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Determination Of A Pyrethroid Insecticide Deltamethrin Residues In Cattle Meat In Baghdad Province/Al-Rusafa

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Abstract

Deltamethrin (DMT) is one of the synthetic pyrethroid compounds having wide insecticidal activity against a large number of ectoparasites and has important uses in veterinary and human health protection. In Iraq DMT has been widely used now in Baghdad and other provinces for sheep, goats and cattle dipping or spraying by the veterinarians and farmers for controlling of ectoparasites in animals and as insecticide in crop production in agricultures. The aims of this study were to determine the levels (ppm) of DMT residues in locally slaughtered cattle's meat consumed in Baghdad province /Al-Rusafa. A total of 50 cattle's meat samples were collected from 10 different regions of Baghdad province Al-Rusafa (Al-Kurrada Int., Al-Arassat, Al-Aadhamiya, New Baghdad, Al-Ghadeer, Al-Baladiyat, Al-Orffaly, Al-Habibiya, Al-Ameen 1st , and Al-Ameen 2nd markets) from October 2015 to March 2016. Five cattle's meat samples were purchased from different butchers per region. These samples were analyzed by using High Performance Liquid Chromatography techniques (HPLC). The residues analysis showed that all cattle's meat samples were positive for DMT residues. The data showed that there were significant (P≤ 0.05) differences in the concentrations (ppm) of DMT residues among the 10 regions of Baghdad province/Al-Rusafa. According to the locations the obtained data were compared with the Maximum residual limits (MRLs) permissible by the WHO and FAO and it was found that there was 2253% violation of the MRLs (above 0.026) in cattle's meat. The results of this study revealed that there were significant differences (P≤ 0.05) in the mean levels of DMT residues (ppm) in cattle's meat, between October and other months during the study period. These data were compared with the Maximum residual limits (MRLs) permissible by the WHO and FAO and it was found that there was 796% violation of the MRLs (above 0.026) in cattle's meat.

Keywords: Deltamethrin, insecticidal activity, HPLC, MRLs.

Introduction

A pesticide is any substance or mixture of substances intended for preventing, destroying, repelling, or mitigating any pest. Pests can be insects, mice and other animals, unwanted plants (weeds), fungi, or microorganisms like bacteria and viruses. By their very nature, most pesticides create some risk of harm to humans, animals, or environment because they are designed to kill or otherwise adversely affect living organisms. At the same time, pesticides are useful to society because of their ability to kill potential disease-causing organisms and control insects, weeds, and other pests (Zhang *etal*.,2011; EPA,2014). Deltamethrin has been widely used in Iraqi provinces for sheep, goat, buffalo and cattle dipping or spray by the veterinarians and farmers. In order to minimize the health risks from the ingestion of food contaminated

with DMT, EPA and WHO, have set maximum residue limits (MRLs) for DMT in different food commodity (FAO/WHO,2015).Meat may contain high levels of pesticide residues as a result of the concentration of residues in the tissues following cattle, sheep and goat dipping or vector control or when they feed on feedstuffs contaminated with these chemicals. Because these chemicals are toxic to living organisms, increased accumulation in the food chain may pose serious health hazards to the general populace (Jeyashree and Vasudevan, 2007; Niewiadowska *et al*., 2010).

Red and white meats are among the most popular food items in the Iraqi diet of many communities in Iraq and because of their fat content, a regular screening of meat is necessary to determine consumer exposure to DMT. Since there is no data on the residual level of DMT in cattle's meat tissue after different routes of exposure, this study was conducted to determine the levels of DMT pesticide residues in cattle's meat samples collected from Baghdad Province/Al-Rusafa, using High-Performance Liquid Chromatography (HPLC).

Materials and Methods

1. Collection and processing of meat samples:

Ten different locations were selected for meat samples collection located in Baghdad Province/AL-Rusafa which included Al-KurradaInt.,Al-Arassat,NewBaghdad,Al-Ghadeer,Al-Baladiyat,Al-Orffaly,Al-Habibiya,Al-Ameen 1st, and Al-Ameen 2ndmarkets.Five samples were purchased from each location from different butchers shops. The average weight/sample was 300 gm (80% lean and 20% fat) from the original of 500 gm. Each sample was packed separately in a sterile polyethylene bag, and the 5samples / location were transferred to the laboratory in an ice box.

