



MULTI PESTICIDE RESIDUES AND OTHER ORGANIC CONTAMINANTS IN LOCAL MEAT CONSUMED IN ASSIUT MARKETS

Eman E. Elsharkawy^a, Ahmed A. Sharkawy^a, Wafaa A. Aly^b

a- Department of Forensic Medicine and Toxicology Faculty of Veterinary Medicine, Assiut University, Egypt

b- Quarantine Veterinarian in Veterinary Quarantine Department of Assiut Airport

ABSTRACT

The present study was carried out as market- basket survey with the aim of determining the concentration levels of some toxic chemical residues in raw local meat sold in Assiut city, Egypt and to assess the potential health risk associated with exposure to these residues from meat consumption. In this work, we tried to compare the situation of OCs, OPs, herbicides and PCBs residues levels in the domestic meat. The study investigated a total of 100 raw meat samples were randomly collected from meat markets in Assiut city. Samples were collected along the year from January to December 2013 from units available in retail stores in Assiut city. Meat samples were subjected to multi residues determination using gas chromatography–mass spectrometry GC/MS determination and analysis. Residues of different pesticides and PCBs were compared with MRLs of different international regulations. Results revealed that OCs detection which include aldrin, p,p'-DDE, lindane, heptachlor epoxide, dieldrin and methoxychlor. The OPs detection includes malathion, parathion-ethyl, parathion-methyl, ethion, chlorpyrifos, diazinon, disulfoton. The only congener of PCBs detected in local meat was hexachlorobiphenyl (PCB 138) with a mean value $4.90 \pm 3.10 \mu\text{g/Kg}$. Its frequency distribution was 33% of samples. This level of residue is lower than the permissible limit. In the present study, the only herbicide residue detected in the examined meat samples was trifluralin. It was present in 67% of examined meat samples. These residue levels were below the permissible limit.

Keywords: *pesticides; herbicides; polychlorinated biphenyles; residues; domestic meat.*

INTRODUCTION

Environmental pollution is considered as one of the most serious problems in the world. The deleterious effect of the environmental pollution by pesticides and other chemicals has been considered as one of the principal research activities (David et al., 2008). Pesticides were mainly used in agriculture. They have high toxic effects, persistence in the environment and posing considerable hazards (Aboul-Enein et al., 2010). They are very resistant to microbial degradation and employed to control ecto- parasites of farm animals and pets. They may concentrate in the adipose tissues and in the blood serum of animals leading to environmental persistence, bioconcentration and biomagnification through the food chain (Ntow et al., 2001). Insecticides, such as the organophosphates and synthetic pyrethroids are common agricultural chemicals associated with meat production, and are used as grain protectants and pest control in pastures and feeds. Herbicides are used to reduce weeds and spraying is common in pastures and in pre-pasture establishment (FSANZ, 2013). The wide

spread usage of pesticides in Egypt led to many problems and constituted hazard in animals. In recent years pesticides in food arises as an important problem of serious public health hazards which may lead to acute or chronic hepatic toxicity for human being (Hassouba et al., 2007).

To protect consumer health, national and international programs have been established to monitor levels of pesticide and PCBs residues present in domestic and imported food and fodder to prevent the marketing of food containing residues that either exceed specific levels (known as tolerances) set by the Environmental Protection Agency or for which no tolerances have been established for that food (OTA, 1988). The aim of the present study is to determine the levels of residues of toxic chemicals associated with the meat supply chain as multi-pesticide residues and other organic contaminants of local meat consumed in Egyptian markets., and to assess the safety risks associated with these chemicals by comparing the results with Maximum Residual Limits (MRLs) established by the international health regulatory agencies.

2. MATERIAL AND METHODS

2.1. Sample collection

A total of 100 raw fresh buffalo meat samples were randomly collected. Samples were collected along the year from January to December 2013 from units available in retail stores in Assiut city, south of Egypt. There was no repetition of batches. Samples were designed as the following: 100 samples from local balady boneless raw meat. These samples of local meat which originated from animals raised and slaughtered in Egypt, and we purchased them from meat markets in Assiut city. The average chemical compound residue concentrations \pm SE were calculated in local meat (n = 5 pooled samples). The meat samples were collected from different areas in Assiut city, Upper Egypt. All tested samples were labeled and stored at -20oC. Samples were homogenized immediately before analysis and the minimum weight used was 50 g.

2.2. Reagent and materials

2.2.1. The standards used in the experiment

Pesticide standards were purchased from Sigma (Poole, UK). The polychlorinated biphenyls (IUPAC Nos. 28, 52, 118 and 138) were from LGC Standards (Teddington, UK). The standards used in the experiment to perform the multi residual detection of toxic chemical in meat were as the following Table 1.

Table 1: The standards used in the experiment

(OCs)	(OPs)	PCBs	Herbicide
Aldrin	Malathion	PCB 28	Trifluralin
p,p'-DDE	Parathion-Ethyl	PCB 52	Alachlor
Lindane	Parathion-Methyl	PCB 118	Atrazine
Hexachlorobenzene (HCB)	Ethion	PCB 138	Propachlor
Heptachlor		PCB 180	Simazine
Heptachlor epoxide	Chlorpyrifos		
Dieldrin	Diazinon		
Methoxychlor	Disulfoton		

2.2.2. Chemical reagents

N-hexane, methyl alcohol, acetone (GC grade or equivalent), anhydrous sodium sulphate (Na₂SO₄) of analytical reagent grade (it was heated at 650 °C for 4 h and kept in a desiccator), cyclohexane. All chemicals and reagents were purchased from Sigma (USA). GPC (gel permeation chromatography) column and PSA (ethylene diamine-N-propylsilylated silica gel chromatography) column [Bond Elut PSA (500 mg)] were purchased from Agilent Technologies, Inc.

2.3 Analytical procedures

According to JAPANESE POSITIVE LIST SYSTEM (2006)

2.3.1. Extraction

20.0 g of meat sample was weighed out and homogenized with 20 ml water. 100 mL of acetone/n-hexane (1:2) was added and performed homogenization again. Then the sample was centrifuged at 2500 rpm for 5 minutes. Organic layer was collected. 50 ml of n-hexane was added to the remaining layer. Perform homogenize the extract and centrifuge for separation at 2500 rpm for 5 minutes. The resulting organic layer was collected and added to the first organic layer. The organic layer was dried over sodium sulfate (anhydrous) and filtered. The filtrate was concentrated to dryness at 40°C or lower. The residue weighed and recorded as an extracted fat weight. Then dissolved in acetone/cyclohexane (1:4).

