PESTICIDE RESIDUES IN GREEN CHILI AND CORIANDER LEAVES COLLECTED FROM GOPALGANJ DISTRICT OF BANGLADESH

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CERTIFICATE

This is to certify that the thesis entitled ***"PESTICIDE RESIDUES IN GREEN CHILI AND CORIANDER LEAVES COLLECTED FROM GOPALGANJ DISTRICT OF BANGLADESH"*** *submitted to the Department of Agricultural Chemistry, Faculty of Agriculture, Sher-e-Bangla Agricultural University, Dhaka-1207, in partial fulfillment of the requirements for the degree of* ***MASTERS OF SCIENCE (M.S.)*** *in* ***AGRICULTURAL CHEMISTRY****, embodies the result of a piece of bona fide research work carried out by* ***SOURAV OJHA****, Registration No.* ***18-09122*** *under my supervision and guidance. No part of the thesis has been submitted for any other degree or diploma.*

I further certify that any help or source of information, received during the course of this investigation has been duly acknowledged.

December, 2020
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Dedicated to
My
Beloved Parents

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ABSTRACT

The study was aimed to analyze pesticide residues in green chili and coriander leaf collected from five major markets of five Upazila (Kotalipara, Tungipara, Gopalganj Sadar, Kashiani and Muksudpur) of Gopalganj district of Bangladesh. The collected samples were carried to the Pesticide Analytical Laboratory, Pesticide Research & Environmental Toxicology Section, Entomology Division of Bangladesh Agricultural Research Institute (BARI), Gazipur. The collected samples were analyzed using Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) extraction technique and Gas Chromatography (GC) coupled with Electron Capture Detector (ECD) for the determination of selected pesticide residues (cypermethrin, lambda-cyhalothrin, acetamiprid and thiram) in 30 samples of green chili and 30 samples of coriander leaf. Among the thirty (30) analyzed samples of green chili, 5 samples (16.67% of the total number of samples) contained residues of cypermethrin and acetamiprid and all the contaminated samples contained residues above the maximum residue limits (MRLs). Out of 30 analyzed samples of coriander leaf, 5 samples (16.67% of the total number of samples) contained residues of cypermethrin and acetamiprid. Among the five contaminated samples, 3 samples contained residues above the maximum residue limits (MRLs). This study reflects the overall scenario of pesticide residues remain in green chili and coriander leaves collected from different markets of Gopalganj district of Bangladesh, which will help the consumer's and policy makers to be aware of their health and safety.

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LIST OF ABBREVIATIONS

BARI	Bangladesh Agricultural Research Institute
GC-MS	Gas Chromatograph-Mass Spectrometry
ADI	Acceptable Daily Intake
CSN	Committee for Standardization
DAS	Days After Spraying
DLLME	Dispersive Liquid–Liquid Microextraction
d-SPE	dispersive solid phase extraction
ECD	Electron Capture Detector
et al	*et alibi* (and others)
Etc	*et cetra* (and so on)
EU	European Union
FAO	Food and Agriculture Organization
FTD	Flame Thermionic Detector
AOAC	Association of Analytical Communities
HPLC	High Performance Liquid Chromatography
HRI	Hazard Risk Index
LC-MS	Liquid Chromatography-Mass Spectrometry
LOD	Limit Of Detection
LOQ	Limit Of Quantification
MRL	Maximum Residue Limit
PDI	Potential Daily Intake
PSA	Primary Secondary Amine
QuEChERS	Quick, Easy, Cheap, Effective, Rugged and Safe
RTL	Retention Time Locked
SAU	Sher-e-Bangla Agricultural University
SBSE	Stir Bar Sorptive Extraction
TOTAD	Through Oven Transfer Adsorption Desorption
UHPLC-MS/MS	Ultra High Performance Liquid Chromatography-Tandem Mass Spectrometry
WHO	World Health Organization

CHAPTER I

INTRODUCTION

Recently, food habits are changing away from traditional commodities towards high-value food products like vegetables, fruits, spices, fishes, etc. In this regard, now-a-days, along with different fruits and vegetables, different salad and spices crops are attaining acceptance day by day as peoples become much concern regarding their health. Moreover, these salad and spices crops are most frequently grown in our country as they give better return over investment for the farmers. Green chili and coriander leaf are generally used with other food items to make it delicious.

Green chili (*Capsicum frutescens L.*) is one of the most profitable spice crop. It is a good source of vitamin A and C. Green chilis are loaded with antioxidants that protect the body against free radicals by acting as natural scavengers. Green chilies have beneficial effects on the cardiovascular system**.** Its production is highly affected due to nearly 35 species of insect pest's viz. thrips, aphids, whitefly, borers, cutworm, plant bugs and other minor pests besides many fungal disease (Sorensen, 2005).

Coriander leaf (*Coriandrum sativum* L.) a member of the Apiaceae family, is widely used as medicinal plant, possessing nutritional as well as medicinal properties (Laribi *et al*., 2015). Coriander leaf is an annual herb. The root, stem, leaves and fruits all have an aromatic odor that most considered pleasant. Coriander leaf is a popular ingredient in the preparation of ayurvedic medicines (Sharma *et al.*, 2012). Coriander leaf seeds reduce fever and promote a feeling of coolness. Coriander leaf juice is highly beneficial in deficiencies of vitamin A, B1, B2, C and Fe. One or two teaspoons of coriander leaf juice, added to fresh buttermilk, is highly beneficial in treating digestive disorders such as indigestion, nausea, dysentery, hepatitis and ulcerative colitis and it is also helpful in typhoid fever. Dry coriander leaf treats diarrhea and chronic dysentery, as well as being useful in acidity. Chutney made from dry coriander leaf, green chilies, grated coconut, ginger and black grapes without seeds are a remedy for abdominal pain due to indigestion (Jansen, 1981).

Vegetables and different spices crops are attacked by insect pests and diseases during production and storage leading to damages that reduce the quality and the yield. In order to reduce the loss and maintain the quality of different agricultural produces, pesticides are used together with other pest management techniques during crop

production to destroy pests and to prevent diseases. The use of pesticides has increased because they have rapid action to control the insect pests and diseases, and are less labor intensive than other pest control methods (Gilden *et al.*, 2010).

Use of pesticide has become indispensable in increasing vegetable production because of its rapid effect, ease of application and availability. The common pesticides used in agriculture include organophosphates, carbamates and pyrithroids etc. Pesticide residues are a public health concern and have been linked to a range of diseases & disorders (Kumar *et al.*, 2012). Pesticide residues also create some risks of environmental hazards. Pesticide residues above the Maximum Residue Level (MRL) in crops are globally and nationally cause great concern. The MRL is the maximum concentration of a pesticide residue (expressed as mg/kg) recommended by the Codex Alimentarius Commission to be legally permitted in or on food commodities and animal feeds (Codex, 2015).

In the developing country like Bangladesh, agriculture plays a key role in the economic performance of the country, contribution to gross domestic production (GDP, 20.01%) foreign exchange earnings and providing employment (47.3%) to a large segments of the population, particularly for the poor (BER, 2010). Farmers and workers of Bangladesh spray pesticides in crop fields without taking any safety measures. As a results they unawarely absorb the toxic items by inhalation and many other different ways. Our farmers spray pesticides without wearing masks, gloves and others proper clothes. Even making spraying pipe clear, they often blow air by mouth. While applying pesticides over 87% farmers use little or no protective measures (Dasgupta *et al.*, 2005). During and after application, pesticides can enter into human body in different ways. The rate of dermal absorption of pesticide residues of different body parts are scalp (3.7%), forehead (4.2%), ear canal (5.4%), abdomen (2.1%), forearm (1.0%), palm (1.3%), genital area (11.8%) and ball of foot (1.6%) (Ogg *et al.*, 2006).