2. Preparation of Deltamethrin standards:

1. Stock solution(SS) [mg/ml] was prepared from Deltamethrin standard in methanol Ab % and kept in freezer at -20°C. The stock standard solution was used for up to 3 months.
2. Suitable concentrations of working standards solutions (WSS) were prepared from the stock solutions (SS) by dilution using methanol Ab %, immediately prior to sample preparation.

3. Sample preparation and separation:

1. One hundred grams of minced meat sample were homogenized for 5 minutes in a homogenizer and stored in a clean, sealed plastic pack at -18 °C in a deep freezer.
2. One hundred grams of the homogenate was placed into a 250 ml polypropylene centrifuge tube with 100 ml of methanol, and Vortexed for 1 minute.
3. The sample was extracted ultrasonically for 10 minutes.
4. The sample was centrifuged at a speed of 4000 r/min for 5 minutes.
5. The supernatant was removed and saved in a clean conical flask (250ml) and evaporated with N2 below 40 °C.
6. The residue was reconstituted in 1ml of 5 % methanol and water.
7. Then 20 µl were subjected to HPLC analysis.

Results and Discussion

1. Determination of the levels (ppm) of DMT residues in cattle's meat samples collected from Baghdad province/Al-Rusafa.

A total of 50 local cattle's meat samples were collected from 10 different regions(5 samples/region) from Baghdad province/Al-Rusafa (Al-Kurrada Int., Al-Arassat, Al-Aadhamiya, New aghdad, Al-Ghadeer, Al-Baladiyat, Al-Orffaly, Al-Habibiya, Al-Ameen 1st, and Al-Ameen 2nd). The HPLC residues analysis revealed that all the 50 samples were positive for DMT residues (Table 1).The results of HPLC analysis from all regions were above the MRLs (0.026), which violate the recommendation of the WHO/FAO for DMT residues (ppm) in cattle's meat. The mean total of violation percentage were 2253% in the 10 regions examined(Table1).

Table 1. Levels of DMT residues (ppm), and violation % in cattle's meat samples collected from different regions of Baghdad Province/Al-Rusafa.

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Parameters / Regions	№. of samples tested	Range	Mean±SD	% Violation (MRLs=0.026)
Kurrada Int.	5	0.69 - 0.91	0.77 ± 0.08	**2962**
Arassat	5	0.39 - 0.65	0.55 ± 0.12	**2115**
Aadhamiya	5	0.03 - 0.48	0.19 ± 0.19	**730**
New Baghdad	5	0.29 - 0.79	0.53 ± 0.27	**2115**
Al-Ghadeer	5	0.27 - 0.45	0.36 ± 0.07	**1384**
Baladiyat	5	0.26 - 0.69	0.43 ± 0.19	**1653**
Al-Orffaly	5	0.14 - 0.84	0.46 ± 0.29	**1769**
Habibiya	5	0.14 - 0.49	0.29 ± 0.13	**1115**
Al-Ameen 1st	5	0.22 - 0.35	0.27 ± 0.05	**1038**
Al-Ameen 2nd	5	0.11 - 0.42	0.28 ± 0.11	**1076**
Total	**50**			
Range		**0.91-0.03**		
Mean±SD			**0.41± 0.17**	
mean total				**2253%**

The overall, one way analysis of variance (ANOVA) revealed that there were significant differences ($p \leq 0.05$) in the residual levels (ppm) of DMT ($F=5.203$, $P=0.0001$). Using post-hoc t-test, Table 2 shows that there were significant differences ($p \leq 0.05$) between the 10 regions.

Table 2. Comparisons of DMT residues (ppm) in cattle's meat samples collected from different regions of Baghdad province/Al-Rusafa.

Parameters / Regions	№.of samples tested	Mean±SD	Significant differences between regions ($p \leq 0.05$)
Kurrada Int. (1)*	5	0.77 ± 0.08	**1x3; 1x5; 1x6;1x7;1x8;1x9**
Arassat (2)	5	0.55 ± 0.12	**2x3;2x8;2x9;2x10**
Aadhamiya (3)	5	0.19 ± 0.19	**3x4;3x7**
New Baghdad (4)	5	0.53 ±0.27	**4x9;4x10**
Al-Ghadeer (5)	5	0.36 ± 0.07	
Baladiyat (6)	5	0.43 ± 0.19	
Al-Orffaly (7)	5	0.46 ± 0.29	
Habibiya (8)	5	0.29± 0.13	
Al-Ameen 1ST (9)	5	0.27 ±0.05	
Al-Ameen 2nd (10)	5	0.28 ±0.11	

*( )=Region's code.