2.3.2. Cleanup

Gel permeation chromatography (GPC)

The solution obtained from the extraction step was centrifuged at 3000 rpm for 5 minutes, and then collected the resulting supernatant. 5 ml of the supernatant was applied to a column for gel permeation chromatography (styrene-divinylbenzene copolymer column). Column was eluted with acetone/cyclohexane (1:4), and collected a volume of solution eluted from the retention time of acrinathrin to the finish time of tricyclazole elution. Concentration of the effluent to dryness at 40°C or lower was done. Then the residue was dissolved in 2 mL of acetone/n-hexane (1:1).

Ethylene diamine-N-propylsilylated silica gel chromatography column (PSA)

The PSA column (500 mg) was conditioned with 10 ml of acetone/n-hexane (1:1). Then applied the solution obtained from the gel permeation chromatography step above. The column was eluted with 20 ml of acetone/n-hexane (1:1). The entire volume of effluent was collected and concentrated to dryness at 40°C or lower. The residue was dissolved in acetone/n-hexane (1:1) to make a 1 ml solution (0.5 ml solution for fat), and then it used as the test solution.

2.3.3. Preparation of standard stock solution

Individual stock standard solutions were prepared by dissolving 10 mg of each compound in 10 ml methanol and stored in amber bottles. A mixed standard solution was prepared from the individual stock solutions with a concentration of 100 mg/L. A series of calibration standards were prepared by diluting 100 mg/L of the mixed standard solution to produce final concentrations of 0.1, 0.2, 0.5, 1.0, and 2.0 mg/L in methanol. Stock and working solutions were stored at 4 °C and used for no longer than 3 months and 1 week, respectively.

2.3.4. Gas chromatograph/mass spectrometer (GC/ MS: 7890A- 5975B)

Analysis was performed using an Agilent Technologies 7890A Gas chromatography equipped with a 5975B Mass spectrometer. Column J&W 122- 7732DB- 1701P, 30 m x 250 μm x 0.25 μm . Carrier gas is helium at flow rate of 1 mL/min. Oven temperature program is 90 °C for 4 min then 30 °C/min to 180 °C for 0 min then 4 °C/min to 270 °C for 0 min then 30 °C/min to 280 °C for 7 min. Inlet Splitless (Front SS Inlet He) and the inlet heater 200°C with injected volume 1 μL . Maximum temperature is 280 °C. The acquisition mode used is SIM with a solvent delay is 7.50 min, tune file tune- f2.U and EMV mode gain factor resulting EM voltage 1741. All samples were treated and analyzed using GC–MS–SIM mode (selected ion monitoring) not scan mode. The procedures were done by ACAL, ANALYTICAL CHEMISTRY UNIT, Department of Chemistry Annex (B), Faculty of Science, Assiut University, Assiut, Egypt.

2.4. Validation of the method

The recovery analysis and the validation of the extraction method were carried out at fortification average levels of 0.02, 0.05, and 0.1 mg/kg, by adding a standard solution to untreated meat samples. Samples were allowed to equilibrate for 30 min prior to extraction, and were processed according to the procedure described above. The recovery assays were replicated five times. All the samples were treated and analyzed using the GC–MS–SIM mode described above. The average rate of the detection limit (LOD) was 96% and the average recovery percent of each tested residues was 99%, indicating that the analytical process was efficient.

Statistical analyses

A value of zero was assigned for results below the limit of detection. The Kruskal – Wallis test was used to verify that all data was normally distributed. The data were expressed as mean \pm standard error (SE). All statistical analysis was done using the statistical package for social sciences (SPSS 16.0) program.

RESULTS

OCs pesticide residues

In meat samples, the OCs detection includes aldrin, p,p'-DDE, lindane, heptachlor epoxide, dieldrin and methoxychlor. Both of HCB and heptachlor were not detected. The mean values of these pesticides were 1.69 ± 0.91 , 1.00 ± 0.79 , 14.26 ± 7.47 , 30.22 ± 10.39 , 5.78 ± 3.80 and 0.39 ± 0.33 $\mu\text{g}/\text{Kg}$, respectively (Table (3) and Fig. (1). In comparison with the MRLs, we found that OCs residues in local meat samples weren't exceeded the permissible limits (Table (6) set by different regulations, especially the lowest one EU, (2008). Each of aldrin, and p,p'-DDE was detected in 43% of examined samples while lindane and heptachlor -epoxide were detected in most of samples in about 83% whereas the dieldrin and methoxychlor were detected in 33% of samples (Fig (2).

OPs pesticide residues

In local meat, the OPs detection includes malathion, parathion-ethyl, parathion-methyl, ethion, chlorpyrifos, diazinon, disulfoton. The mean values of these pesticides were 0.56 ± 0.36 , 1.25 ± 0.79 , 94.15 ± 44.22 , 37.53 ± 24.43 , 9.67 ± 8.13 , 111.7 ± 35.98 and 2.20 ± 1.88 $\mu\text{g}/\text{Kg}$, respectively (Table 4 and Fig. (1). It was found that parathion-methyl, ethion and diazinon exceeded the permissible limits set by EU,

(2011, 2012 and 2013) regulations (Table (7)). As the lowest permissible limits of EU are 10, 10 and 20 µg/Kg respectively where the samples had 94.15±44.22, 37.53±24.43 and 111.7±35.98 µg/Kg, respectively. The most dominated pesticide was parathion-methyl which was detected in 100% of samples then followed by diazinon which was detected in 83% of samples. Each of malathion, parathion-ethyl and ethion was detected in 33% of samples while chlorpyrifos and disulfoton were detected in 43% of samples (Fig (3)).

PCBs and trifluralin residues

In the examined meat samples, the only congener of PCBs detected was PCB 138 with a mean value 4.90±3.10 µg/Kg (Table 5 and Fig 1). Its frequency distribution was 33% of samples Fig. (4), this level of residue is lower than the permissible limit set by EU commission, (2011) (Table (8)). The only herbicide residues found in 67% of meat samples was trifluralin Fig. (4). Its average concentration was 0.21± 0.08 µg/Kg (Table 5 and Fig. 1), which is below the permissible limit set by EU commission, (2012) (Table 8).