A pesticide which is a modern chemical input and has become established global practice. Pesticides are used to control pests in order to meet the growing demands of vegetable. Pesticides are the poisonous chemical substances which are used in certain circumstances to kill specific target pests (Wassemann, 1972). Insecticides play a major role in the management of these insect pests. Pest control still depends on the use of different groups of pesticides such as organophosphate which causes bad

effects on human health (Gajbhiye *et al*., 1985 and Rai *et al*., 1980). Pesticide residue refers to the pesticides that may remain on or in food after they are applied to food crops. WHO (2016), define pesticide residue as any substance or mixture of substances in food for man or animals resulting from the use of pesticide and includes any specified derivatives, such as degradation and conversion products, metabolites, reaction products, and impurities that are considered to be of toxicological significance. Pesticide residue is the remaining of pesticide active ingredient, its metabolites or breakdown products present in the environment after its application, spillage or dumping (Dasika *et al.,* 2012). The presence of pesticide residue is a concern for the human consumer as it is a potential harmful effect on other non-target organism than pest and disease (Krikothaile and Spanoghe, 2011).

Now a days, pesticide residue in food has become a consumer issue and the people have the right to know how much pesticide is incorporated in the food they eat. At present, in Bangladesh many research works on pesticide residues in vegetables and other matrices have been conducted in the Pesticide Analytical Laboratory, Entomology Division of Bangladesh Agricultural Research Institute (BARI), Gazipur (Prodhan *et al*., 2018; Kabir *et al*., 2007; Islam *et al*., 2014; Kabir *et al*., 2008; Prodhan *et al.,* 2009; Prodhan *et al.,* 2010).

The identification and quantification of pesticide in the vegetables and spices crops are becoming the public interest. On the other hand, limited records are available on the analysis of pesticide residues remain in green chili and coriander leaf collected from Gopalganj district. Keeping this view, this study has been initiated with the following objectives:

- To collect and process green chili and coriander leaf from different markets of Gopalganj district.
- To determine the types and concentration of different pesticides present in the samples.
- To know whether the levels of detected pesticide residues are above the Maximum Residue Limit (MRL) or not.

CHAPTER II

REVIEW OF LITERATURE

In this chapter challenge has been made to assessment literatures for updating the information regarding the existing status of research and knowledge about the determination of pesticide residues in fruits and vegetables. Available and accessible sources of information have been systematically reviewed and summarized with essential comments as appropriately as possible. In spite of the fact, there have been inadequate source of information, most of the relevant information available in and around Bangladesh was collected and reviewed. It is discovered that most of the information on the aspects searched as mentioned above are mostly available from research station and information of farmers' field condition are scanty. However, a significant number of study-reports on insecticides residues in vegetable crops conducted under farmers' field conditions are available. The studies on the quantification of detected insecticides residues below or above the Maximum Residue Limit (MRL) of vegetables in Bangladesh are rarely reported. With this background, the information collected from different sources have been reviewed and presented below:

Nahar *et al.* (2020) conducted a study to assess the health hazards associated with the residual effect of pesticides in two common vegetables (cauliflower and tomato) collected from five different markets of a northern city of Bangladesh. A total of 80 samples (i.e. 40 of each vegetable) were collected for the analysis of seven major organophosphorus insecticides namely acephate, chlorpyrifos, diazinon, dimethoate, fenitrothion, malathion and quinalphos used in that region. Modified QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) extraction technique and GC-FTD (Gas Chromatography coupled with Flame Thermionic Detector) used for the analysis of the samples. Results indicate that 11 (6 cauliflower, 5 tomato) analyzed samples contained residues which is about 14% of the total number of samples. Most of the samples contaminated diazinon at a level above EU-MRLs. However, health risk assessment based on ADI, the contaminated samples were safe. Continuous monitoring together with a sample traceability system is suggested to protect consumers' health from the cumulative effects of other contaminated dietary products.

Collimore *et al.* (2020) performed a study following the newly modified QuEChERS method for multiresidue determination of organophosphate (OPP) and organochlorine pesticide (OCP) residues in fruits and vegetables. The method incorporated a solvent extraction with acetonitrile followed by partitioning with magnesium sulphate (MgSO4) and sodium chloride (NaCl). The last step involved a dispersive solid phase extraction (d-SPE) clean-up prior to gas chromatography with electron capture detection (GC-ECD) analysis. Apples and lettuce were the selected matrices for fruits and vegetables, respectively in the method development stages. Various combinations of sorbents were tested in the clean-up step. Florisil and MgSO4 (FM) d-SPE proved to be the best combination for the clean-up step for both OCPs and OPPs in both the fruit and vegetable matrices. Recovery values fell within the acceptable range of 70 to 120% (RSD ≤ 20%). The newly modified QuEChERS method works as a cheaper alternative for the analysis of pesticide residues in fruits and vegetables. The efficacy of the method was examined on several fruits and vegetables from the Central Division of Trinidad and Tobago. OCP and OPP residues were found in 61% of the samples, most of which were above the maximum residue limits.

Luo *et al.* (2020) initiated a research programme on the residue analysis, risk assessment and processing factors of tebufenozide in okra fruits under field conditions. A simple LC-MS/MS method was carried out and validated for determining the tebufenozide residues in okra fruits. The recoveries of tebufenozide in okra fruits were > 72% with relative standard deviations of 0.6%-6.1%. The dissipation rates of tebufenozide were different in okra fruits cultivated under open land and greenhouse field conditions because of the discriminating humidity and temperature conditions. The dietary intake of the tebufenozide residues from okra fruit consumption for Chinese shoppers was fairly low, with approximately no potential health risk. The processing factor values of washing, blanching and soaking were all.

Shao *et al*. (2020) conducted a study following a modified QuEChERS protocol with a nitrogen-doped graphitized carbon derived from dicyandiamide sludge as a clean-up sorbent to determine 20 organochlorine pesticides in tomatoes using gas chromatography coupled to a triple quadruple mass analyzer. The modified protocol exhibited the advantages of environmental friendly and low-cost. The use of

dicyandiamide sludge to produce NGB as an adsorbent for QuEChERS opened up a new field for the comprehensive utilization of dicyandiamide waste. Under the optimum conditions, the modified method provided excellent linearity with correlation coefficient higher than 0.9980 and low limits of detection ranging from 0.001 to 0.1 μg/kg. The mean recoveries of the majority of pesticides in tomatoes ranged from 71.2 to 95.3% with relative standard deviation lower than 20%.

Mao *et al*. (2020) carried out an analysis of organophosphorus and pyrethroid pesticides in organic and conventional vegetables using QuEChERS extraction combined with dispersive liquid-liquid micro extraction based on the solidification of floating organic droplet. The key parameters were optimized through orthogonal array experimental design and statistical analysis. The linearity of the calibration curves was satisfied in matrix-matched standard solution with R2 ≥ 0.99. The limits of detection and limits of quantification were 0.3–1.5 and 0.9–4.7 μg/kg, respectively. The average recoveries of pesticides were 61.6–119.4% with relative standard deviations < 16.1%. Furthermore, the method was applied successfully to analyze the pesticides in 15 pairs of organic and conventional vegetables. These results shows the efficiency, reliability and robustness of the developed method.

