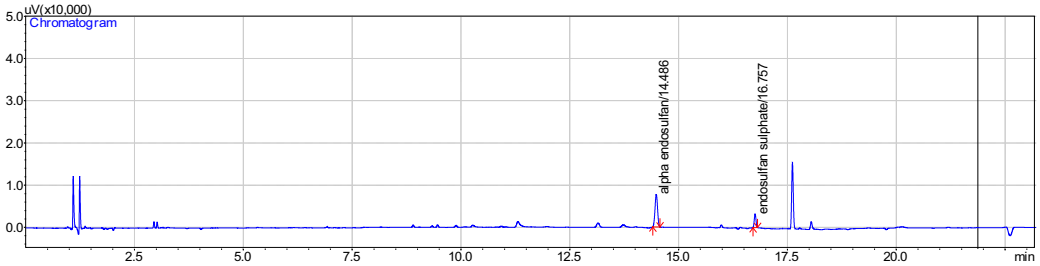

Fig. 2a. MS- 10, showing presence of OC pesticides

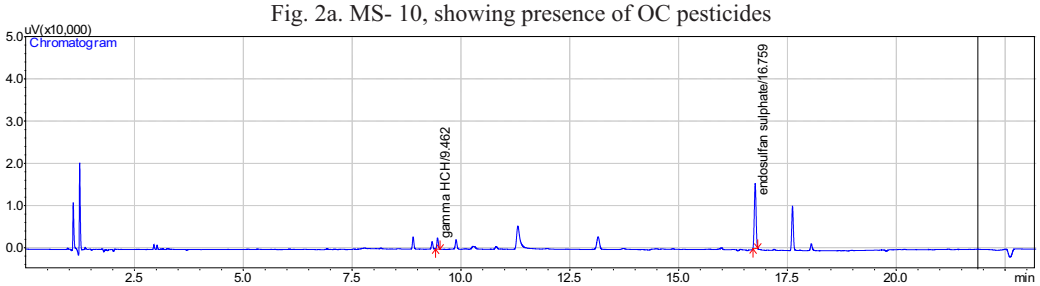

Fig. 2b. MS- 11, showing presence of OC pesticides

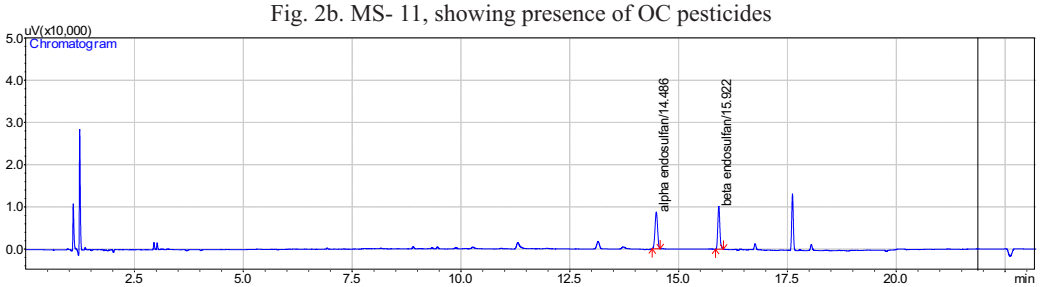

Fig. 2c. MS- 14, showing presence of OC pesticides

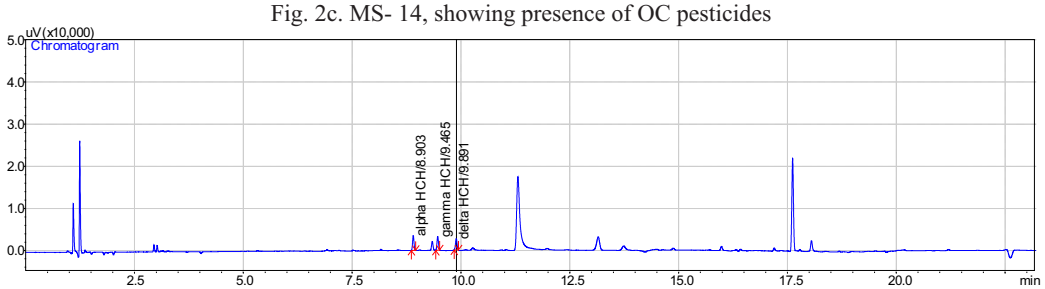

Fig. 2d. MS- 43, showing presence of OC pesticides

Fig. 2 (2a to 2d). Chromatograms of random broiler chicken meat samples showing presence of OC pesticides