DISCUSSION

In domestic examined meat samples, the OCs detection includes aldrin, p,p'-DDE, lindane, heptachlor epoxide, dieldrin and methoxychlor. Both of HCB and heptachlor were not detected. The mean values of these pesticides were 1.69±0.91, 1.00±0.79, 14.26±7.47, 30.22±10.39, 5.78±3.80 and 0.39±0.33 µg/Kg, respectively. In comparison with the MRLs, we found that OCs residues in local meat samples weren't exceeded the permissible limits set by different regulations. Each of aldrin, and p,p'-DDE was detected in 43% of examined samples while lindane and heptachlor -epoxide were detected in most of samples in about 83% whereas the dieldrin and methoxychlor were detected in 33% of samples. In Egypt, an investigation dealt with the gas liquid chromatographic analysis of meat and fat samples from cows and buffalos for determination of residues of OCs pesticides. The results revealed that total HCH was 16.9, 647.36 and 4.06, 303.08 ppb for meat and fat samples of cows and buffalos respectively. For DDT, the values were 7.8, 266.5 and 8.87, 998.43 ppb; for heptachlor and heptachlor epoxide, they were 1.76, 39.8 and 0.83 and 41.88 ppb, for dieldrin and aldrin, the values were 10.16, 161.67 and 1.77, 54.4 ppb respectively (Mustafa, 1992). The levels in our meat samples were lower than these levels for lindane and DDT while they were higher than the levels of heptachlor -epoxide and dieldrin. Resembling to our data Khalafalla et al. (1993) could detect aldrin, dieldrin, lindane and DDT in fat samples of slaughtered animals in Egypt. None of examined samples exceeded the permissible limits of DDT, while beef muscle and fat as well as buffalo fat showed high level of lindane residue. In earlier study conducted in Assiut, pesticide residues studied in 164 tissue samples (120 fat, 20 liver and 24 muscle samples) collected from Assiut Governorate during March - April 1992 by GC-ECD. Liver and muscle samples were contained negligible amounts of OCs pesticides contained relatively higher amounts that rarely exceeded the permissible limits. DDT derivatives (p, p-DDT, 0, p-DDD and p, p-DDE), total HCH isomers(alpha-, beta-, gamma- and delta-isomers) and HCB were the most frequent pesticides, followed by aldrin and dieldrin, endrin and heptachlor and heptachlor epoxide. Only one buffalo fat sample (1.3%) exceeded for endrin released by the Codex Committee on pesticide Residue (CCPR) of FAO/WHO in 1990 (Salem et al., 1998). In another survey in Egypt, there were about 77 beef carcasses and 82 buffalo carcasses were contaminated with OCs pesticides. The main OCs pesticides could be detected in the tissues of beef and buffalo carcasses were DDT, aldrin and dieldrin

with variable levels (Gad-Allah, 1999). In recent studies performed in Egypt by Sallam et al. (2008) determined OCs pesticide in a total of 270 meat samples; comprising the muscle, liver, and kidney collected from 90 carcasses (30 each camel, cattle and sheep) slaughtered in Sharkia Province. All samples were analyzed for their residual content of DDT and isomers (HCHs), lindane (γ - HCH), aldrin, dieldrin, endrin, HCB, toxaphene, and chlordane compounds. The results indicated that 54.4% (49/90), 51.1% (46/90), 47.8% (43/90), 44.4% (40/90), 33.3% (30/90) and 15.6% (14/90) of the examined carcasses were contaminated with DDTs, HCHs, lindane, aldrin, dieldrin and endrin, respectively. The other contaminants (HCB, toxaphene, and chlordane) were only present in less than 10% of the analyzed carcasses. These frequencies of distribution are similar to the data obtained in the present study for aldrin (43%) and dieldrin (33%) where they lower for lindane (83%) and higher for DDT(43%). As well as by Aboul-Enein et al. (2010), they documented that all of locally raised bovine samples contained residues of OCs compounds. Aldrin, endosulfan II, dieldrin and p,p, DDT was the most frequently found pesticides, while endrin was the least frequent one. These findings are similar to our results, where no sample exceeded the maximum residue limits given by EC and FAO/WHO (Codex alimentarius) except for endosulfan was not detected in the present study. In Jordan, The results indicated that 49% of the examined meat samples were contaminated with OCs residues and this level was lower than the percentage obtained in the present study where the presence of OCs reached to 53%. Moreover, they detected 4.6% of meat contaminated samples showed concentration above the MRLs of the FAO/WHO (2006), while our results not showed any OCs residues above MRL of different regulations. So the data in this study are surprising and raise questions about pesticides banned for more than 20 years in Jordan (Ahmed et al., 2010). In several studies with resemble results to our study and also from some countries described as developing countries as our country Egypt. In Croatia domestic meat, the levels of pollution with the compounds studied were considerably lower than in imported meats. The differences were most noticeable in lindane and DDT levels in beef and those of DDT in pork. The average level of lindane in domestic and imported beef was 0.004 and 0.020 mg/kg, respectively. Domestic beef contained on the average 0.013 mg/kg and the imported beef 0.059 mg/kg DDT, respectively. While the average amount of DDT in local pork was 0.014 mg/kg, the average for imported pork was 0.041 mg/kg (Kipčić et al., 2002). In Ghana, meat from Kumasi and Buoho abattoirs were analyzed for OCs pesticide. Lindane was detected in 40 % of the fat from Kumasi. The average concentration of lindane in beef fat from Kumasi was 4.04 μ g/kg. This level is below the WHO limit of 6.0 μ g/kg. Aldrin occurred in 30 % of the samples with mean value of 2.06 \pm 1.48 μ g/kg. Endosulfan was detected in 90 % of the samples. The mean endosulfan in beef fat in Kumasi was 21.35 \pm 3.85 μ g/kg. The highest level of endosulfan detected was 105.58 μ g/kg. DDT and, its metabolite, DDE were detected in all the fat samples analyzed at a mean concentration of 545.24 \pm 247.06 μ g/kg. This sample contained the highest amount of DDT of all the samples analyzed (Darko and Acquah, 2007). Residue levels of OCs pesticides in a total of 90 cattle samples comprising meat, liver and kidney collected from carcasses slaughtered in six towns in West Shoa Zone, Ethiopia. The concentration level of the studied OCs followed the order: (DDT) > endosulfan > o,p'-DDT >lindane>dieldrin>endrin>aldrin>chlorothanolin while the order of contamination in the analyzed organs was liver > kidney > meat (Letta and Attah, 2013). Although the residual contents of the OCs detected in all the contaminated samples analyzed from the three previous studies (Croatia, Ghana and

Ethiopia), they were below the respective maximal permissible levels set by international organizations, and these findings are similar to our results in this study. The results from this study showed that residues of OCs were presented at concentrations below but close to the EC (2008) MRLs which represent the low MRL between the different organization and also Egypt follow their direction in the meat samples. These pesticides might have originated from the feed of the cattle or from pesticides used as 'dip' to control ectoparasites on the cattle or from maternal transfer through the breast milk. Education, training and information activities on OCs pesticides and their residues should be established and strengthened.