Alnedhary *et al.* (2020) undertook a research on the quantification of pesticide residues in some vegetables using Gas chromatography-Mass Spectrometry. The method applied for the analysis of four pesticides of different classes; dimethoate (Organophosphorus), fenvalerate (Pyrethroid), difenoconazole (Triazole) and deltamethrin (Pyrethroid) on four types of vegetables (i.e. tomato, potato, cucumber, and carrot). The procedures simply involve the use of acetonitrile containing 1% acetic acid for the extraction, and for cleanup; a manually prepared solid-phase extraction cartridge containing primary secondary amine (PSA) and normal charcoal were used. The validated GC-MS analysis method for the pesticide residues in the selected vegetables has high linearity with R2 ranged from 0.9965 to 0.9999. The precision of the method estimated as relative standard deviation (%RSD) was ≤ 9.4% for all target pesticides which were indicative of the high repeatability of the optimized method. The accuracy calculated as average recoveries (%R) was between 80.52% and 99.63%. LODs for target pesticides in spiked cucumber, tomato, carrot, and potato samples ranged between 0.0950 and 0.5590 ng/g. The combined sample

preparation method is cost-effective and has shown good simplification, recovery and cleanup capacity and proved to be efficient and suitable for the proposed application.

Regassa *et al.* (2020) developed a research programme on the determination of residue levels of DDT and its metabolites in khat and cabbage samples using QuEChERS sample preparation method combined with GC-MS detection. Parameters that primarily affect the extraction efficiency of the analytes were optimized. The significance of the use of cleanup was examined and its optimum amount was found to be 6 mg PSA and 12 mg C18. The optimum values for acetonitrile volume and pH of the sample was found to be 3 mL and 7, respectively. The linearity of the analytical response was acceptable with correlation coefficients of 0.992 or better. The precision associated with the analytical method, expressed as %RSD were lower than 8.6 and 9.1% for the intraday and interday precision, respectively. The limit of detection (LOD) and limit of quantification (LOQ) of the proposed method for cabbage sample were in the range of 2x10-5–4x10-5 mg/kg and 9x10-5–14x10-5 mg/kg, respectively. The LOD and LOQ of the proposed method for khat sample were in the range of 2x10-5–6x10-5 mg/kg and 7x10-5–19x10-5 mg/kg, respectively. The recoveries of the method were ranging from 97.16 to 107.99 for cabbage and 72.1 to 90.55 for khat sample. The analytical applications of this method indicated the presence of p, p'-DDE, p, p'-DDD and p, p'-DDT in both cabbage and khat samples. The amount of p, p'-DDE, p, p'-DDD and p, p'-DDT in cabbage were found to be 0.004, 0.01 and 0.01 mg/kg, respectively. The amount of p, p'-DDE, p, p'-DDD and p, p'-DDT in khat were found to be 0.01, 0.03 and 0.07 mg/kg, respectively. The results indicate that there should be continuous monitoring of DDT and its metabolites residues in cabbage and khat samples which in turn is helpful to assess the potential risk of the residues to consumers' health.

Ramadan *et al.* (2020) conducted a research on the evaluation of pesticide residues in vegetables to determine the pesticide residues in 10 different vegetable commodities from the Asir region, Saudi Arabia. They evaluated 211 vegetable samples, collected from supermarkets between March 2018 and September 2018, for a total of 80 different pesticides using ultrahigh-performance liquid chromatography–tandem mass spectrometry (UHPLC-MS/MS) and gas chromatography–tandem mass spectrometry (GC-MS/MS) after extraction with a multi-residue method (the QuEChERS method). The results were assessed according to the maximum residue limit (MRL) provided

by European regulations for each pesticide in each commodity. All lettuce, cauliflower, and carrot samples were found to be free from pesticide residues. A total of 145 samples (68.7%) contained detectable pesticide residues at or lower than MRLs, and 44 samples (20.9%) contained detectable pesticide residues above MRLs. MRL values were exceeded most often in chili pepper (14 samples) and cucumber (10 samples). Methyl, imidacloprid, metalaxyl, and cyproconazole were the most frequently detected pesticides. Based on the results of this study, we recommend that a government-supported program for the monitoring of pesticide residues in vegetables be established to promote consumers' health and achieve sustainable farming systems.

Abdelbagi *et al*. (2020) performed a study to investigate the presence of pesticide residues in fresh eggplants in Khartoum State, Sudan. Eggplant fruit samples from three different regions in Khartoum State (central vegetable market, east Nile farms, and west Nile farms) were analyzed for residues of commonly used pesticides. Pesticide residues were analyzed by gas chromatography coupled with mass spectrometry and results were expressed in µg/kg fruit. Out of the 11 active ingredients analyzed, residues were identified for four pesticides (imidacloprid, dimethoate, endosulfan (α and β isomers) and 2, 4-D). Levels of omethoate, diazinon, malathion, chlorpyrifos, atrazine, and pendimethalin were below the detection limits. Residues of four insecticides out of the 11 analyzed (imidacloprid, dimethoate, endosulfan (α, β isomers), and 2, 4-D) were detected in the current study. The health implications of these violative levels should be regularly observed along with strict enforcement of laws and regulations coupled with agricultural extension interventions.

Kowalska *et al.* (2020) undertook a research to compare the content of pesticide residues (250) in unprocessed plant products from farms situated in the eastern part of Poland. The content of pesticide residues in the analyzed samples was assayed with the use of the QuEChERS (Quick Easy Cheap Effective Rugged Safe) method combined with HPLC-MS/MS (high performance liquid chromatography with tandem mass spectrometry) analysis. The analyses revealed that among 160 analyzed samples, pesticide residues were detected in 83 samples (approximately 52%), while in 77 samples (approximately 48%), no presence of those substances was noted. In all the samples in which the presence of the sought compounds was identified, their levels

did not exceed the Maximum Residue Levels (MRL). The most often identified ones were azoxystrobin—detected in 36 samples (22.5%), linuron—assayed in 33 samples (20.6%), chlorpyrifos and carbendazim—each detected in 13 samples (8.1%), metalaxyl and metalaxyl M—in 11 samples (6.9%), and acetamiprid—in 7 samples (4.4%).

Lim *et al*. (2020) investigated pesticide residues in 207 agricultural products distributed by direct trade in the northern area of Gyeonggi Province. A total of 94 general agricultural products and 113 eco-friendly agricultural products collected from local grocers and cooperative stores were analyzed by multiresidue method for 263 pesticides using GC (gas chromatography)/ECD (electron capture detector), GC/NPD (nitrogen phosphorus detector), GC-MS/MS (tandem mass spectrometry), LC (liquid chromatography)/PDA (photodiode array detector), LC/FLD (fluorescence detector), LCMS/ MS. All samples showing pesticide residues were general agricultural products collected from local food stores. The pesticide residue levels of 14 samples (6.8%) were below the maximum residue limits (MRLs) and one of them (0.5%) exceeded the MRLs. Sixteen pesticides were detected from samples of the following produce items: spinach, young cabbage, perilla leaves, mallow, cucumber, chives and water dropwort. The safety of the detected pesticides was assessed by monitoring the daily intake estimate (EDI) and the daily intake allowance (ADI) based on the amount of pesticides detected. The ADI percentage range (the ratio of EDI to ADI) was 0.0134-61.6259% and there was no health risk connected with consuming agricultural products in which pesticide residues were detected.

Bempah *et al*. (2020) initiated a research to investigate the organochlorine, organophosphorus and synthetic pyrethroid pesticide residues in fruits and vegetables from markets in Ghana. For this purpose, a total of 309 fruits and vegetable samples, were collected and analyzed by gas chromatography with electron capture detector. The obtained results showed that the predominance of organochlorine followed by organophosphorus and synthetic pyrethroid pesticides in most of the analyzed samples. The detected concentrations of them were most significant in vegetable samples. The results obtained showed that 39.2 % of the fruits and vegetable samples analyzed contained no detectable level of the monitored pesticides, 51.0 % of the samples gave results with trace levels of pesticide residues below the maximum residue limit (MRL), while 9.8 % of the samples were above the MRL. The findings

point to the urgent need to establish reliable monitoring programs for pesticides, so that any exceedance in concentration over environmental quality standards can be detected and appropriate actions taken.

