



Analysis of Pesticide Residual Levels in Maize (*Zea mays L.*) Grain, Flour and Processed Items from Selected Areas of Dhaka, Bangladesh

ANUJ KUMER DAS¹ and G. M. M. ANWARUL HASAN^{2*}

¹Hi-Tech Health Care Ltd. Banani, Dhaka-1213, Bangladesh.

²Institute of Food Science and Technology, Bangladesh Council of Scientific and Industrial Research, Dr. Qudrat-I-Khuda Road, Dhanmondi, Dhaka-1205, Bangladesh

*Corresponding author E-mail: pd-cbirmdp@bcsir.gov.bd

<http://dx.doi.org/10.13005/ojc/380319>

(Received: February 23, 2022; Accepted: April 24, 2022)

ABSTRACT

In this study, the residual levels of Organophosphorus pesticides (OPPs) and organochlorine pesticides (OCPs) in maize grain, maize flour and processed items were determined. A total of 90 samples were collected and analyzed by Gas chromatography Tandem Mass spectrometry (GC-MS/MS). Several OPPs residues including Dichlorvos ($964.38 \pm 143.21 \mu\text{g/kg}$), Methyl Parathion ($43.98 \pm 12.90 \mu\text{g/kg}$) and Dursban (Chlorpyrifos) ($39.82 \pm 10.75 \mu\text{g/kg}$) were detected in maize grain samples while Dichlorvos ($128.65 \pm 22.78 \mu\text{g/kg}$) and Dursban (Chlorpyrifos) ($12.54 \pm 7.66 \mu\text{g/kg}$) were detected in maize flour samples and no OPPs residues were detected in processed maize items. Among the OCPs residues, p',p'-DDE ($6.54 \pm 2.66 \mu\text{g/kg}$), p',p'-DDD ($3.54 \pm 2.98 \mu\text{g/kg}$) and p',p'-DDT ($4.32 \pm 2.98 \mu\text{g/kg}$) were detected in maize grain samples while no residues were detected in maize flour and processed items. None of detected pesticide residual concentration exceeds the maximum residue limits (MRLs). From this analysis it can be concluded that, there have no potential health hazards from raw and processed maize items from the studied area.

Keywords: Organophosphorus pesticides, Organochlorine pesticides, Gas chromatography, Tandem Mass spectrometry.

INTRODUCTION

Agricultural production depends on food cultivation. For better production of agricultural crops, it is necessary to spray pesticides to protect the crops from pests. OPPs are widely used throughout the world for agricultural production because they are inexpensive and stable¹. The activity of acetyl cholinesterase and acetylcholine

can be inhibited by OPPs which affects central nervous system and may lead one to death. Organophosphates are low persistent and high bio-efficient therefore, farmers prefer to use this group of pesticides for crop production. The ecosystem is affected due to the uses of pesticides². OCPs are widely used in mid twentieth century to protect the agricultural crops. OCPs are relatively stable and have bioaccumulation ability. After the application of



OCPs in soil and plants, these toxic residues may transfer into the higher trophic levels through food chain. OCPs residues can be detected in fatty foods including meat, egg, milk, vegetable oil, nuts, oat etc. Human exposure to OCPs is mainly through food. OCPs are also distributed throughout the environment and thus unwanted human exposure can be occurred through inhalation^{3,4,5}. OCPs mode of action is to target the enzymes of the insects, therefore, the mode of action should be same for human also. So, the presence of these pesticide residues in food is a major health and environmental concern. The common exposure routes of pesticide residues to human body are through foods like fruits, vegetables, cereal crops, water and foods of other varieties. Therefore, pesticide residues analysis is an important task of consumers, producers and food quality control authorities⁶. OPPs and OCPs residues contaminate surrounding environmental elements like soil and water. Thus, the ecosystem is also affected which is one of the major environmental issue.