In the examined meat, the OPs detection includes malathion, parathion-ethyl, parathion-methyl, ethion, chlorpyrifos, diazinon, disulfoton. The mean values of these pesticides were 0.56 ± 0.36 , 1.25 ± 0.79 , 94.15 ± 44.22 , 37.53 ± 24.43 , 9.67 ± 8.13 , 111.7 ± 35.98 and 2.20 ± 1.88 $\mu\text{g}/\text{Kg}$, respectively. It was found that parathion-methyl, ethion and diazinon exceeded the permissible limits set by EU (2011, 2012 and 2013) regulations. As the lowest permissible limits of EU are 10, 10 and $20\mu\text{g}/\text{Kg}$, respectively where the samples had 94.15 ± 44.22 , 37.53 ± 24.43 and 111.7 ± 35.98 $\mu\text{g}/\text{Kg}$, respectively. The most dominated pesticide was parathion-methyl which was detected in 100% of samples then followed by diazinon which was detected in 83% of samples. Each of malathion, parathion-ethyl and ethion was detected in 33% of samples while chlorpyrifos and disulfoton were detected in 43% of samples. Similarly, in locally raised bovine meat OPs pesticides could be detected. Ethoprophos, cadusafos, chlorpyrifos and azinphos-methyl were the most frequently found. It was recorded that 20% of samples which obtained from Badrashen contained residues of ethoprophos and had exceeded the maximum residue limits. For azinphos-methyl, only one sample contained residues of this pesticide, obtained from Giza, and had exceeded the maximum residue limits (Aboul-Enein et al., 2010). In Tanta, Egypt, Heikal (2008) could detect diazinon in liver, kidney of cattle and camel. He recorded that diazinon residues in 26, 13, 20 and 6% respectively of the examined samples were exceeded the MRL according to EOS, (1991). In a recent study conducted on raw buffalo milk samples collected from Assiut city by Shaker and Elsharkawy (2015), three OPs pesticides chlorpyrifos, malathion, and parathion-methyl were detected in the milk samples. The concentrations of malathion and chlorpyrifos residues exceeded the 2008 EC maximum residual limits (MRLs) by 44 and 33 % of the examined samples, respectively. Although, OPs pesticides are regarded as being low persistent compared with OCs, but some reports have indicated that residues of OPs are persisting for extended periods in organic soil and surrounding drainage systems in Egypt by Abdel-Halim et al. (2006). Due to their low price and effective ability to control pests, weeds and diseases (He et al., 2009), they had been widely used and became more and more important in agricultural production after OCs were forbidden. OPs residues can concentrate and diffuse by the effect of biological enrichment and food chains; therefore, it might appear in food products and pose a potential risk for human health (Sun et al., 2011).

In examined meat, the only congener of PCBs detected was hexachlorobiphenyl PCB 138 with a mean value 4.90 ± 3.10 $\mu\text{g}/\text{Kg}$. Its frequency distribution was 33% of samples. This level of residue is lower than the permissible limit set by EU commission (2011). In this regard, a total of 12,563 food and feed samples collected in the period 1995 - 2008 from 18 EU Member States, Iceland and Norway were retained for a detailed analysis of the occurrence of the six indicator NDL-PCBs (28, 52, 101, 138, 153, and 180). Overall, 18.8% of the results for single congeners were below the limit of quantification

(LOQ) but their distribution varied highly between food and feed groups. PCB-153 and PCB-138 were the most commonly detected congeners. In food, the highest mean contamination level was observed in fish and fish derived products followed by eggs, milk and their products, and meat and meat products from terrestrial animals (EFSA, 2010). Similarly, among 220 samples of pork tissues from southern Italy, only few samples of smoked ham exceeded the limit of European Community (Esposito et al., 2001). More chlorinated congeners were the most frequent in meat and meat products (heptachlorobiphenyl PCB 180 > hexachlorobiphenyl PCBs 153 > hexachlorobiphenyl PCB 138 > tetrachlorobiphenyl PCB 52 = trichlorobiphenyl PCB 28 = dichlorobiphenyl PCB 10). The frequencies of congeners 153 and 138 are in agreement with values found in Italian diet (11 and 14%; Zucato et al., 1999). However, the frequencies of PCB 180 and 153 in meat and meat products (41% and 16%) were low when compared to most environmental extracts (Lopez-Leiton et al., 2001). This finding could be related to differences in the contamination pattern among different countries. Meat and meat products from ruminants and pigs had an average NDL PCBs content of less than 5 ng.g⁻¹ fat (4.64 and 3.69 ng.g⁻¹ fat, respectively). Among animal fats, the greatest average level of contamination was discovered in the fat of ruminants (8.71 ng.g⁻¹ fat) (EFSA, 2010). Andrée et al. (2010) reported an average content of the sum of PCB-6 indicator congeners in beef (5.33 ng.g⁻¹ fat) and pork (1.41 ng.g⁻¹ fat). In comparison with these average values for the sum concentration of PCBs and the values calculated from documentation on 18 EU member states, Iceland and Norway,

the average NDL PCB concentrations we found were approximately twice higher in the meat of cattle and pigs (Drápal et al., 2013). In Ismailia city, Egypt, the sum concentration of 17 PCBs congeners were measured in samples of animal origin (meat, dairy products and seafood) and their levels varied between 2.5 and 322 ng.g⁻¹ fat, corresponding to 1.7-216 ng.g⁻¹ fat for the seven indicator PCBs. The highest values were measured in seafood, the lowest in dairy products. Hexa-PCB 153, 138 and hepta-PCB180 were the congeners with the highest contribution. PCBs congener profiles found in most of the samples PCBs' contamination levels reported in this paper were many times lower than in developed countries (Loutfy et al., 2008). In the present study, the only herbicide residue detected in the examined meat samples was trifluralin. It was detected in 67% of domestic examined meat samples. The mean average concentrations were 0.21±0.08 µg /Kg, respectively. These residues levels were below the permissible limit set by EU commission (2012). Trifluralin is a selective, preemergence herbicide used to control a wide range of annual grasses and broadleaf weeds. It is typically soil incorporated, is highly absorbent to soil, and essentially non-soluble in water. The potential for bioaccumulation under environmental conditions depend largely on the elimination rates in biota and the environment (Han et al., 2007). US EPA (1996) indicates that uptake of trifluralin bound to sediment by aquatic animals is likely to occur. The latter may result from oral uptake of sediment particles carrying trifluralin on their surface or by uptake of trifluralin dissolving from the sediment particles (EUTTF, 2002). Available data indicate that trifluralin can accumulate to detectable levels reaching 0.290 µg/g in fish and benthic invertebrates. Trifluralin residues accumulated in bluegill sunfish exposed to 0.0059 ppm and these residues eliminated from the fish tissues after 14 days of exposure to pesticide free water (US.EPA, 1996). The available information on the properties of trifluralin in the environment suggested that the chemical is expected to be moderately persistent. It is not expected to be mobile, as a consequence of adsorption to soil. Although trifluralin is volatile, in the

