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Assessment of pesticide residues in beef feed and meat in Bangladesh: A safety issues

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Abstract

The use of pesticides in agricultural sectors is rising due to the growing demand for food in the world, but the presence of pesticide residues in agricultural commodities has become a major health concern for consumers and is associated with problems of food safety. Pesticides in meats and milk were derived from feeds. The present study was conducted to find the pesticide residue in cattle feed ingredients, commercial beef cattle feed and meat of beef cattle. Commercial beef cattle feed, Heart, kidney, liver, lung and muscle samples of beef cattle were collected from four district of Bangladesh were analyzed by GC-MS (Gas chromatography–mass spectrometry) for organochlorine and organophosphorus pesticide residue. There were no organoclhorine and organophosphorus pesticide residues in collected raw materials and commercial beef feed except Saudia and Fresh feed. In case of commercial beef feed. Aldrin was found in Saudia feed and Fresh feed at the level of 77 and 336 µg/kg, respectively. The results indicate that the concentration of aldrin in commercial beef cattle feed named Saudia and Fresh feed were above maximum residue limit (MRL) values (20 µg/kg) set by WHO/FAO. Contaminants existed at different levels in cattle feed of Bangladesh.

Introduction

Livestock plays a vital role in promoting national economy of Bangladesh which is one of the most important sub-sectors of agriculture (Hasan et al., 2021; Kamal et al., 2019; Kamal et al., 2022; Kobir et al., 2022). Now a days, feed and food safety are the main issues in public health in Bangladesh (Hashem et al., 2020). Poultry, goats/sheep and cattle produced 40%, 12% and 48%, respectively. The safety of livestock products directly related to feeds (Kamal et al., 2020; Kamal et al., 2016). Food safety hazards associated with animal feed can be biological, chemical or physical (Rahman et al., 2020). Animal feeds and forages contain a wide range of contaminants and toxins arising from anthropogenic and natural sources (Hossain et al., 2016). In this report, the distribution of pesticides in feed ingredients, complete feeds and beef meat and organs is presented. A 'pesticide' is something that prevents, destroys, or controls a harmful organism ('pest') or disease, or protects plants or plant products during production, storage and transport. The term includes, amongst others: herbicides, fungicides, insecticides, acaricides, nematicides, molluscicides, rodenticides, growth regulators, repellents, rodenticides and biocides (Pesticides - European Commission). Organochlorine (OC) pesticides are synthetic pesticides widely used all over the world, even though their use has been banned or restricted in the past several decades. This has led to many researchers' investigations their occurrence, distribution and concentrations in meat, fish, fruit, vegetables and water (Lehotay et al., 2005). The greater use of pesticides for high agricultural production has led to increased pollution of environmental compartments – soil, water and air. The characteristics of pesticides, such as high lipophilicity, bioaccumulation, long half-life and potential of long range transport, have increased the chances of contaminating the air, water and soil, even after many years of application.

The residues of pesticides not only affect the public health but also cause economic losses to the livestock industry. Not only these affect the health of livestock and human beings but also affect the quality of animal products. Pesticide residues accumulate in the animals either by direct contact with pesticide or by indirect contact with environment. Pesticides are used in crops for pest control and they leave the residues in the feed and fodder consumed by animals (Reddy et al., 2015). One of the major aims of the study was to gain insight into the contamination with organochlorine and organophosphate pesticides in feed ingredients, compound cattle feed and beef. The results can be used to define priorities for national monitoring plans

Methods and Materials

Site of the Experiment

Collected samples (nine commercial beef feed and seven feed ingredients) of feed ingredients and commercial beef cattle feeds were analyzed at National Food Safety Laboratory (NFSL) under Institute of Public Health, Dhaka, Bangladesh.

Chemicals and Reagents

Standard pesticides of 98–99% purity were collected from Dr. Ehrenstorfer GmbH (Augsburg, Germany). Acetonitrile, toluene, sodium chloride, PSA Bond silica (primary and secondary amine), graphite car-bon black (GCB), triphenylphosphate (TPP) and anhydrous magnesium sulphate were obtained from Sigma-Aldrich (St. Louis, MO, USA). For analysis, high performance liquid chromatography (HPLC) grade organic solvents were used.

Sample preparation, extraction and determination of pesticide residues

Both organophosphate (OP) organochlorine (OC) pesticides with their metabolites were analysed for beef feed and meat samples. OC, OP groups were run separately for the analysis of the respective samples following the method of QuEChERS and quantification and detection of pesticides by GC-MS analysis. The extraction and clean-up were done based on the QuEChERS sample preparation method for pesticides (Anastassiades et al., 2003). The collected beef feed and meat samples were chopped and blended separately in an electric blender with microcutters (Preethi Steel Max MF-212, Preethi Kitchen Appliances Pvt. Ltd. India) to obtain an isolated homogenous composite of beef feed and meat. In a 50 ml centrifuge tube, an aliquot of 10 g homogenized sample and 10 ml of acetonitrile was mixed. The mixture was vortexed for one minute followed by adding 4 g of magnesium sulphate and 1 g of sodium chloride. The sample was centrifuged at 5000 rpm for 5 min and the supernatant was removed for clean-up.