Maize ranks number 3rd crop in Bangladesh just after rice and wheat. In the last one decade, the maize production of Bangladesh have raised to 5.4 million metric tonnes⁷. However, still the annual demand of maize is 6.5 million to 7.0 million metric tonnes. Maize is commonly used as poultry feed and human food. Maize is one of the oldest and important crops of the world. Among the cereals, maize is the higher yielding crop which has multiple uses. Maize is also an important cereal crop in Bangladesh. Pesticides are extensively used for better production of crops like maize. Pests can attack the crops in any stage of its production from farming to storage. Through using pesticides the crops production can be increased.

Several varieties of pesticides have been used in Bangladesh. OPPs are using widely throughout the world. Although the uses of OCPs are banned in couple of years ago but still their existence can be detected because of their persistent and bioaccumulation nature. As we know, maize is a one of the major components of livestock feed and grinded maize is mixed with the other feed ingredients. The pesticide residues in grains are mostly located in the outer layer of grains⁸. Hence, pesticides contents vary upon further processing techniques.

In developing countries, dichlorvos is used extensively for domestic pest control⁹. Prolonged

exposure may cause death, genotoxic, neurological, carcinogenic and other health problems. dichlorvos is classified as class 1B, "highly hazardous" by WHO¹⁰. Methyl parathion poisoning may lead to toxicity like sweating, dizziness, vomiting, diarrhea, cardiac arrest and death in extreme cases. Although the uses of POPs (Persistent Organic Pollutants) like DDT & Heptachlor import and production has been banned in Bangladesh but, still several POPs including DDT are present in market with different brand names or label. They remain in the environment for longer period. So, there have possibility to exist these pesticide residues in the crops like maize.

The world's population is increasing day by day. To feed the increasing population, it is necessary to increase the crop production worldwide¹¹. For better crop production it is necessary to apply pesticides. For public health safety, the presence of pesticide residual level in common foods like maize must be monitored. Pesticide residues are detected commonly through GC¹², GC-MS¹³, HPLC¹⁴, and LC-MS/MS¹⁵. LC-MS/MS and GC-MS have excellent detection capacity. In this study, both OCPs and OPPs were detected from maize grain, flour and processed items through GC-MS/MS because of its accuracy and sensitivity. This study will provide important clues about the pesticide residual contamination in food crops like maize and access potential health hazards.

EXPERIMENTAL

Sample collection

Raw maize grain samples, flour and processed maize items were collected from ten sampling sites of Dhaka division, Bangladesh for pesticide residual analysis. A total of 90 samples (30 samples of each category) were randomly collected from the local markets. To avoid any contamination and for protection from moisture, the samples were stored in Glass bottles. Samples were collected during July, 2021 to August, 2021.

Chemicals and reagents

A standard mixture of eight OPPs residues including dichlorvos, MOCAP (Ethoprophos), Disulfoton, Methyl parathion, Ronnel, Dursban (Chlorpyrifos), Tokuthion and Guthion (Azinphos-methyl) were purchased from Sigma-Aldrich, USA. A

reference standard (with 98% purity) was purchased from Sigma-Aldrich, Germany which contains a cocktail of 19 different OCPs residues (Listed in Table 1). Analytical grade chemicals and reagents with highest purity were used in this analysis.

Table 1: Retention time, LOQ, LOD and Coefficient of Determination (R²) values of pesticide residues

Pesticide residues	LOQ($\mu\text{g}/\text{kg}$)	LOD($\mu\text{g}/\text{kg}$)	Coefficient of Determination(R ²)
OPPs			
Dichlorvos	100.04	14.23	0.9997
MOCAP (Ethoprophos)	93.76	23.54	0.999
Disulfoton	48.97	28.18	0.9978
Methyl Parathion	64.02	19.57	0.9965
Ronnel	98.32	22.38	0.9876
Dursban (Chlorpyrifos)	69.92	29.53	0.9786
Tokuthion	92.38	22.09	0.9994
Guthion (Azinphos-methyl)	82.29	26.08	0.9791
OCPs			
Aldrin	78.45	22.68	0.967
p',p'-DDE	68.66	19.65	0.943
p',p'-DDD	78.19	24.78	0.995
Cis-Chordane	66.79	21.65	0.967
Heptachlorepoxide	100.43	28.59	0.954
Heptachlor	98.44	31.24	0.965
p',p'-DDT	83.52	32.31	0.976
Methoxychlor	99.29	21.09	0.999
Endrin	100.98	19.67	0.998
Endosulfan sulfate	89.31	22.65	0.978
HCH	67.63	17.78	0.966
α -Endosulfan	73.23	16.92	0.975
β -Endosulfan	77.43	22.98	0.956
α -HCH (Lindane-I)	63.68	29.54	0.954
β -HCH (Lindane-II)	88.86	27.43	0.955
γ -HCH (Lindane-III)	72.52	18.75	0.999
δ -HCH (Lindane-IV)	82.37	16.66	0.967
Trans-Chordane	78.46	26.54	0.997
Phthalic Acid	94.34	21.23	0.999
Eldrine ketone	87.53	25.65	0.983