field it dissipates primarily by soil binding and (secondarily) by biotic degradation. For trifluralin to be effective, it has to be incorporated into the soil at the time of application. Incorporation limits dissipation via surface active processes, primarily volatilization and photodegradation. Once incorporated, soil binding is the initial route of dissipation. This is followed by biotic degradation processes with a half-life of 116 to 201 days. A number of transitional degradates have been identified (US.EPA, 2009). There are no available data for trifluralin in meat or meat products, while there were few studies related to soil and vegetables which mentioned the detection of trifluralin residues in them (Mostafa et al., 1982). Therefore, U.S.EPA (2004) recommended that a ruminant metabolism study and a ruminant feeding study are required to determine the appropriate tolerance expression and tolerance levels of trifluralin residues for ruminant commodities. As well as, an analytical method for determining trifluralin residues in ruminant fat, meat, meat byproducts, and milk is also required in conjunction with the required cattle feeding study. Recently, the Analysis and Testing Center, Shenyang Agricultural University, China, they could determine the trifluralin residues in bovine milk. The found levels of the analyte in milk samples were lower than maximum residues levels (MRL) (Tian, 2011). In recent studies, trifluralin has been reported to cause a significant increase in thyroid follicular cell tumors in male Fischer 344 rats. The increased levels of TSH with chronic exposure to trifluralin would exert a continuous stimulation of the thyroid gland leading to cellular hypertrophy and proliferation predisposing to the development of follicular cell tumors in rats (Saghir et al., 2008). There was a possible link between trifluralin exposure and colon cancer among pesticide applicators exposed to trifluralin in the agricultural health study in a prospective cohort study of licensed pesticide applicators and their spouses in Iowa and North Carolina (Kanga et al., 2008).

The surprising findings in this study are the high values realized for the OPs. Furthermore, several OPs residue levels exceeded the MRLs set by EU regulations in the examined local meat. These results indicate a recent and extensive use of these types of pesticides in the investigated areas since they rapidly deteriorate and do not persist for a long time in the environment. Moreover, this study, in our knowledge, is the first Egyptian study that detected the herbicide trifluralin in animal tissues. These pesticides and their derivatives have been banned in most parts of the world; however, the results of the study have shown that they are still being used in Egypt and also other countries in the world. Therefore, we recommend that there should be a continuous program designed for analyzing and monitoring meat and meat products in the Egyptian local market before using to make sure that the levels of pesticide residues don't exceed the MLR. Also the usage of the banned pesticide should be stopped. In addition, Government should apply a training program to the farmers and show them the hazardous effects of using such banned pesticides or even using pesticides higher than the recommended doses. At regional level (i.e. African Countries), a strategic plan for monitoring and getting rid of POPs in the continent should be set up and implemented through coordination between all governments. Among issues of top priorities are to find alternative non- combustion technologies for disposing obsolete pesticides, and to use alternative control measures for mosquitoes' management and other vector-borne diseases (Mansour, 2009).

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Table (2): Parameters used for determination of pesticide residues in meat samples by GC-MS.

Pesticides	Retention time, min.	Group start time (number/min.)	Target ion (m/Z)	Limit of detection (LOD), µg/L	% Recovery
Trifluralin	9.461	I/7.5	306.00	0.0010	99.147
Disulfaton	11.124	IV/11.05	89.00	0.0007	101.333
Ethion	18.460	XV/18.40	231.00	0.0003	97.207
Methoxychlor	20.692	XVII/20.50	228.00	0.0191	74.610
Diazinon	10.797	III/10.75	137.00	0.0001	100.830
Heptachloro Epoxide	14.755	XII/14.68	353.00	0.0010	109.789
Aldrin	13.418	X/13.36	65.00	0.0001	114.684
p,p`-DDE	16.516	V/12.05	105.00	0.0001	76.344
Lindane	10.939	III/10.75	183.00	0.0001	99.473
Parathion methyl	12.307	VI/12.29	125.00	0.0001	92.263
Parathion Ethyl	14.446	XI/13.69	291.00	0.0004	100.596
PCB 28	12.082	V/12.05	57.00	0.0001	109.639
Dieldrin	16.600	XIII/16.38	262.90	0.0002	94.461
Chloropyrifos	13.418	X/13.36	196.90	0.0002	100.904
PCB 138	20.034	XVI/19.92	359.90	0.0001	130.361
PCB 52	13.093	VIII/13.04	292.00	0.0002	100.772

Table (3): Concentrations of OCs pesticide residues in examined meat samples

Pesticides	Local(balady) meat			
		Concentration range ($\mu\text{g/Kg}$)		Mean concentration ($\mu\text{g/Kg}$)
Aldrin	1	5		
	6	- 5		1.69 ± 0.91
	7	0		
p,p'-DDE	0	4	1	0
	1	- 8	0	± 7
	1	5	0	9
Lindane	2	5	1	7
	2	- 0	4	± 4
	1	0	2	7
Hexachlorobenzene		<LOD		<LOD
Heptachlor epoxide	1	6	3	1
	6	4	0	0
	0	- 0	2	± 3
Dieldrin	0	0	2	9
	1	2		3
	3	1	5	± 8
Methoxychlor	3	- 3	7	0
	3	3	8	
	0	2	0	0
Heptachlor	3	- 0	3	± 3
	3	0	9	3
		<LOD		<LOD

Table (4): Concentrations of OPs pesticide residues in examined meat samples

Pesticides	Local(balady) meat			
		Concentration range ($\mu\text{g/Kg}$)		Mean concentration ($\mu\text{g/Kg}$)
Malathion	1			0.
	.	2.0	0.	\pm 3
	3	- 0	5	6
	8		6	
Parathion-Ethyl	3		1.	0.
	.	4.1	2	\pm 7
	3	- 0	5	9
	7			
Parathion-Methyl	3		9	4
	.	27.	4.	\pm 4.
	1	- 66	1	2
	9		5	2
Ethion	9			
	0		3	2
	.	13	7.	\pm 4.
	2	- 5.0	5	4
Chlorpyrifos	0		3	3
	1		9.	8.
	.	50.	6	\pm 1
	7	- 00	7	3
Diazinon	5			
	2		1	3
	8	23	1	\pm 5.
	.	- 8.9	1	9
Disulfoton	3	8	1.	8
	0		7	
	0		2	1.
	.	11.	.	\pm 8
Disulfoton	7	- 55	2	8
	6		0	