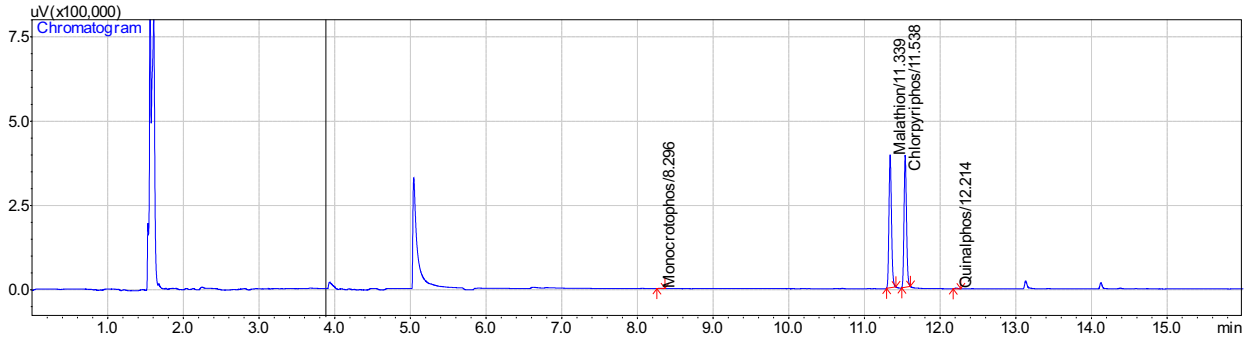

Fig. 3a. Sample MS-1, showing presence of OP pesticides

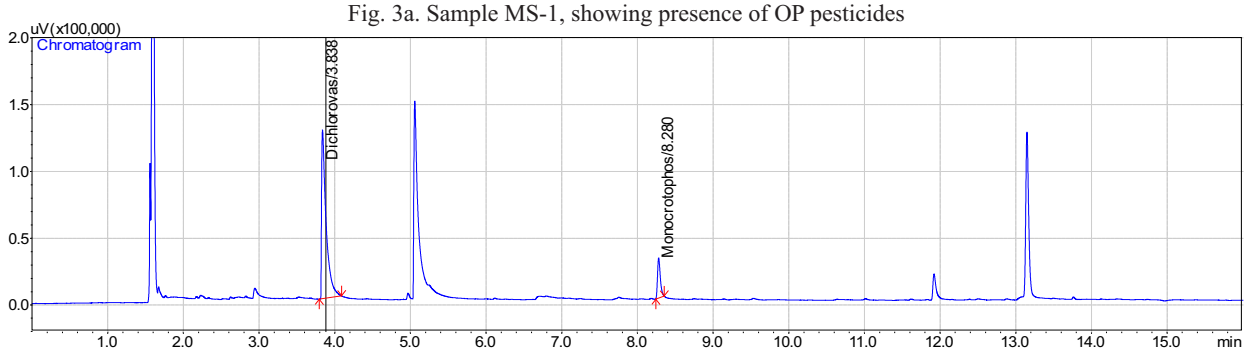

Fig. 3b. Sample MS-23, showing presence of OP pesticides

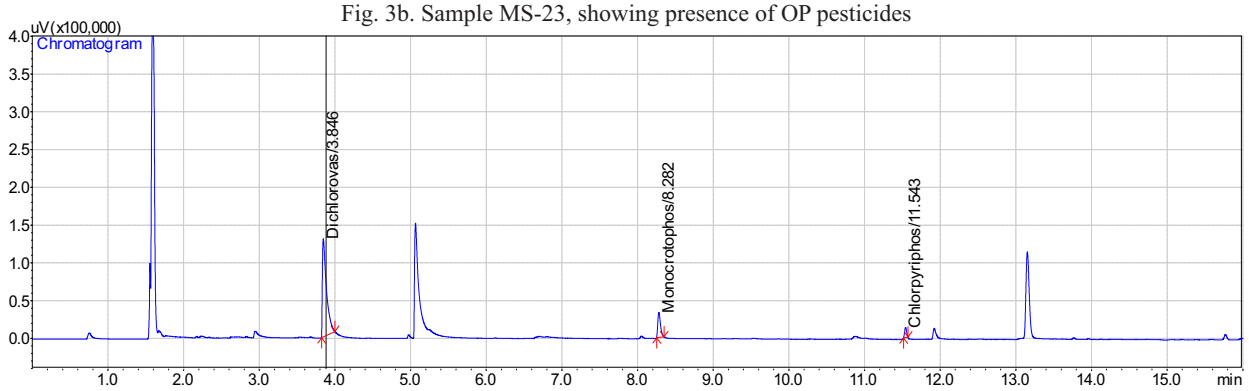

Fig. 3c. Sample MS-43, showing presence of OP pesticides

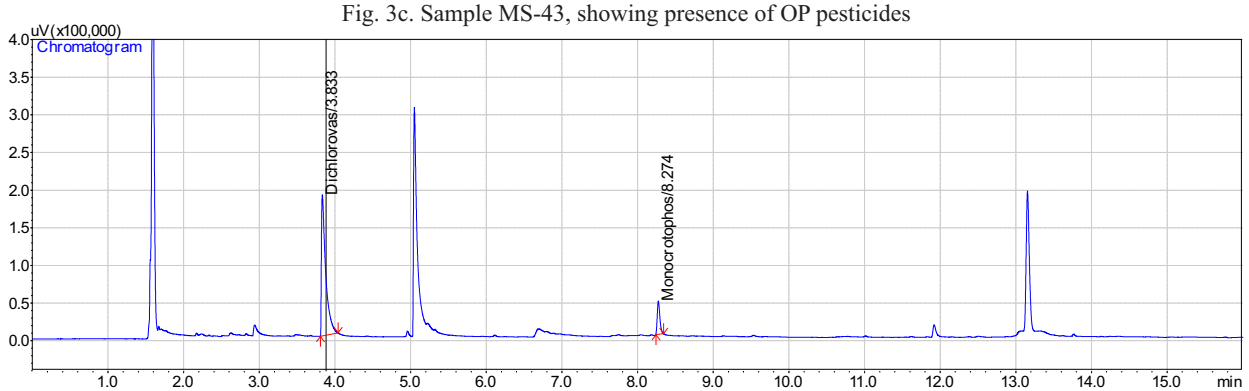

Fig. 3d. Sample MS-46, showing presence of OP pesticides

Fig. 3 (3a to 3d). Chromatograms of randomly collected broiler chicken meat samples showing presence of OP pesticides

detection and quantification were carried out on 50 samples of broiler meat collected randomly from DI lab brought by poultry farmers from nearby places of Hisar. The results obtained are enumerated in the table 3 & 4. The chromatographs of meat samples found positive for OC and OP pesticides are shown in Fig. 2 & Fig. 3, respectively.

Out of the 50 meat samples, 12 samples (24%) revealed the presence of OC pesticide residues (Table 3), however all the three pyrethroids (λ-cyhalothrin, cypermethrin and deltamethrin) were not detected in any of the sample. Most prevalent OC was HCH found positive in 9 samples with prevalence of 18% followed by endosulfan found positive in 5 samples with prevalence of 10%.

Among different isomers of HCH highest occurrence was of, δ-HCH (14%), followed by γ-HCH (8%), α-HCH (4%) and β-HCH (2%), whereas for endosulfan all the three isomers (α, β and endosulfan sulphate) were found to be equally occurring with prevalence rate of 6% each. On comparison with MRL's, 7 out of 12 positive samples were found to violate MRL