During clean-up, 2 mL supernatant was transferred into another tube that contained 50 mg of primary and secondary amine (PSA), 50 mg of graphite carbon black (GCB) and 150 mg of magnesium sulphate. After proper agitation and centrifugation at 10000 rpm for 5 min, the aliquots of the extract were evaporated through nitrogen system and reconstituted with 1 mL toluene for GC-MS analysis. Further, each pesticide standard solution (1 mg/ml) was prepared by diluting acetonitrile at a different concentration for standard curve preparation. A 2% triphenylphosphate (TPP) solution in acetonitrile with 1% acetic acid was used as quality control (QC) standard for the GC-MS analysis. A Shimadzu (GC-MS QP 2010 Ultra, Japan) gas chromatograph equipped with mass selective detector and analytical column setup was a Restek (Bellefonte, PA) Rxi-5MS with fused silica (30 m long × 0.25 mm internal diameter × 1.0 µm film thickness) used for analysis.

The operating condition of OC and OP pesticides were splitless injection mode, 250°C injector and interface temperature, 1 min sampling time, helium gas as a carrier with flow rate 0.75 mL/min, linear velocity in flow control mode, 71.2 KPa pressure, 14 mL/min total flow rate, 1 ml/min column flow, 37 cm/s linear velocity, 3 mL/min purge flow and injection volume of 1 µL. The temperature was programmed for organophosphate pesticide from an initial value of 90°C, ramped to 180°C at 25°C/min, and to 270°C at 3°C/min, and was increased to 300°C at 20°C for 3 min and total run time was 40 min. Likewise, the operating condition of organochlorine pesticide was split mode, split ratio 10, 250°C injection port temperature, 1 min sampling time, helium gas as carrier with flow rate 0.75 mL/min, linear velocity flow of control mode, 124.6 KPa pressure, 19.5 ml/min of total flow, 1.5 ml/min of column flow, 46 cm/s of linear velocity 3 ml/min of purge flow and injection volume was 1µL. The initial value of 120°C, ramped up to 200°C at 45°C for 3 min, and to 240°C at 5°C for 10 min, and finally raised to 310°C at 10°C for 3 min and total run time was 34 min. For the detection of all analytes, analysis was performed in selected ion monitoring (SIM) mode and a minimum of four ions were considered for each pesticide.

Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

The organochlorine pesticides and their metabolites were analyzed with GC-MS: α-BHC, δ-BHC, β-BHC, γ-BHC, Heptachlor, Aldrin, Heptachlor epoxide, γ-Chlordane, α-Chlordane, α-Endosulfan, 4,40-DDE, Dieldrin, Endrin, 4,40-DDD, β-Endosulfan, 4,40-DDT and Endosulfan sulphate. Similarly, the organophosphate pesticides (Dimethoate, Chlorpyrifos and Quinalphos) were analysed with validated GC-MS method.

Method validation

The linearity of the calibration curves was assessed at 10, 40 and 200 mg/mL (equivalent to 0.01, 0.04 and 0.2 mg/kg) for all by duplicate analysis by using six concentrations levels. The obtained coefficient of determination (R2) was higher than 0.97 for all the pesticides. The accuracy and precision parameters of the study method were obtained over the entire procedure by recovery analysis. The recoveries were between 80 and 120%, RSD for precision was <10% and RSD for intermediate precision was <15%. The results of the study indicate that the method had acceptable reproducibility and satisfactory. Therefore, present study method provided a good possibility to quantify the pesticide residues in beef feed and meat.

Results and discussion

Pesticide residue in feed ingredients

Collected samples of feed ingredients were analyzed for nine (9) organochlorine and nine (9) organophosphate pesticides. The results revealed that, there were no organoclhorine and organophosphorus pesticide residues in feed ingredients (Table 1).

Table 1. Pesticide residue in feed ingredients

Sample name	Description of analysis	Results (ppb)
Feed ingredients 1. Sesame oil cake 2. Wheat bran 3. Maize 4. Mustard oil cake 5. Matikalai hull 6. Soybean meal 7. Straw	**Organochlorine Pesticides :** 1. Heptachlor 2. Aldrin 3. Dieldrin 4. Endrin 5. B-Endosulfan 6. 4'DDT 7. Endrin Aldehyde 8. Endosulfan sulphate 9. Methoxychlor **Organophosphorus Pesticides :** 1. Ethoprophos 2. Dimethoate 3. Diazinon 4. Metalaxyl 5. Fenitrothion 6. Malathion 7. Fenthion 8. Chlorphyrifos 9. Quinalphos	Not detected

Nuapia et al. (2016) investigated the level of organochlorine pesticides in the raw food and found that DDE recorded the highest mean concentration (253.58 ± 4.78 mg kg1) in beef from Johannesburg, and a-BHC recorded the lowest mean concentration (38.54 ± 7.46 mg kg1) in beans from Kinshasa. Khilare et al. (2016) found 310.368 ppb ΣHCH and 195.057 Σendosulfan in meat, respectively.