Table (5): Concentrations of PCBs and herbicide residues in examined meat samples

Pesticides	MRL μ/kg	Not Detect ed	Within or below MRL	E x c e e d e d M R L
PCB 28	<LOD	<LOD		
PCB 52	<LOD	<LOD		
PCB 138	1	1	3	
	4.	5	4	
	2	.	.	± .
	9	1	9	1
	-	0	0	0
PCB 118	<LOD	<LOD		
PCB 180	<LOD	<LOD		
Trifluralin	0.	0	0	0
	2	.	.	± .
	0	-	2	0
		5	1	8
Alachlor	<LOD	<LOD		
Atrazine	<LOD	<LOD		
Propachlor	<LOD	<LOD		
Simazine	<LOD	<LOD		

	F	C S	E	Ba	Ba	B
	CS	AI	U*			a
	CS	•				
Aldrin	2 0 0	- - -	20 0		■	
p,p'-DDE		5 0 0 0	10 00		■	
Lindane	2 0 0 0	- - -	20		■	
Hexachlorobenzene		- - -	20 0	■		
Heptachlor epoxide	2 0 0	2 0 0	20 0		■	
Dieldrin	2 0 0	- - -	20 0		■	
Methoxychlor	- -	- -	10		■	
Heptachlor	2 0 0	2 0 0	20 0	■		

EU* : The Comparison done according to EU which is the lowest MRLs between different regulations.

Table (6): Frequency distribution of OCs pesticide residues in the examined meat samples compared with the recommended permissible limits.

Table (7): Frequency distribution of OPs pesticide residues in the examined meat samples compared with the recommended permissible limits.

Pesticides	F	MRL	Not	Within	Exceeded
		μ/kg	Detected	or below MRL	MRL
	CS	E	Ba	Ba	Ba
	AI	U*			
	CS				
	•				
	4				
Malathion	-	0	2		
	-	0	0	■	
	-	0			
Parathion -Ethyl	-	-	5	■	
	-	-	0		
	-	-			
Parathion -Methyl	-	-	1		■
	-	-	0		
	-	-			
Ethion	2	2			
	5	0	1		■
	0	0	0		
	0	0			
Chlorpyri fos	1				
	0	5	--	■	
	0	0	-		
	0				
Diazinon	2	-	2		■
	0	-	0		
	0	-			

	0			
Disulfoton	-	-	1	■
	-	-	0	
	-	-		

EU* : The Comparison done according to EU which is the lowest MRLs between different regulations.

Table (8): Frequency distribution of PCBs and Trifluralin residues in the examined meat samples compared with the recommended permissible limits.

Pesticides	F	MRL μ/kg	Not Detected	Within or below MRL	Exceeded MRL
PCB 28	-	3	4	■	
	-	0	0		
PCB 52	-	-	4	■	
	-	-	0		
PCB 138	-	-	4	■	
	-	-	0		

PCB 118	-	-	-	■	
PCB 180	-	-	4	■	
Trifluralin	-	-	1		■
Alachlor	-	2	1	■	
Atrazine	-	0	0	■	
Propachlor	-	2	-	■	
Simazine	-	0	0	■	
	-	2	2	■	
	-	0	0	■	
	-	2	1	■	
	-	0	0	■	

EU* : The Comparison done according to EU which is the lowest MRLs between different regulations.