Chaikasem *et al*. (2020) carried out a study to investigate the level of pesticide residues and the potential health risk associated with vegetables, surface water, and sediment obtained from the river basin area. The concentrations of 87 pesticides from four main groups namely; organophosphate, organochlorine, pyrethroid, and persistent organic pollutants (POPs) were verified by triple quadrupole GC-MS/MS. The concentration of Parathion methyl, Methidathion, Bromophos methyl, Chlorfenvinphos, Triazophos, Azinphos ethyl, and D-trans-Phenothrin in the sediment samples ranged from 12.99-19.95 µg kg-1. The surface water sample mainly contains p, p'-DDT <0.012 µg L-1 followed by Endrin and Dieldrin <0.08 µg L-1, and Aldrin, Alpha-BHC, Heptachlor <0.004 µg L-1, respectively. The PTI was detected at 0.4 in the sum of surface water samples. The detectable pesticide residues were found in 95% of 20 vegetable samples. The positive of screening vegetables were most obviously contaminated with organophosphate (95%) followed by pyrethroid (40%), organochlorine (20%) and POPs (5%), respectively. The highest concentration of 0.04 mg kg-1 was recorded for Dicrotophos in Kitchen mint (*Mentha cordifolia* Opiz ex Fresen). Fenpropathrin recorded the lowest concentration of 3.2x10-3 mg kg-1 in corn (*Zea mays* L.). The highest PTI (31.20) was found in corn. The combined risk index of pesticide residues showed significant health risk to humans more than individual risk index. The health risk indices show that the detected pesticides considered a serious public health problem in the studied area, and there is a need to increment their monitoring to reduce their misuse.

Puvvala *et al.* (2020) evaluated a study on the impact of IPM and non-IPM practices were undertaken during *Rabi* season of 2018-19 at College of Horticulture, Venkataramannagudem, West Godavari district, Andhra Pradesh with an objective of estimating the pesticide residue levels in fruits harvested from okra plots. IPM plot of okra includes common IPM practices and need based sequential application of botanicals and bioagents. Whereas, sequential spraying of synthetic pesticides was undertaken in non-IPM plot of okra. Levels of pesticide residues in the okra fruits obtained from IPM and non-IPM plots was estimated at Pesticide Residue and Food Quality Analysis Laboratory (PRFQAL), University of Agricultural Sciences,

Raichur, Karnataka. The residues of imidachloprid (6.7 ppm), thiomethoxam (3.8 ppm), flubendiamide (7.9 ppm), chlorantraniliprole (6.5 ppm) were identified in the harvested okra fruits from non-IPM plot which are far above the maximum residue limits (MRL), however no pesticides were detected in the okra fruits from IPM plot.

Islam *et al.* (2019) carried out a study for the determination of major organophosphate insecticide residues in cabbage samples from different markets of Dhaka. The study was conducted to analyse 4 organophosphorus pesticide namely Chlorpyrifos, Diazinon, Fenitrothion and Quinalphos residues in cabbage. Between September 2016 and March 2017, 50 Cabbage samples were collected from 5 vegetables markets of Dhaka city, namely Rampura kacha bazar, Jatrabari krishi market, Kawran Bazar, Taltola Bazar and Mohammadpur Krishi Market. The collected samples were extracted and analyzed by QuEChERS based Gas Chromatography coupled with Flame Thermionic Detector (GC-FTD) method. Total 6 samples (12%) contained pesticide residues and interestingly all of them were above the MRLs set by EC. Among the four organophosphorus insecticides tested only Chlorpyrifos and Diazinon were detected above the MRLs. This research represents a snapshot situation of contamination of pesticides in one of the common winter vegetables available in Dhaka City's local markets linked to consumer safety.

Islam *et al.* (2019a) initiated a study for the analysis of pesticide residues in bitter gourd using modified QuEChERS extraction coupled with Gas Chromatography. This study was undertaken to monitor the presence of seven organophosphorous pesticide residues like acephate, dimethoate, fenitrothion, chlorpyrifos, quinalphos, diazinon and malathion in bitter gourd. 65 samples were collected from retail markets located at the adjacent area of Jahangirnagar University, Savar, Dhaka, Bangladesh namely Genda bazaar, Savar bazaar, Nayarhat bazaar, Islampur bazaar, Pallibiddut bazaar, Baipayl bazaar and Sreepur bazaar. The samples were extracted by modified quick, easy, cheap, effective, rugged and safe (QuEChERS) method and analyzed by gas chromatography coupled with flame thermo ionic detector (GC-FTD). Among the 65 analyzed samples, eight (12.3% of the total number of samples) were contaminated with pesticide residues and all of them contained residues above Maximum Residue Limit (MRL) set by European Commission (EC). Another fifty-seven samples (87.7% of the total number of samples) contained no detectable pesticide residues of the sought pesticides. The findings from this current study showed the common scenario

of pesticide residues in daily consumed vegetables of Savar, Dhaka, Bangladesh that pointed to the imminent health hazards. Therefore, it is suggested to control the overuse of pesticide in vegetable field strictly as well as to increase the awareness of the harmful effect of pesticide residues in vegetables for the growers and the consumers as well.

Islam *et al.* (2019b) established a method for the determination of major organophosphorus Pesticide Residues in Eggplant using modified QuEChERS Extraction and Gas Chromatography. This investigation was undertaken to scrutinize the entity of seven Organophosphorus pesticide residues like acephate, dimethoate, fenitrothion, chlorpyrifos, quinalphos, diazinon and malathion in eggplant. Seventy eight eggplant samples were collected from retail markets located at the surrounding area of Jahangirnagar University, Savar, Dhaka, Bangladesh namely Genda bazaar, Savar bazaar, Nayarhat bazaar, Islampur bazaar, Pallibiddut bazar, Baipayl bazaar and Sreepur bazaar. The samples were extracted by modified quick, easy, cheap, effective, rugged and safe (QuEChERS) method and analyzed by Gas Chromatography coupled with Flame Thermionic Detector (GC-FTD). Among the seventy eight analyzed samples, nine (11.5%) were contaminated by pesticide residues. Two of them were exceeded the EU-MRL (EC, 2015). Another sixty nine samples (88.5%) were free from the contamination of the sought pesticides. The findings from this current study showed the subsistence of pesticide residues in daily consumed vegetables of Savar, Dhaka, Bangladesh that pointed to the imminent health hazards. So, public awareness about the pesticides and other related matter should be increased for practicing a pesticide free agriculture as well as gain contaminate free environment.

Polat *et al*. (2019) evaluated a study on the analysis of pesticide residues in bitter gourd using modified QuEChERS extraction coupled with Gas Chromatography. This study was undertaken to monitor the presence of seven organophosphorous pesticide residues like acephate, dimethoate, fenitrothion, chlorpyrifos, quinalphos, diazinon and malathion in bitter gourd. 65 samples were collected from retail markets located at the adjacent area of Jahangirnagar University, Savar, Dhaka, Bangladesh namely Genda bazaar, Savar bazaar, Nayarhat bazaar, Islampur bazaar, Pallibiddut bazaar, Baipayl bazaar and Sreepur bazaar. The samples were extracted by modified quick, easy, cheap, effective, rugged and safe (QuEChERS) method and analyzed by gas chromatography coupled with flame thermo ionic detector (GC-FTD). Among the 65

analyzed samples, eight (12.3% of the total number of samples) were contaminated with pesticide residues and all of them contained residues above Maximum Residue Limit (MRL) set by European Commission (EC). Another fifty-seven samples (87.7% of the total number of samples) contained no detectable pesticide residues of the sought pesticides. The findings from this current study showed the common scenario of pesticide residues in daily consumed vegetables of Savar, Dhaka, Bangladesh that pointed to the imminent health hazards.