Pesticide residue in commercial beef cattle feed

Collected samples of commercial beef cattle feeds were analyzed for nine (9) organochlorine and nine (9) organophosphate pesticides. The results revealed that, there were no organochlorine and organophosphorus pesticide residues in commercial beef mixed feed except Saudia and united feed (Table 2). In case of commercial beef feed, Aldrin was found in Saudia feed and United feed at the level of 77 and 336 ppb, respectively.

Table 2. Pesticide residue in commercial beef cattle feed

Sample name	Description of analysis	Results (ppb)
Commercial beef cattle feed: 1. Gain feed 2. ACI feed 3. Care feed 4. Teer feed 5. Saudia feed 6. Fresh feed 7. Provita feed 8. Index feed 9. IFAEL feed	**Organochlorine pesticides:** 1. Heptachlor 2. Aldrin 3. Dieldrin 4. Endrin 5. B-Endosulfan 6. 4'DDT 7. Endrin Aldehyde 8. Endosulfan sulphate 9. Methoxychlor **Organophosphorus pesticides:** 1. Ethoprophos 2. Dimethoate 3. Diazinon 4. Metalaxyl 5. Fenitrothion 6. Malathion 7. Fenthion 8. Chlorphyrifos 9. Quinalphos	Not detected
Commercial beef cattle feed 1. Saudia feed 2. United feed	Aldrin Aldrin All other pesticide residues as mentioned above were not detected	77* 336* (MRL=20 ppb)

*Exceeds maximum residue levels (MRL)

According to Joint FAO/WHO food standards programme (2006), maximum residue limit of aldrin is 0.02 ppm that means 20 ppb. So the aldrin level of Saudia and united feed was much higher than the maximum residue limit, which was alarming. Aldrin is an organochlorine insecticide that was widely used until the 1990s, when it was banned in most countries. It is a colourless solid. Before the ban, it was heavily used as a pesticide to treat seed and soil in Bangladesh. Aldrin and related "cyclodiene" pesticides (a term for pesticides derived from Hexachlorocyclopentadiene) became notorious as persistent organic pollutants (Metcalf, 2002). Khilare et al. (2016) found 618.281 ppb ΣHCH and 135.626 ppb Σendosulfan in poultry feed, respectively.

Sabere et al. (2013) reported the range of pesticide residues in the leaf samples was from N/D to 579.60 ppb, in stem samples from N/D to 368.93 ppb and in the rice grain from N/D to 22.37 ppb. Some of the rice samples had levels that exceeded the standard MRL (as stipulated by WHO).

Rahman et al., (2021) determined pesticide residues (17 organochlorine, 5 pyrethroid and 3 organophosphate) in 77 fish feed, 112 fish and 135 vegetables samples (total of 324) from the different locations in Bangladesh, using quick easy cheap effective rugged and safe (QuEChERS) extraction followed by gas chromatography-mass spectrometry (GC-MS) analysis. The 77 fish feed samples analyzed with this method appeared to be free of pyrethroid pesticide residues. Organochlorine pesticide residues aldrin (0.03 ± 0.01 mg/kg) and pyrethroid pesticide residues permethrin (0.08 ± 0.01 mg/kg) were detected in fish samples of catla (Catla catla) from Rajshahi Durgapur and bata (Labeo bata) from Satkhira Kaligonj, respectively which is similar to our study.

Pesticide residues in different organs and muscle of beef cattle

Collected organ and muscle samples were analyzed for nine (9) organochlorine and nine (9) organophosphate pesticides. The results revealed that, organochlorine and organophosphorus pesticide residues of organs and muscle samples were not detected. (Table 3).

Table 3. Pesticide residues in different organs and muscle of beef cattle

Sample name	Description of analysis	Results (ppb)
1. Heart 2. Kidney 3. Liver 4. Lung 5. Muscle	**Organochlorine pesticides:** 1. Heptachlor 2. Aldrin 3. Dieldrin 4. Endrin 5. B-Endosulfan 6. 4'DDT 7. Endrin Aldehyde 8. Endosulfan sulphate 9. Methoxychlor **Organophosphorus pesticides:** 1. Ethoprophos 2. Dimethoate 3. Diazinon 4. Metalaxyl 5. Fenitrothion 6. Malathion 7. Fenthion 8. Chlorphyrifos 9. Quinalphos	Not detected

Conclusions

Organochlorine and organophosphorus pesticide residues were evaluated in cattle feed and meat and found that only aldrin was present in commercial beef feed named Saudia and Fresh at the level of 77 & 336 µg/kg, respectively, which was banned in all over the world. Maximum residue limit of aldrin for cereal grains is 20 µg/kg. In that case, aldrin concentration of Fresh and Saudia feed was higher than the MRL level set by WHO & FAO. Animal products like milk and meat are often found to be contaminated with residues of persistent pesticides and other toxic substances. The major source of entry of these compounds to animal body is the contaminated feed and fodder. So, unless the residues are managed at this stage, it is very difficult to prevent contamination in milk and meat. Therefore, the status of residue level of most persistent organochlorinated pesticides (OCP) and organo phosphate (OP) in feed and animal products should be monitored regularly.

Conflicts of Interest

The authors declare that there are no potential conflicts of interests.

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