Sample processing

Sample processing was started after about 24 h of sample collection. The samples were homogenized first and then sonicated by using ultrasonic Bath (XUB10, Grant Instruments Ltd.) for 30 min just after extraction with acetone (20 mL) from 20 g of homogenized samples. While the extracts were in ultrasonic bath a mixture of dichloromethane: cyclohexane (1:1, 20 mL) was added. Later, the mixture was filtered by using glass wool containing anhydrous sodium sulfate. After that, sodium sulfate was removed through washing with dichloromethane: cyclohexane (1:1, 5 mL).

A rotary evaporator at 40°C was used for sample concentration and the samples were filled up to 2 mL using cyclohexane¹⁶. The processed samples were used for further analysis.

Quality control of the method

All of the glassware was washed with distilled water and cleaner and later was rinsed with acetone. Other tools used in this study were cleaned before and after every use. Working solutions in the range of 0.5 to 2 $\mu\text{g}/\text{mL}$ was prepared from the standard solution and was stored at -20°C. Both method and instrument performance was tested through blank and recovery tests. Blank samples were spiked with two known (50 $\mu\text{g}/\text{mL}$ and 100 $\mu\text{g}/\text{mL}$) standard solutions for recovery performance evaluation. The spiked samples were homogenized, extracted and analyzed using the same procedure of analyzed samples. The minimum amount of sample produced a peak 3 times higher compared to baseline noise was considered as limit of detection. The limit of quantification was identified based on the noise background to peak ratio of 1:10. All of the blank samples were tested for pesticide residues and no traces were detected.

Analysis of pesticide residues

This study was performed at Institute of food Science and Technology, BCSIR, Dhaka, Bangladesh. The pesticide residues were analyzed by using Gas chromatograph (TRACE 1310, Thermo Fisher Scientific, USA) coupled with a Mass Spectrometer (TSQ DUO, Thermo Scientific, USA). Analytical separations were carried out using Trace GOLD™ TG-5MS GC Column (0.25mm X0.25 μm X0.25m). The initial temperature was 90°C for 2 min and later increased to 260°C at 5°C/min and held for 5 minutes. The carrier gas was Helium at 2.1 mL/min flow rate. The temperature of the injection port was 250°C and injection volume was 1 μL . The detector temperature was about 320°C during the sample analysis. The operational condition of the mass spectrometer was electron impact (EI) ionization at 0.2V with 230°C ion source temperature in full scan mode in the range of 45–500 m/z. For analysis of OCPs residues, same column with helium as carrier gas at a flow of 1.2 mL/min was used. Injection port temperature was 230°C. Temperature profile was fixed in the range of 80°C to 290°C with 2 μL injection volume. A Mass Spectrometer (Model: TSQ DUO, Thermo Scientific, USA) was applied to detect spectral data. Each pesticide residues were

identified through comparing the retention times with that of the standards and spectral data. Pesticide residues were quantified using peak heights.

Statistical analysis

Spectral data were collected by Mass Spectrometer. Pesticide residues were analyzed through comparing with the retention times of the standard mix. Analyzed results were organized through Microsoft Excel. Graph Pad Instat software was used for statistical analysis¹⁷.