These results may be attributed to several reasons such as the use of contaminated fodder with DMT. The uptake of DMT residues in the soil, vegetables, water and other herbage may have a role in the contamination of the meat. Also after dipping, the animals need longer time for drying; this leads to prolong skin exposure to DMT and then high skin absorption. The animal may consume feed and feedlots contaminated with DMT from the soil since DMT have strong tendency to bind onto soil organic matter (Tomlin, 2006). These results were partially consistent with the result of Paton and Petters on (1997) who showed that the livestock reared on dieldrin contaminated soil, crops, and fodders may accumulate considerable residues in edible tissues. Furthermore, Keyyu *et al*. (2003) mentioned that most

farmers in developing countries do not often care about withdrawal periods in treated animals. The MRLs recorded by this study were exceeded for all of the samples analysed, and the recoded data were higher than the maximum residual limits established by WHO (1989); and EC (2005) which is 0.062 ppm in cattle's meat .The high residues probably reflect the ingestion of high contamination levels of DMT pesticides from soil or contaminants in feed . The residues detected were considered significant from the consumer public health safety points of view. These results were in agreement with Da-L jung *et.al*.(2014), who found that DMT residues in beef meat samples was accurately determined at a very low level of 10mg/kg (10ppb), using Gas chromatography(GC) method. Stefanelli *et al*.(2009) who found multiresidues of organochlorine and pyrethroids in 50 commercial beef meat samples from the market area of Italy/Rome were observed above their respective limit of detection (LOD) and this is agreement with the results of this study. These results were in agreement with Abdurrahman (2016) who found that the levels (ppm) of DMT sheep and goat meat in Iraq/Sulaimaniya province (5 regions) have had high levels (ppm) of DMT residues which violate the compliance of MRLs that established by WHO and FAO (2012).The results were also in agreement with Muhammad *et. al*. (2010) who found that the meat samples of cattle from three sites in south-west of Faisalabad-Pakistan were higher than the available (MRLs) that recommended by WHO and FAO (2012).However, the results of this study were incompatible with the results of Turyahikayo (2013) who indicated that there were no pyrethroid residues detected in the cattle meat, around Lake Mburo National Park, South Western Uganda. The significant differences between regions could be attributed to several reasons such as difference in age of animals, the site that animals come from, source of water, type of rearing and differences in withdrawal time. Another viewpoint about high levels (ppm) of DMT residues above the MRLs, Chauhan and Lokesh (2006) explain that the pesticide residues which could be related to several reasons including : the farmers may use pesticides more frequently and in increased doses than the recommended doses or procedures, most farmers having low formal education they are not able to understand and read the instructions mentioned on pesticide, most of them have impression in their mind that spray use of more pesticide will lead to higher production.

Meat may contain high levels of pesticide residues as a result of concentration of residues in the tissues following cattle dipping or vector control or when they feed on feedstuffs contaminated with these chemicals, another reasons were mention by Vijaya and Ravindra (2015) that the direct contact of the animals with pesticides during control of external parasites on animal and insects and fly control in cattle yards and sheep shed.

2. Determination of the levels (ppm) of DMT residues in cattle's meat samples collected during the months of study period.

The concentration levels (ppm) of DMT residues in the cattle's meat samples (10 samples/month) were determined during the months of the study period (October, December, January, February, and March), (Table 3).

Table 3. Levels of DMT residues(ppm), and violation% in cattle's meat samples collected during the study periods(months).

Parameters / Months	№.of samples tested	Range	Mean±SD	% Violation (MRLs=0.026)
October	10	0.39-0.91	0.66 ± 0.16	**2538**
December	10	0.03-0.97	0.36 ± 0.28	**1384**
January	10	0.27-0.69	0.39 ± 0.14	**1500**
February	10	0.14-0.84	0.38 ± 0.24	**1461**
March	10	0.11-0.42	0.28 ± 0.08	**1077**
Total	**50**			
Range		**0.03-0.97**		
Mean±SD			**0.41± 0.14**	
Mean total				**796%**

The overall analysis of variance (ANOVA) revealed that there were significant differences ($p \leq 0.05$) in the residual levels (ppm) of DMT ($F=5.538$, $P=0.0010$) between the five months.