Fig. (1): Levels of different types of pesticide residues in examined local meat

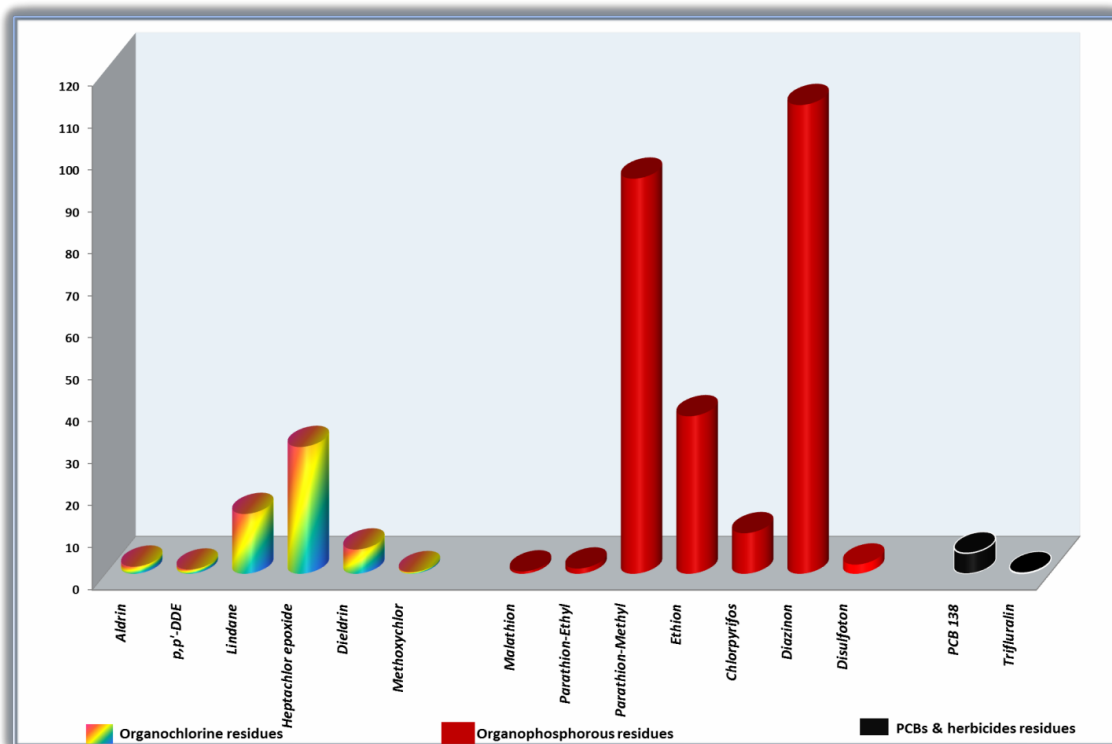


Fig. (2): Frequency (%) of OCs pesticide residues in examined local meat

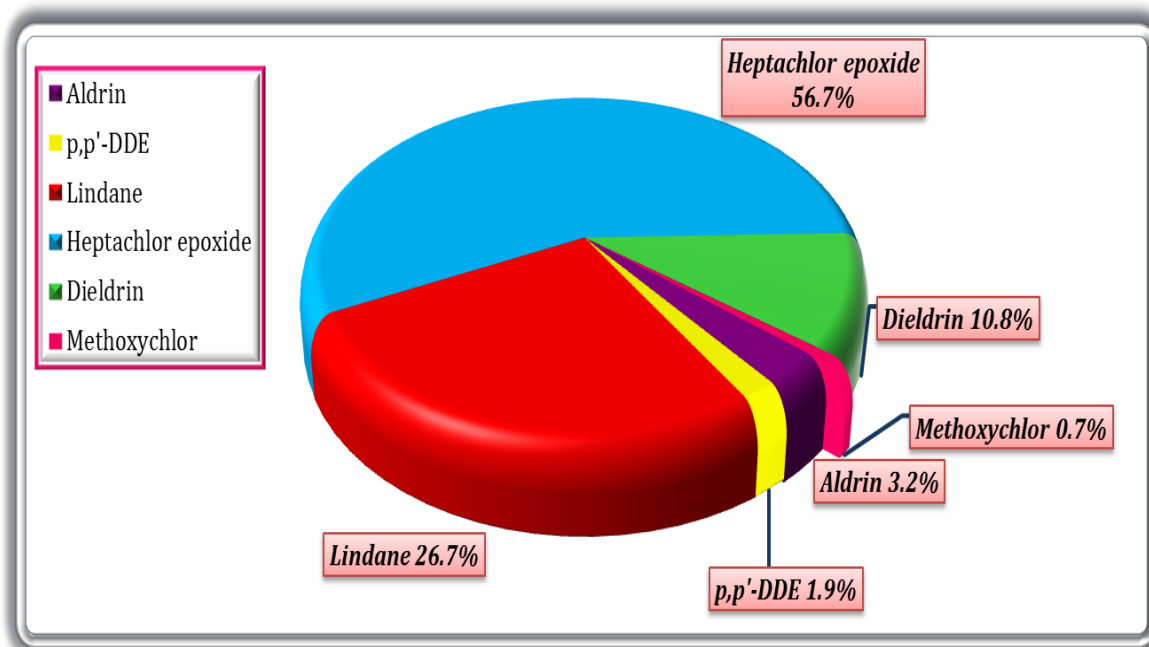


Fig. (3): Frequency (%) of OPs pesticide residues in examined local meat

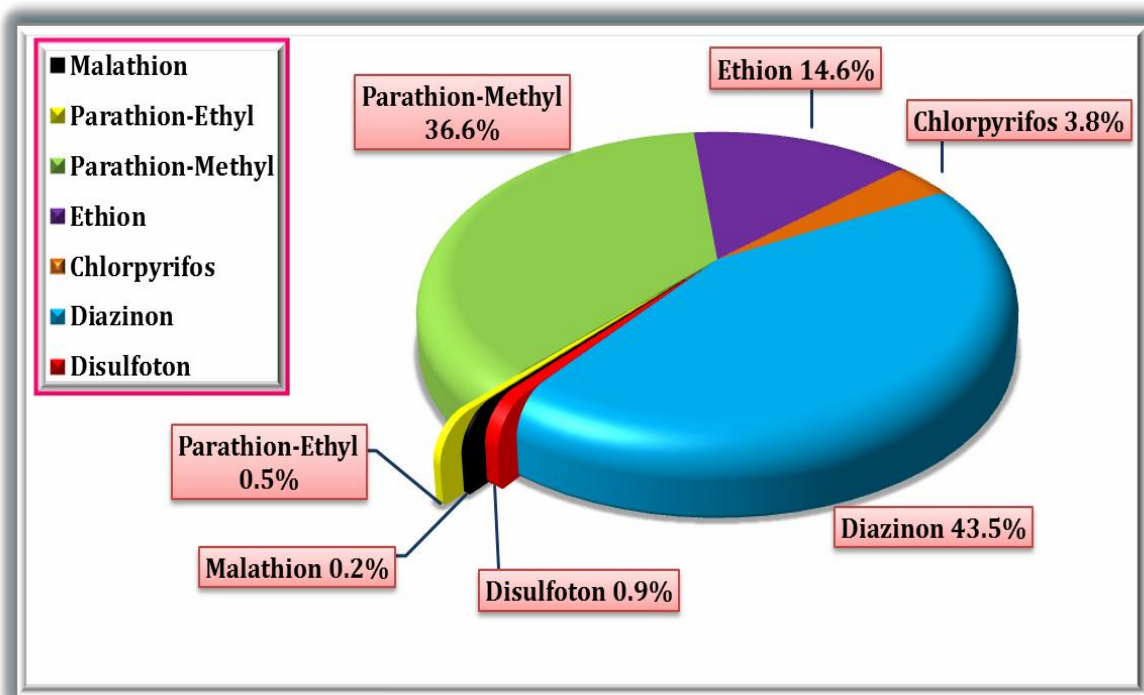
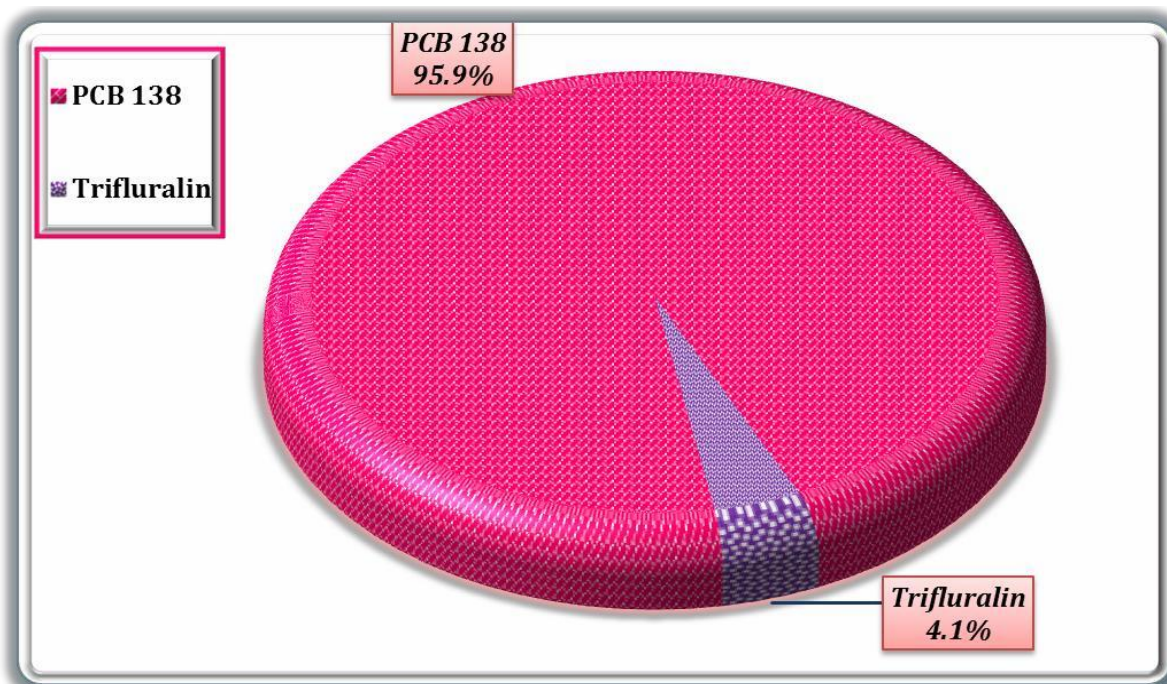