Tankiewicz (2019) carried out a study on the determination of selected priority pesticides in high water content fruits and vegetables by modified QuEChERS and GC-ECD with GC-MS/MS. Chosen compounds are commonly detected in fruit and vegetable crops, and some of their metabolites have even been found in human urine. In addition, some of them are known or suspected carcinogens according to the International Agency for Research of Cancer. Extraction and clean up parameters were optimized, thus the original QuEChERS method was modified to decrease solvent usage, in accordance with 'green chemistry' principles. The proposed methodology was validated in terms of selectivity, specificity, linearity, precision and accuracy. The obtained limits of detection (LODs) for all investigated pesticides ranged from 5.6 µg/kg to 15 µg/kg and limits of quantification (LOQs) from 17 µg/kg to 45 µg/kg. The obtained data demonstrated the good reproducibility and stability of the procedure in the tested concentration range up to 10 µg/kg, with relative standard deviations (RSDs) lower than 10%. Recoveries for spiked pear samples at LOQ level for each pesticide were from 90% to 107% with RSDs lower than 9.6%.

Hasan *et al.* (2019) conducted a study on the analysis of pesticide residues in selected vegetable collected from wet markets of Bangladesh. Translocation of pesticides in selected vegetable samples was determined using gas chromatography-mass spectrophotometry (GC-MS) procedures and quick, easy, cheap, effective, rugged and safe (QuEChERS) method with acetate buffering (AOAC Official Method 2007.01) was used for sample preparation. Pesticide residues above the maximum residue levels (MRLs) were found in 3 urinal, 2 country bean and 1 tomato samples. The result revealed that country bean collected from Kawranbazar, Dhaka and Jessore sadar wet market contained 44.92 µg/kg and 38.65 µg/kg Dimethoate residue, respectively. Brinjal sample collected from Jatrabari, Dhaka, Khalil bazar and Mithapukur bazar wet market contained 32.54 µg/kg, 25.82 µg/kg and 20.65 µg/kg

Quinalphos residue respectively. Tomato sample collected from Narsingdi sadar wet market contained 8.50 µg/kg Quinalphos residue, which was lower than Maximum Residue Levels (10 µg/kg).

Rani *et al.* (2019) developed a domestic methods for the removal of pesticide residues in chilies. Normally available chilies in the market are treated with five pesticides which includes Bifenthrin, Deltamethrin, Hexaconazole, Lamda cyhalothrin and Profenophos and the treated sample was subjected to four different household methods for removal of residues like T1 (Running Tap water wash, T2 (Boiling for 10 min), T3 (Soaking in 2% salt solution for 10 min), T4 (Soaking in 2% salt solution for 10 min+ boiling for 10 min). The residues present in the sample were analyzed by using QTOF GC/MS instrument. Among the four methods 2% salt solution + boiling method had greatest residue removal effect for Deltamethrin (72.90%) to Hexaconazole (37.23%).

Shen *et al*. (2019) established a method for the determination of five acylpyrazole pesticide residues in edible vegetable oils using gas chromatography-negative chemical ionization-mass spectrometry (GC-NCI-MS). The pesticides were extracted from a sample with acetonitrile under freezing conditions. A simple cleanup step known as QuEChERS was then conducted. After being identified by GC-NCI-MS, the extracts were quantified using an external standard method that employs a matrix correction standard curve. The linearity of the method was good between 20 and 1000 µg/L, and all limits of quantification were less than 2 µg/kg. Recoveries of all pesticides were in the range of 82.7%-112.4% at the three spiked levels of 0.01, 0.02, and 0.05 mg/kg, and all relative standard deviations were not more than 12.3%.

Tankiewicz (2019) has been developed a modified quick, easy, cheap, efficient, rugged and safe (QuEChERS) method coupled to gas chromatography with electron capture detector (GC-ECD) for simultaneous determination of selected electronegative pesticides in fruits and vegetables with high water content. The chosen compounds are commonly detected in fruit and vegetable crops, and some of their metabolites have even been found in human urine. Extraction and clean up parameters were optimized, thus the original QuEChERS method was modified to decrease solvent usage, in accordance with ‘green chemistry’ principles. The proposed methodology was validated in terms of selectivity, specificity, linearity, precision and accuracy. The obtained limits of detection (LODs) for all investigated

pesticides ranged from 5.6 µg kg-1 to 15 µg kg-1 and limits of quantification (LOQs) from 17 µg kg-1 to 45 µg kg-1. The obtained data demonstrated the good reproducibility and stability of the procedure in the tested concentration range up to 10 mg kg-1, with relative standard deviations (RSDs) lower than 10%. Recoveries for spiked pear samples at LOQ level for each pesticide were from 90% to 107% with RSDs lower than 9.6%. The suitability of the developed procedure was tested on various fruit and vegetable samples available on the market at different seasons. The proposed methodology is applicable for detection and monitoring of selected pesticides not only in fruits and vegetables with high water content, but also in samples containing large amounts of pigments and dyes.

Hadiana *et al*. (2019) studied forty-eight pesticide residues from different chemical structures including organochlorine, organophosphorus, organonitrogen, dicarboximides, strobilurin, triazine, pyrethroids, and other chemical groups. Pesticide residues in 85 fruits and vegetables were determined and confirmed by GC-MS. The pesticide was extracted with ethyl-acetate, then, the extracts cleaned using high performance gel permeation column chromatography (GPC) and solid phase column (SPE). The mean recoveries of the pesticides were between 81 and 136%. The reproducibility of the relative standard deviation values was 2.1% and 14.8%. Pesticide residues were more frequently found in vegetables (65.5%) than in fruits (26.7%). The limits of detection and quantification of pesticide residues for the method were ranged from 0.003 to 0.06 µg/g and between 0.01 to 0.1 µg/g respectively. The analyzed samples did not contain residues from the monitored pesticides that were higher than the accepted maximum residue limits (MRLs) as adapted by the FAO/WHO Codex alimentarius commission.

Prodhan *et al.* (2018) conducted a research work on the variability of pesticide residues in eggplant units collected from a field trial and marketplaces in Greece. In total, 120 samples from a trial field and 142 samples from different marketplaces in Thessaloniki, Greece, were collected to estimate the variability of pesticide residues in eggplant units. They were extracted by the QuEChERS method and the residues were determined by LC–MS/MS. For the field samples, the level of estimated cypermethrin and deltamethrin residues were 0.01-0.349 mg/Kg and 0.01-0.097 mg/Kg, respectively; and the unit-to-unit variability factors (VFs) obtained for cypermethrin and deltamethrin residues were 2.54 and 2.51, respectively. The mean

residue levels of both pesticides were higher in the composite samples than in the individual samples. The average VFs for the marketplace samples was 3.89. The eggplant units exposed to pesticides were higher in residues than the non-exposed units.