The HPLC analysis revealed that all the 50 samples were positive for DMT residues. The results of HPLC analysis for all months were above the MRLs (0.026), which violate the recommendation of the WHO/FAO for DMT residues (ppm) in cattle's meat. The mean total of the violation % recorded in the five months was 796% (Table 3).

Table 4.Comparisons of DMT residues (ppm) in cattle's meat samples collected from Baghdad province/Al-Rusafa during the study period.

Parameters / Months	№.of samples tested	Mean±SD	Significant differences between months (p≤0.05)
October (1)*	10	0.66 ± 0.16	
December (2)	10	0.36 ± 0.28	**1x2**
January (3)	10	0.39 ± 0.14	**1x3**
February (4)	10	0.38 ± 0.24	**1x4**
March (5)	10	0.28 ± 0.08	**1x5**

*( )=Month's code.

The results of this study could be explained by the study carried out by Ian (2004), who suggested that variation in levels of pesticide residues during different season may be due to physical and chemical factors of the environment, so the environmental variations can affect the concentration of pesticide residues. Moreover, the highest Mean±SD value was recorded in October (0.66±0.16), and the lowest Mean±SD value was recorded in March (0.28±0.08). Another factor which could contributed to the high DMT residues levels in cattle's meat samples collected in October as suggested by Aydin *et al*.(2013) is that contamination of drinking water of the animal farms may be attributed to spraying of animal by DMT during certain months without any precautions to avoid contamination of drinkers as covering or emptying of drinkers and filling or washing sprayer near them, as well as absence of special area for pesticides application. The frequent application of DMT in the agricultural during growing seasons of cereals and veterinary sectors (June, July, August and September) could be attributed, while limited application occurred during January. February, and March. Moreover, Bedi (2012) mentioned that DMT has been registered for vast applications for control of insect on cotton, rice, wheat, vegetables, fruits, grain storage and for public health programs in mosquito control to prevent transmission of vector borne diseases. Also the ministry of agriculture in Iraq use DMT during summer season for treatment the *spring generation* of *Ommatissus lybicus* Deberg, that infect palm (Iraqi Ministry of Agriculture, 2016) ,this may lead to more soil and water contamination with DMT, which may lead to increased residues of DMT in meat.

Nag and Raikwar (2010) mention that considerable portion of such pesticide residue may enter into the food chain or may penetrate inside the animal's body and accumulate in the fat tissues and later may appear in the meat. Further, crops are heavily sprayed with pyrethoids in the field to protect them from pest and insect attack, and these crops are using for feeding of animals (Muhammad *et al*., 2012).The reason of the significant differences between moths could be due to that farmers didn't have commitment with dipping schedule, and slaughtered animals without knowledge on the withholding periods. The Department of health and human service public (2003) mentioned that pesticides frequently are used on crops, fruits and vegetables because of certain pyrethroids, such as permethrin, phenothrin, and resmethrin, are sprayed to control mosquitos during the spring and summer this could be another source of pyrethroid residues in animals commodities. Anwar *et al*. (2011) reported that the residue of pyrethroid had the same levels in different months and seasons in Pakistan, this could be attributed to the fact that there were no differences between the climate temperature during their study and the type of pesticides being used. Also, pesticides applied directly to the soil may be washed off by rain into nearby bodies of surface water or percolate through the soil to lower soil layers and ground water (Kamrin,1997), or may be due to illegal uses of pesticide at certain season (Aydin *et al*., 2013).

The results of this study revealed that climate temperature variations during the study months had have great effect on the levels of pesticide residues in meat samples.

Conclusions

From the data obtained from this study, can be concluded cattle's meat sold in Baghdad Province/Al-Rusafa (10 regions) were highly contaminated with Deltamethrin residues (ppm), which violate the compliance (MRL) established by the WHO and FAO. Deltamethrin residues determined in October were significantly higher than that reported in the other months of the study, which reflects the high veterinary and agricultural usage before and/or during this month.

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