Fig. (4): Frequency (%) of PCBs and trifluralin residues in examined local meat



متبقيات العديد من مبيدات الآفات وبعض الملوثات العضوية الآخري في اللحوم المحلية المستهلكة في السوق في مدينته اسويوط

أ.د/أيمن عز الدولة الشرقاوي^١، أ.د/ أحمد عبد الباقي شرقاوي^١، د/وفاء أحمد على محمود^٢

^١ قسم الطب الشرعي والسموم البيطرية- كلية الطب البيطري- جامعة أسويوط

^٢ باحث بدرجة الدكتوراه الطب الشرعي والسموم البيطرية في الحجر hgبيطري بمطار اسويوط الدولي

الملخص :

اهتمت هذه الدراسة بالكشف و تحديد النسب المختلفة لمستويات المتبقيات لبعض المواد الكيميائية السامة الموجودة في اللحوم الجاموسي الغير مطهيه والتي تتد اول في مدينته اسويوط. وقد اشتملت هذه الدراسة علي قياس مستويات المتبقيات للمبيدات الكلورنيه العضويه والمبيدات الفسفوريه العضويه وبعض مبيدات الاعشاب وايضا المواد البولي كربيديز في عينات اللحوم البلديه. وقد تم فحص وتحليل عدد ١٠٠ عينه من اللحوم البلديه الغير مطهيه و التي تم تجميعها عشوائيا من محلات بيع اللحوم في مدينته اسويوط وذلك في مدي زمني يقدر ب ١٢ شهر من يناير الي ديسمبر ٢٠١٣. وقد تم استخلاص العينات باستخدام العمود الصلب للاستخلاص و التنظيف solid phase column. وقد تم اجراء الكشف المتعدد لتركيز المتبقيات باستخدام تقنية الفصل الكروماتوجرافي الغازي / مطياف الكتلة (GC/MS) كما تم حساب المتوسط الحسابي لتركيز المتبقيات \pm الخطأ المعياري. وقد قورنت نتائج الدراسة مع الحدود القصوى للمنظمات الدولية المختلفة المعنية بالأمر وكان الحد الأساسي للمقارنة هو 'الحدود القصوى الاتحاد الأوروبي'.

وقد اشارت النتائج الي تواجد متبقيات مبيدات الآفات الكلورية العضوية على النحو التالي ألدرين، الودي DDE ، الليندان، والهبتاكلور إيبوكسيد، داي ألدرين، وميزواوكسي كلور. قيم المتوسط الحسابي لهذه المبيدات كانت 0.91 ± 1.69 ، 1.00 ± 0.79 ، 0.26 ± 1.47 ، 0.22 ± 0.39 ، 0.78 ± 0.39 و 0.33 ± 0.39 ميكروجرام/كجم على التوالي. ولم يتم اكتشاف كل من سداسي كلورو البنزين، وسباعي الكلور ووجد أن بقايا هذه المبيدات لم تتجاوز الحد المسموح به. وقد اظهرت النتائج كل من الألدرين، و DDE في ٤٣% من العينات بينما الليندان وسباعي الكلور- إيبوكسيد وُجد في معظم العينات في حوالي ٨٣% في حين وجدنا الادي ألدرين وميزو اوكسي كلور في ٣٣% من العينات. واشتمل الكشف عن متبقيات مبيدات الآفات الفوسفورية العضوية في اللحوم المحلية مركبات كل من مالاثيون، إيثيل الباراثيون، باراثيون الميثيل، اثيون، والكلوربيريفوس، ديازينون، ديسولفوتون. وكان المتوسط الحسابي لهذه المبيدات كالتالي: 0.36 ± 0.56 ، 1.25 ± 0.79 ، 1.15 ± 0.22 ، 0.53 ± 0.43 ، 0.67 ± 0.13 ، 1.17 ± 0.98 و 1.88 ± 0.20 ميكروجرام/كجم على التوالي. ووجد أن باراثيون الميثيل، اثيون وديازينون تجاوزا الحدود المسموح بها. وكان مبيد الآفات الأكثر تواجداً في العينات هو ميثيل الباراثيون التي تم اكتشافه في جميع العينات ثم يليها الديازينون التي تم الكشف عنه في ٨٣% من العينات. تم الكشف عن كل من مالاثيون، إيثيل الباراثيون واثيون في ٣٣% من العينات في حين تم اكتشاف الكلوربيريفوس وديسولفوتون في ٤٣% من العينات. وايضا في الدراسة الحالية ، كانت المتجانسات الوحيدة لثنائي الفينيل متعدد الكلور التي تم اكتشافها في اللحوم المحلية هو ثنائي الفينيل متعدد الكلور ١٣٨ بقيمة متوسطة 4.90 ± 3.10 ميكروجرام/كجم بنسبة ٣٣% من العينات. وكان أقل من الحد المسموح به دوليا. وقد تم الكشف علي المبيد الاعشاب التايفلورين وكانت نسبه تواجده العينات التي تم فحصها ٦٧% وقد كانت ايضا تحت الحدود المسموح بها دوليا. ويجدر الاشارة هنا الي النتائج المفاجئة لهذه الدراسة و المتعلقة بالقيم العالية لمستويات تركيزات متبقيات مبيدات الآفات الفوسفورية العضوية والتي سجلت ارتفاعا ملحوظا في عينات اللحوم . ذلك ومن خلال تقييم المخاطر التي تم مناقشتها في هذه الدراسة وايضا المخاطر الصحية المحتملة والمرتبطة باستهلاك اللحوم والتي لا يمكن تجاهلها، لذا نوصي بضرورة الفحص المنتظم و المستمر للمتبقيات الكيميائية في اللحوم المستوردة والبلدية لضمان صحة وسلامة المستهلكين.