set by one or the other agency used for comparison. Endosulfan and lindane (HCH) were found to violate MRL set forth by Codex and EU. α-HCH violated the MRL's set forth by EU in both the samples found positive and β-HCH violated the MRL in one of the sample found positive.

In case of OP pesticides, out of 50 samples, 10 samples (20%) were found to possess OP pesticide residues. Table 4 shows the details along with concentrations of OP pesticides in all the 50 samples. Overall prevalence amongst total 50 samples indicated that monocrotphos was detected in maximum number of samples (8.0, 16%) followed by 10% samples positive for chlorpyriphos, 6% each positive for dichlorovas and malathion and the least 2% positive for quinalphos. The pirimiphos methyl, fenitrothion and edifenphos were not detected in any of the samples. Out of 10 positive samples, 5 were found to violate MRL set by one or other agency used for comparison. Chlorpyriphos was found to be violating the MRL's in all the 5 samples (as per FSSAI, Codex and EU). While malathion violated MRL in all the 3 samples as per EU and dichlorovas was found to violate the MRL in one sample out of three positive samples as per Codex. The monocrotophos detected in 8 samples were found to be within MRL values in all the samples. The pirimiphos methyl, fenitrothion and edifenphos were not detected in any of the samples and for quinalphos, MRL was not available in any of the agencies. Further studies are required to strengthen the data obtained by our study.

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