Prodhan *et al*. (2018a) conducted a study to determine the pre harvest interval (PHI) for quinalphos in Eggplant, Cabbage and Yard long bean; malathion in Eggplant, Yard Long bean and Cauliflower; cypermethrin in Tomato and Yard long bean; and diazinon in Eggplant and Yard long bean depending on Maximum Residue Limit (MRL) set by FAO/ WHO. Five supervised field trials were conducted and sprayed with the field dose (2 ml/L of water) of each pesticide except cypermethrin (1 ml/L of water). Samples were collected at 0, 1, 3, 5, 7, 10, 12, 15 and 18 days after spray. The collected samples were analyzed using Gas Chromatography (GC) coupled with Flame Thermionic Detector (FTD) and Electron Capture Detector (ECD) for the determination of pesticide residues. The level of residues were above MRL up to 10 DAS for quinalphos in Cabbage, 7 DAS in Eggplant, 5 DAS in Yard long bean; for malathion 5 DAS in Yard long bean and Eggplant, 7 DAS in cauliflower; for diazinon 5 DAS in Yard long bean and Eggplant; and 3 DAS for cypermethrin in Yard long bean and Tomato. The determined PHI for quinalphos were 12 DAS in Cabbage and 10 DAS in Eggplant and 7 DAS in Yard long bean; For malathion 7 DAS in Yard long bean and Eggplant and 10 DAS in cauliflower; For diazinon 7 DAS in Yard long bean and Eggplant; For cypermethrin 5 DAS in Yard long bean and Tomato.

Prodhan *et al.* (2018b) conducted a study to quantify the residue loss of Quinalphos, Diazinon and Fenitrothion from eggplant and Malathion from Yard long bean through washing and cooking procedures. Samples were collected from the Research field of Entomology Division of Bangladesh Agricultural Research Institute (BARI). The samples were analyzed using a simple Gas Chromatographic technique. Washing with water reduced 34% Quinalphos, 28% Diazinon and 41% Fenitrothion and heating with water at 100 ^{0}C reduced 95% Quinalphos, 84% Diazinon and 100% Fenitrothion from eggplant. Washing with water reduced 45% Malathion and heating with water at 100 ^{0}C reduced 100% Malathion from Yard long bean. Effect of O_3 sterilizer in reducing pesticide residues from eggplant was also investigated in this study and found that O_3 sterilizer reduced 79.00% Diazinon and 62.50% Quinalphos while

washing with only water reduced 60.50% Diazinon and 40.00% Quinalphos from eggplant.

Prodhan *et al.* (2018c) has been developed and validated a simple and efficient multiple organochlorine pesticide residues analytical method using quick, easy, cheap, effective, rugged and safe (QuEChERS) extraction technique and Gas Chromatography coupled with Electron Capture Detector (ECD) for the determination of 19 organochlorine pesticides (Alpha BHC, Delta BHC, Beta BHC, Gama BHC, Heptachlor, Aldrin, Heptachlor Epoxide, Gama Chlordane, Alpha Chlordane, Alpha Endosulfan, 4,4 DDE, Dieldrin, Endrin, 4,4 DDD, Beta Endosulfan, 4,4 DDT, Endrin Aldehyde, Endosulfan sulphate, Methoxychlor, and Endrin Ketone) in shrimp. The method was validated by evaluating the accuracy, precision and linearity limit of detection (LOD) and limit of quantification (LOQ). The average recoveries of the selected pesticides ranged from 84% to 106% with RSDr ≤ 14% in four fortification levels of 0.05, 0.1, 0.2 and 0.3 mg/Kg. The linearity was ≥0.996 for all of the selected pesticides with matrix matched calibration standards. The LOD ranged from 0.003 to 0.009 mg/Kg and the LOQ was 0.05 mg/Kg. This method was applied successfully for the residue analysis of 40 shrimp samples collected from different market places in Bangladesh.

Fang *et al*. (2018) investigated the sources of vegetables consumed by farmers, their perception of pesticide related food safety risks and the behaviors they engage into protect themselves, and explores the implications for the social co-governance (shehui gongzhi) of food safety emphasized by China's recent Food Safety Law. The investigation site is a county in Yunnan Province where vegetable cultivation is the major source of income and livelihood for local farmers. They surveyed 417 farmers and collected 776 vegetable samples from 377 surveyed farmer households and tested them for organophosphate and carbamate pesticide residues using PR-12N Rapid Detection Instrument for Pesticide Residues. The results indicated that farmers know about the risks caused by pesticides used in vegetables and they avoid these risks by consuming vegetables planted in home gardens or private plots that use little or no pesticides. These private plots vegetables had the lowest positive rate of pesticide residues (6.10%), compared with vegetable samples from commercial farmland (13.73%) and markets (12.66%), and the difference was statistically significant ($X2=9.69$, $0.005<P<0.010$). This indicate that the efforts of farmers to protect

themselves from pesticide-related food safety risks. However, the effect is limited due to the environmental pollution caused by the massive use of pesticides in commercial vegetable growing. But this have a negative impact on the social co-governance of food safety set out in the new Food Safety Law.

Forkuoh *et al.* (2018) conducted a study to assess the level of organochlorine pesticide (OCP) residues in fruits and to determine the potential health risks associated with the exposure to these pesticides. 120 fruits samples (watermelon, pineapple, and banana) were collected from five communities and a local market and analyzed for organochlorine pesticide residues. The results showed that the concentrations ranged from ND (not detectable)–48.22 ng/g for DDTs, ND–19.03ng/g for HCHs, ND–4.10 ng/g for CHLs, ND–22.84 ng/g for Aldrin, and ND–11.53 ng/g for other OCPs. In watermelon, levels of methoxychlor, Aldrin and gamma-hexachlorocyclohexane (HCH) exceeded the maximum residue limits. Aldrin in watermelon could pose potential toxicity to the consumer revealed by estimated health risk. Estimated averagedaily intake for Aldrin was above the acceptable average daily intake.

Freitas *et al.* (2018) conducted a study based on matrix solid-phase dispersion (MSPD) extraction and gas chromatography with electron-capture detection (GC-ECD) to determine pesticide residues in the following tropical fruits: banana, mango, melon, papaya and pineapple. C18, silica gel and ethyl acetate presented the best results in the extraction, clean-up and elution steps, respectively in the MSPD proposed method. To minimize the matrix effect in the chromatographic determination spiked blank samples were used. The validation process was conducted at three different concentration levels of spiked samples (0.50-2.50 µg g-1) in within-a-day and in among days assays. The limits of detection for the pesticides ranged from 4.0 to 23 µg kg-1. The method showed acceptable selectivity, coefficients of correlation higher than 0.997, and recovery between 76-105%. The within-a-day precision was assessed through the relative standard deviation (2.8-19%) for the different levels of spiked samples tested.

Zhijuan *et al.* (2018) developed a method for the determination of 50 pesticides in fruits by gas chromatography-tandem mass spectrometry (GC-MS/MS). The three QuEChERS methods (the original one without buffer, the one with acetate buffer and the one with citrate buffer) were compared. The purification effects of primary

secondary amine (PSA) and SinChERS-Nano column were also investigated. The results showed that the acetate buffer and the citrate buffer had positive influence on the extraction compared to the original method without buffer, and there was no significant difference between the two methods using buffers. As the extraction method, the QuEChERS method using acetate buffer was chosen finally. SinChERS-Nano column was revealed to have a better cleaning effect by comparing the purification effect images and the total ion current (TIC) chromatograms and was chosen for cleanup. The recoveries of methamidophos, acephate, omethoate, chlorothalonil and dicofol were in ranged of 71.2%-129.2%, the other 45 pesticides were ranged from 79.1% to 122.3%. The limits of detection (LODs) were 0.3-3.0 μ g/kg and the limits of quantification (LOQs) were 1.0-10.0 μ g/kg. The method is rapid and suitable for the screening of the 50 pesticide residues in citrus, grapes and other fruit samples.