RESULTS AND DISCUSSION

Method validation

The method validation results indicated that, this method is suitable for analysis of both OPPs and OCPs residues. The percent recoveries of the pesticide residues are shown in Table 2. Recovery values were between 66.74% to 95.09% and 64.79% to 100.65% for OPPs and OCPs pesticides residues respectively. Table 3 represents the retention time, limit of detection, limit of quantification and linearity (R^2) values of the pesticide residues. The LOD values were in the range of 14.23-29.53 $\mu\text{g}/\text{kg}$ and 16.66-32.31 $\mu\text{g}/\text{kg}$ for OPPs and OCPs pesticide residues respectively. The detected LOQ values were in the range of 48.97-100.04 $\mu\text{g}/\text{kg}$ and 66.79-100.98 $\mu\text{g}/\text{kg}$ for OPPs and OCPs pesticide residues respectively. The Coefficient of Determination (R^2) values were in the range of 0.9786-1.000 and 0.943-1.000 for OPPs and OCPs pesticide residues respectively. The chromatogram of OPPs and OCPs standard mix is represented in Fig. 1 and 2 respectively.

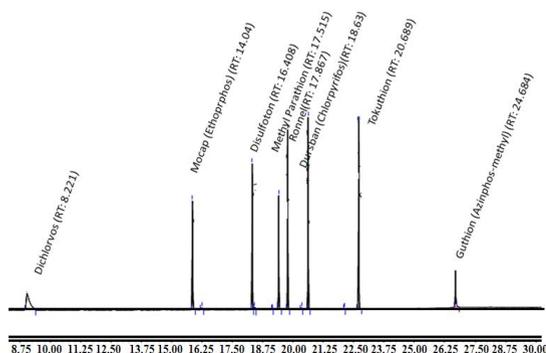


Fig. 1. Chromatogram representing the peaks of Dichlorvos, MOCAP (Ethoprophos), Disulfoton, Methyl parathion, Ronnel, Dursban (Chlorpyrifos), Tokuthion and Guthion (Azinphos-methyl) in standard solution

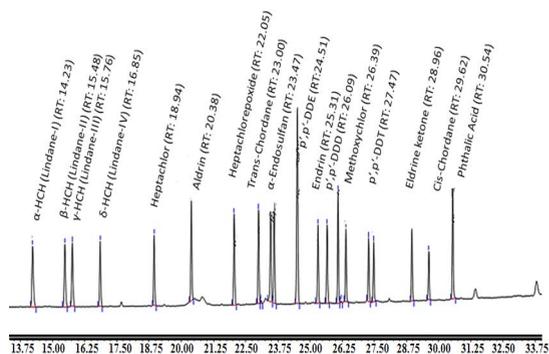


Fig. 2. Chromatogram representing peaks of OCPs pesticide residues in standard solution

Table 2: Pesticide residues and their percent recoveries

Pesticide residues	Spike levels($\mu\text{g}/\text{L}$)	
	50	100
OPP		
Dichlorvos	68.32 \pm 19.76	74.38 \pm 11.87
MOCAP (Ethoprophos)	74.09 \pm 13.98	66.74 \pm 12.32
Disulfoton	88.07 \pm 16.77	91.96 \pm 27.54
Mthyl Parathion	85.98 \pm 17.94	95.09 \pm 15.56
Ronnel	98.97 \pm 21.67	87.54 \pm 14.98
Dursban (Chlorpyrifos)	68.95 \pm 13.94	65.98 \pm 10.76
Tokuthion	75.93 \pm 15.55	78.65 \pm 12.22
Guthion (Azinphos-methyl)	83.21 \pm 19.54	89.62 \pm 11.54
OCP		
Aldrin	92.21 \pm 3.39	86.54 \pm 4.48
p',p'-DDE	67.98 \pm 4.87	88.90 \pm 12.78
p',p'-DDD	89.43 \pm 2.89	93.67 \pm 5.69
Cis-Chordane	81.28 \pm 5.98	67.84 \pm 6.67
Heptachlorepoxide	88.26 \pm 4.87	92.86 \pm 5.68
Heptachlor	73.96 \pm 4.98	72.18 \pm 6.89
p',p'-DDT	77.76 \pm 3.70	64.79 \pm 5.54
Methoxychlor	84.56 \pm 4.39	66.95 \pm 5.45
Endrin	96.97 \pm 5.43	91.56 \pm 6.67
Endosulfan sulfate	99.54 \pm 3.97	87.54 \pm 4.39
HCH	74.68 \pm 5.43	75.64 \pm 4.78
α -Endosulfan	78.39 \pm 6.54	78.54 \pm 5.66
β -Endosulfan	86.98 \pm 7.45	92.09 \pm 6.43
α -HCH	98.40 \pm 6.39	73.16 \pm 6.04
β -HCH	87.54 \pm 6.42	81.32 \pm 4.98
γ -HCH	88.53 \pm 6.39	98.44 \pm 6.62
δ -HCH	92.98 \pm 7.37	92.76 \pm 5.39
Trans-Chordane	97.45 \pm 4.89	100.65 \pm 6.48
Phthalic Acid	90.09 \pm 6.49	88.54 \pm 8.89
Eldrine ketone	83.63 \pm 4.98	73.98 \pm 6.67