Hakme *et al.* (2018) performed a study on the analysis of pesticide residues in olive oil and other vegetable oils. Pesticide residue analysis in olive oil presents difficulties due to the high amount of co-eluted compounds resulting in high matrix effect. Different extraction/clean-up methods including gel permeation chromatography, liquid/liquid extraction, solid-phase extraction and other extraction methods are applied to overcome these difficulties. Recent approaches such as the addition of the freezing-out step and the application of Enhanced Matrix Removal-Lipid sorbent (EMR-Lipid) are reported. Gas chromatography and liquid chromatography coupled to mass spectrometry are considered the gold standard technologies covering a wide scope of pesticides. This review recapitulates the methods most widely used for the determination of pesticide residues in vegetable oils. As a continuation of previous reviews, the work conducted is an update review of methods from 2006 in this field, evaluating their strengths and limitations. Main analytical parameters of the different extraction procedures and detection methods are discussed in terms of recoveries, robustness, limit of quantification, and matrix effect.

Ibrahim *et al*. (2018) conducted a study on the determination of Organochlorine Pesticide Residues in Pumpkin, Spinach and Sorrel Leaves Grown in Akwanga, Nasiriya State, Nigeria. They collected leafy vegetable samples of pumpkin leaves, spinach leaves, and sorrel leaves were collected from a farm in Akwanga and were tested for the presence of residues of organochlorine pesticides. By using GC/MS, the

concentrations of all the pesticide residues in the vegetable samples were determined. Among all the samples organochlorine pesticide p, p'-DDT was detected in pumpkin (0.75 mg/kg), spinach (0.319 mg/kg) and sorrel (0.219 mg/kg). ∂-BHC and ɣ-BHC were detected only in pumpkin leaves (0.359 mg/kg and 0.647 mg/kg respectively). Dieldrin was detected in spinach and sorrel (0.124 mg/kg and 0.053 mg/kg respectively). Endrin was detectedin pumpkin (0.732 mg/kg) and Aldrin in sorrel (0.095 mg/kg). All these values were above the maximum residue limit (MRL) value of the pesticides. Endosulfan II was detected in sorrel (0.306 mg/kg) below the MRL. The pesticide residues found in vegetables were above the maximum residue limits (MRLs) that callfor laws to regulate the use and circulation of such chemicals. Routine monitoring of pesticide residues in this study area is necessary for the prevention, control and reduction of environmental pollution, to minimize health risks.

Hayat *et al*. (2018) conducted a study in the Department of Entomology, University of Sargodha, Sargodha, Pakistan during the year 2015 to evaluate the residual level of insecticides. For the simultaneous screening of roughly 22 insecticides in fruits, vegetables, pollen, nectar and water samples in three zones of Punjab Pakistan, optimized analytical methods gas chromatography–mass spectrometry (GC–MS) and high performance liquid chromatography (HPLC) were adopted. 50 samples (34.96%) were found positive for one or more insecticides, out of total of 143 samples analyzed (59 fruits & vegetables, 36 pollen, 36 nectar and 27 water samples). Fruits and vegetables 24(40.67%), pollen 8(22.22%) and nectar 6(16.66%) and water 18(66.67%) samples were found pesticide residue. thirteen insecticides were detected in 27 water-samples of three zones of Punjab (Pakistan) ranging from 0.02 to 0.8 µg/L. Different insecticides (carbosulfan, profenofos, cypermethrin, endosulfan sulfate and chlorpyriphos-methyl) were frequently detected in the fruit and vegetable samples. The results suggest that consumers of Punjab province are exposed to the lower concentrations of insecticides that can cause long-lasting disorders.

Liang *et al*. (2018) performed a study to analyze multiple pesticide residues in vegetables using gas chromatography. In total, 420 vegetables samples of 10 different types of fresh vegetables were analyzed. The pesticide residues that exceeded MRLs of forbidden pesticides found were: carbofuran 0.110 mg/kg (kidney bean) and methamidophos 0.037 /kg (celery), methamidophos 0.037 mg/kg (tomato), aldicarb

0.013mg/kg (kidney bean) in September 2009, omethoate 2.200 mg/kg (celery) in November 2009, carbofuran 0.052 mg/kg (green pepper) in April 2010, parathion 0.056 mg/kg (celery) and carbofuran 0.030 mg/kg (celery) in July 2010. Also, chlorpyrifos used as unforbidden pesticide was most frequently found above MRL, rape (0.820 mg/ kg) and celery (0.365 mg/kg) in January 2009, celery (0.330 mg/kg) in May 2009, lettuce (0.298 mg/kg) in September 2009, rape (0.910 mg/kg) in April 2010 and lettuce (0.230 mg/kg) in July 2010. In addition, cypermethrin used as unforbidden pesticide was found above MRL only once in rape (1.270 mg/kg) in May 2009 and none of unforbidden pesticides above MRL was found in November 2009 and January 2010. Most of the samples (96%) were up to the national standard.

Anna *et al*. (2018) invented a Suspect Screening Strategy by UPLC-Q-Tof-MS for the detection of pesticide metabolites in fruit and vegetable samples. Based on a retrospectively created accurate mass compound database, a suspect screening approach was established for pesticides of high concern applied to a wide scope of plant-derived commodities. The metabolite database contained a total of 648 pesticide metabolites originating from 58 active compounds. In 500 samples from daily routine analysis, 96 samples with positive detects for a total of 26 pesticides were re-analyzed for the occurrence of corresponding metabolites. Forty-seven different phase-I and phase-II metabolites were identified, respectively. The developed metabolite database can be applied for a suspect screening approach for pesticide metabolites identification in all kinds of fruits and vegetables.

Abubakar *et al.* (2018) developed a quick, easy, cheap, effective, rugged and safe technique (QuEChERS) coupled with dispersive solid-phase extraction (dSPE) to overcome the setback challenges experienced by the previous technologies for the determination of pesticide residue. Conclusively, the reviewed QuEChERS-dSPE techniques and the recent cleanup modifications justifiably prove to be reliable for routine determination and monitoring the concentration levels of pesticide residues using advanced instruments such as high-performance liquid chromatography, liquid chromatography–mass spectrometry and gas chromatography–mass spectrometry.

Şeyda *et al.* (2018) used a gas chromatography-tandem mass spectrometry (GC-MS/MS) which has been widely used in recent years and has high separation power, selectivity and ability to identify pesticides. In this analytical method, QuEChERS methodology is used. By this method, 123 pesticide residues as well as their

degradation products were quantitatively assayed by GC-MS/MS and method validations in tomatoe, lemon, lettuce, almonds, raisins, honey, green pepper, milk and flour. As potential reference matrixes for the target tomatoe was selected. The steps of concentration and solvent exchange were performed in the resultant extracts for the purpose of improving analytical performance in terms of recovery, precision, linearity, of reducing the amount of coextracts. To identify and quantify the pesticides, multiple reaction monitoring (MRM) was used. The samples were extracted with 1% acetic acid in acetonitrile, anhydrous magnesium acetate, anhydrous magnesium sulfate and clearing agent. For all pesticides, good linear calibrations with coefficients (R2) ≥0.99 for nearly all of the analytes were obtained. Quantitation limit of most of the pesticides were in the range of 5-10 ng/g, and recovery of the method validation accuracy parameter was done at two different concentrations 10 ng/g and 50 ng/g were 88.6 -99.7% and CV 1.60 – 14.0%.

Hasan *et al*. (2017) initiated a study to quantify pesticide residues in country bean collected from different markets of Dhaka city. The collected samples were analyzed using modified QuEChERS Extraction and Gas Chromatography. They have been detected two organophosphorus insecticides (Dimethoate and Quinalphos) in the analyzed country bean samples. Among the 50 analyzed samples of country bean, 10 samples (20%) contained residues of Dimethoate and Quinalphos, of which 5 samples were above the maximum residue limits (MRLs). Most of the contaminated samples (8 samples) contained residue of Dimethoate.