Pesticide residues in maize grain, maize flour and maize processed items

All of the collected maize grain samples were processed for pesticide residual analysis. Among the OPPs pesticides, Dichlorvos (964.38 \pm 143.21 $\mu\text{g}/\text{kg}$), Methyl Parathion (43.98 \pm 12.90 $\mu\text{g}/\text{kg}$) and Chlorpyrifos (39.82 \pm 10.75 $\mu\text{g}/\text{kg}$) were detected while

MOCAP (Ethoprophos), Disulfoton, Ronnel, Tokuthion and Guthion (Azinphos-methyl) were not detected in the maize grain samples. OCPs residues including p',p'-DDE (6.54±2.66 µg/kg), p, 'p'-DDT (3.54±2.98 µg/kg) and p',p'-DDT (4.32±2.98 µg/kg) were detected in maize grain samples.

Table 3: Detected concentrations of detected pesticide residues

Pesticide compounds	Mean concentration(µg/Kg)		
	Maize grains	Maize flour	Processed items
Dichlorvos	964.38±143.21	128.65±22.78	ND
MOCAP (Ethoprophos)	ND	ND	ND
Disulfoton	ND	ND	ND
Mthyl Parathion	43.98±12.90	ND	ND
Ronnel	N D	ND	ND
Dursban (Chlorpyrifos)	39.82±10.75	12.54±7.66	ND
Tokuthion	ND	ND	ND
Guthion (Azinphos-methyl)	ND	ND	ND
Aldrin	ND	ND	ND
p',p'-DDE	6.54±2.66	ND	ND
p',p'-DDD	3.54±2.98	ND	ND
Cis-Chordane	ND	ND	ND
Heptachlorepoxide	ND	ND	ND
Heptachlor	ND	ND	ND
p',p'-DDT	4.32±2.98	ND	ND
Methoxychlor	ND	ND	ND
Endrin	ND	ND	ND
Endosulfan sulfate	ND	ND	ND
HCH	ND	ND	ND
α-Endosulfan	ND	ND	ND
β-Endosulfan	ND	ND	ND
α-HCH	ND	ND	ND
β-HCH	ND	ND	ND
γ-HCH	ND	ND	ND
δ-HCH	ND	ND	ND
Trans-Chordane	ND	ND	ND
Phthalic Acid	ND	ND	ND
Eldrine ketone	ND	ND	ND

ND: Not detected.

OCPs residues such as Aldrin, Cis-Chordane, Heptachlorepoxide, Heptachlor, Methoxychlor, Endrin, Endosulfan sulfate, α-Endosulfan, β-Endosulfan, HCH, α-HCH, β-HCH, γ-HCH, δ-HCH, Trans-Chordane, Phthalic acid and Eldrine ketone were not detected in maize grain samples.

Maize flour samples were purchased from the local shops for pesticide residual analysis. Only two OPPs residues including Dichlorvos (128.65±22.78 µg/kg) and Chlorpyrifos (12.54±7.66

µg/kg) were detected in maize flour samples while no OPPs pesticide residues were detected in processed maize items. No OCPs residues were detected in both maize flour and processed maize items. Several food processing steps reduce the pesticide residual concentration in grains¹⁸. Lower concentration of pesticides were detected in maize flour and processed items might be because of the effect of processing or the raw maize used for preparing of flour and processed items contained lower pesticide residues.

The presence of pesticide residues in maize grain and flour samples indicated that, the extensive uses of OCPs in past times and they still exist due to their persistent nature. Several previous studies have reported the presence of OCPs residues in cow pea grains, maize, wheat and rice samples¹⁹⁻²⁷. This study revealed that, there has some pesticide residual contamination in maize grains from different locations of Dhaka, Bangladesh. Both maize flour and processed maize items contained no or very little OPPs residues which might be because of contamination from the raw grains or contamination during storage and processing. The presence of p',p'-DDD in maize samples indicated the uses of the insecticide for pesticide control of insects.

Through breakdown of DDT, DDE enters into the environment. DDE is the main metabolites of DDT. Therefore, the presence of DDE in analyzed samples indicated the uses of DDT during crop production. General population is exposed to DDT through food items. Low DDT dose has very little effect on human body but higher dose may leads to several health problems²⁸ and long term exposures may lead to several severe effects like tumor development and reproductive procedure²⁹.

The detected pesticide residues were below to the maximum residues limits (MRLs)³⁰ which indicated that, there is no potential health risk after consumption of maize grown in those areas. From this analysis, several pesticide residues have been detected maize grains and maize flour but no residues were detected in the processed items. The presence of OCPs residues might be a reflection of past uses of these pesticides that bio accumulated or transferred to the crops from contaminated soil where pesticides were applied. The detected residue levels of OCPs are much lower compared to the

other studies from African countries indicated the less use of those pesticides in Bangladesh compared to those countries.

CONCLUSION

This study revealed the presence of OPPs residual levels in maize grains and flour samples and the presence of OCPs residues were only limited to the grain samples. No pesticide residues were detected in the processed samples collected from local markets of Dhaka, Bangladesh. None of the detected residues exceeds the MRLs values determined by FAO/WHO. Although the level of contamination may not pose danger to human but longer exposure may cause severe effects on

human body. A strict control over import, sales and uses of these POPs is necessary. As food is the major route of human exposure of these pesticide residues therefore proper monitoring of foods is recommended for public health safety.

ACKNOWLEDGMENT

The authors would like to thank Institute of Food Science and Technology, BCSIR, Dhaka, Bangladesh for financial support and research facilities.

Conflict of interest

The authors declare that there is no conflict of interest.

REFERENCES

1. Uygun, U.; Senoz, B.; and Koxsel, H. *Food Chem.*, **2008**, *109*(2), 355–360.
2. Sharma, D.; Nagpal, A.; Pakade, Y. B.; and Katnoria, J. K. *Talanta*, **2010**, *82*, 1077–1089.
3. Patel, K.; Fussell, R. J.; Hetmanski, M.; Goodall, D. M.; Keely, B. J. *J. Chromatogr. A.*, **2005**, *1068*(2), 289–296.
4. Schenck, F.J.; and Donoghue, D.J. *J. Agric. Food Chem.*, **2000**, *48*(12), 6412–6415.
5. Muralidharan, S.; Dhananjayan, V.; Risebrough, R.; Prakash, V.; Jayakumar, R.; and Bloom, P. H. *Bull Environ Contam Toxicol.*, **2008**, *81*(6), 561–565.
6. Srivastava, A. K.; Trivedi, P.; Srivastava, M. K.; Lohani, M.; and Srivastava, L. P. *Environ. Monit. Assess.*, **2011**, *176*, 465–472.
7. The Financial Express online Report. Maize production in Bangladesh rises sharply in last decade (<https://www.thefinancialexpress.com.bd/trade/maize-production-in-bangladesh-rises-sharply-in-last-decade-1605332676>), **2020**, (Accessed on 20-01-2022).
8. Udeaan, A.S.; and Bindra, O.S. Malathion residues in different fractions of treated food grains and their finished derivatives. In: Bindra, O. S., Kalra, R. L. (eds.), Progress and problems in pesticide residue analysis. Punjab Agricultural University and Indian Council of Agricultural Research, Ludhiana., **1973**, 61–67.
9. Binukumar, B.K.; Gill, K.D. *Indian J. Exp. Biol.*, **2010**, *48*, 697–709.
10. WHO: World Health Organization. International Programme on chemical safety. WHO recommended classification of pesticide by hazards and guidelines to classification 1994–1995., **1992**.
11. FAO. The future of food and agriculture: Trends and challenges., **2017**.
12. S. M. Yousefi.; Shemirani, F.; and Ghorbanian, S. A. *Talanta*, **2017**, *168*, 73–81.
13. Chormey, D. S.; Büyükpınar, Ç.; Turak, F.; Komesli, O. T.; and Bakırdere, S. *Environ. Monit. Assess.*, **2017**, *189*(6), 1–10.
14. Harshit, D.; Charmy, K.; and Nrupesh, P. *Food Chem.*, **2017**, *230*, 448–453.
15. Timofeeva, I.; Shishov, A.; Kanashina, D.; Dzema, D.; and Bulatov, A. *Talanta*, **2017**, *167*, 761–767.
16. Åkerblom, M. Environmental Monitoring of Pesticides Residues, Guidelines for the SADC Region, Swedish Science Press, Uppsala, Sweden., **1995**.
17. Motulsky, H. Graph Pad Software, InStat Guide to Choosing and Interpreting Statistical Tests, GraphPad Software, Inc., San Diego, Calif, USA., **1998**.
18. Kaushik, G.; Satya, S.; and Naik, S.N. *Food Res. Int.*, **2009**, *42*, 26–40.
19. Olufade, Y.A.; Sosan, M.B.; and Oyekunle, J.A.O. *Ife J. Sci.*, **2014**, *16*(2), 161–170.

20. Reksa-Naik, S.N; and Prasad, R. Pesticide residue in organic and conventional food-risk analysis. *Chemical Health and Safety.*, **2006**, *13*, 12-19.
21. Toteja, G. S.; Mukherjee, A.; Diwakar, S.; Singh, P.; and Saxena, B. N. Residues of DDT and HCH pesticides in rice samples from different geographical regions of India: a multicentre study. *Food Addit Contam.*, **2003**, *20*(10), 933-939.
22. Bakore, N.; John, P. J.; and Bhatnagar, P. *Environ. Monit. Assess.*, **2004**, *98*(1), 381-389.
23. Guler, G. O.; Cakmak, Y. S.; Dagli, Z.; Aktumsek, A.; and Ozparlak, H. *Food Chem. Toxicol.*, **2010**, *48*(5), 1218-1221.
24. Mawussi, G.; Sanda, K.; Merlina, G.; and Pinelli, E. *Food Addit Contam Part A Chem Anal Control Expo Risk Assess.*, **2009**, *26*(3), 348-354.
25. Mahugija, J. A. M.; Kayombo, A.; and Peter, R. *Chemosphere.*, **2017**, *185*, 137–144.
26. Akoto, O.; Andoh, H.; Darko, G.; Eshun, K.; and Osei-Fosu, P. *Chemosphere.*, **2013**, *92*(1), 67-73.
27. Sosan, M. B.; Oyekunle, J. A. O.; and Odewale, G. O. *Niger. J. Entomol.*, **2018**, *34*, 25–37.
28. Agency for Toxic Substances and Disease Registry (ATSDR), Toxicological Profile for DDT, DDE, and DDD, Draft for Public Comment. U.S. Department of Health and Human Services., **2002**. (<https://www.atsdr.cdc.gov/toxprofiles/tp.asp?id%481&tid%420>).
29. Harada, T.; Takeda, M.; Kojima, S.; and Tomiyama, N. Toxicity and carcinogenicity of dichlorodiphenyltrichloroethane (DDT), *Toxicol. Res.*, **2016**, *32*(1), 21–33.
30. PFA. Prevention of Food Adulteration Act 1954. Act No. 37 with Prevention of Food Adulteration Rules 1955 and Notification and Commodity Index (16th ed.). Lucknow: Eastern Book., **1954**.