Sebastian *et al*. (2017) developed a multiresidue QuEChERS method by gas chromatography as well as high-performance liquid chromatography for the determination of pesticides. The results indicated that 27% of the total samples contained pesticide residues above the maximum residue limits of each active ingredient. The maximum estimated daily intake obtained for carbon disulphide (CS2), methamidophos, azoxystrobin and cypermethrin were 0.57, 0.07, 0.06 and 0.05 mg kg−1, respectively, which was higher than their acceptable daily intake. It is concluded that inhabitants of the North Central agricultural area of Chile are not exposed to health risks through the consumption of leafy vegetables with the exception of methamidophos.

Elguetaa *et al*. (2017) conducted a study to investigate pesticide residue concentrations and potential human health risk with 118 vegetable samples. The

pesticide residues were determined using the multi residue QuEChERS method by gas chromatography as well as high-performance liquid chromatography. The results indicated that 27% of the total samples contained pesticide residues above the maximum residue limits of each active ingredient. The maximum estimated daily intake obtained for carbon disulphide (CS2), methamidophos, azoxystrobin and cypermethrin were 0.57, 0.07, 0.06 and 0.05 mg kg-1, respectively, which was higher than their acceptable daily intake.

Akter *et al.* (2017) conducted a study for the determination of pesticide residues in eggplant collected from different local markets of Mymensingh Sadar, Mymensingh. The collected samples were extracted using modified QuEChERS Extraction and analyzed with Gas chromatography. This study reflects the overall scenario of pesticide residue contamination in eggplant available in the local markets of Mymensingh Sadar, Mymensingh. In this study, a simple and efficient multiple pesticide residue analytical method based on QuEChERS extraction and gas chromatography-flame thermionic detector (GC-FTD) was used for the determination of pesticide residues. Among the 50 analyzed samples, 11 (22% of the total number of the samples) contained residues of diazinon, dimethoate, quinalfos, and chlorpyrifos, of which, 2 had multiple pesticide residues and 5 samples contained residue above the European Union maximum residue limit (EU-MRLs). Chlorpyrifos was detected as the most used pesticide in eggplant in the studied area.

Zhang *et al*. (2016) has been developed a rapid, efficient, and environmentally friendly method using quick, easy, cheap, effective, rugged, and safe (QuEChERS) extraction technique combined with ionic liquid-based dispersive liquid-liquid micro extraction (QuEChERS-IL-DLLME) in combination to high-performance liquid chromatography coupled with photodiode array detection (HPLC-PDA) for the determination of six triazole fungicides (triazolone, triadimenol, epoxiconazole, flusilazole, tebuconazole, and diniconazole) in various fruits (pear, apple, and grapefruit). And the proposed method was successfully applied for the determination of trace amounts of triazole fungicides in various fruits including pear, apple, and grapefruit.

Prodhan *et al.* (2016) has been developed and validated a multiresidue analytical method for the determination of pesticide residues in cauliflower collected from different market places in Thessaloniki, Greece. In this study, the liquid

chromatography tandem mass spectrometry (LC-MS/MS) was used for the quantification of pesticide residues at trace levels. Among the 120 analyzed samples, 48 (40% of the total no. of samples) were found to have pesticide residues. The detected pesticides were chlorpyrifos, cypermethrin, deltamethrin and indoxacarb.

Prodhan *et al.* (2016a) has been developed and validated a multiresidue analytical method to determine pesticide residues in cabbage collected from different market places in Thessaloniki, Greece. In this study, the modified QuEChERS extraction in combination to liquid chromatography tandem mass spectrometry (LC-MS/MS) was used for the quantification of pesticide residues at trace levels. Among the 132 analyzed samples, 41 (31% of the total no. of samples) had pesticide residues, of which, 2 had multiple pesticide residues and 39 had single pesticide residues. Of the detected pesticides, three were insecticides (chlorpyrifos, cypermethrin and deltamethrin) and two were fungicides (fluopicolide and propamocarb hydrochloride).

Prodhan *et al.* (2015) undertook an experiment to determine pesticide residues in 72 fresh eggplant samples collected from different market in Thessaloniki, Greece with Liquid Chromatography-Mass Spectrometry by adopting QuEChERS extraction method. Among the 72 analyzed samples, 34 (47 % of the total number of samples) had pesticide residues, of which, 5 had multiple pesticide residues and 29 had single pesticide residue. Only one sample contained residue above the EU-MRLs (European Union- Maximum Residue Limits).

Prodhan *et al.* (2015a) has been developed and validated a precise and an effective analytical method to determine pesticide residues in melon collected from different market places in Thessaloniki, Greece. In this study, the modified QuEChERS extraction in combination to liquid chromatography tandem mass spectrometry (LC-MS/MS) was used for the quantification of pesticide residues at trace levels. In this study, both insecticides and fungicides have been detected in melon samples. Among the 122 analyzed samples, 32 (26% of the total number of samples) were found to have pesticide residues.

Mukherjee *et al*. (2015) carried out a research on the analytical method validation and comparison of two extraction techniques for screening of azoxystrobin from widely used crops using LC–MS/MS, where a simple analytical method was developed and validated in chilli, tomato, grape and mango fruits using liquid chromatography

tandem mass spectrometry. The method comprised of extraction with ethyl acetate and cyclohexane mixture followed by d-SPE cleanup employing modified quick, easy, cheap, effective, rugged and safe (QuEChERS) extraction method and quantified in LC–MS/MS using gradient elution. The method was validated in concentration ranging from 0.01 to 0.1l µg g-1. The recovery of azoxystrobin in different crops was ranging from 84.36 to 95.64 % at three different concentration levels of analytes with relative standard deviation of 4–14 %. The global uncertainty was calculated at limit of quantification level i.e. 0.011 µg g-1. The PHI values of azoxystrobin in chilli, tomato, grape and mango fruits were determined as 4.76, 3.90, 4.06 and 10.74 days respectively.

Biziuk *et al*. (2015) applied a multiresidue methods for the determination of currently used pesticides in fruits and vegetables using QuEChERS technique. They stated that all over the world, applied pesticides are some of the most common pollutants of the environment because of their stability, mobility, their consequent long-term adverse effects on living organisms in general and human health in particular. The analysis of food samples for the presence of pesticides causes a lot of difficulties in consideration of specificity of sample preparation based on multistage operations of purification of sample containing vestigial amount of analyte with simultaneous large amount of interferents.

Satpathy *et al*. (2014) conducted a research on the development and validation of multi-residue analysis of 82 pesticides in grapes and pomegranate as per the requirements of the European Union (EU) and codex alimentarius using GC-MS/MS. They validated the QuEChERS based multi-residue method for the extraction of 82 pesticides belonging to various chemical classes from grapes and pomegranate (commodities with high sugar and low lipid contents). They found that matrix-matched calibration results have demonstrated good reproducibility, robustness and linearity and spiking levels for the recovery experiments as 0.005, 0.01 and 0.1 mg/kg for GC-MS/MS analyses. They also found the mean recoveries mostly ranged between 70 and 110 % (91% on average), and RSD were generally below 12% (7.3% on average).For all compounds LODs were 0.001 to 0.005 mg/kg and LOQs were 0.005 to 0.020 mg/kg.

Islam *et al.* (2014) conducted a survey in intensive vegetable growing area in the Narsingdi district of Bangladesh regarding pesticides used by farmers on three major

vegetables like eggplant, cauliflower, and country bean. On the basis of questionnaires, 23 farmers were interviewed and it was noted that fourteen pesticides belonging to different groups were found to be commonly used on the selected vegetables by the respondent farmers to control the major pests. In two selected locations of Narsinghdi 8.33 to 45.00 percent farmers were recorded to apply different pesticides every day and, in some cases, even twice in a day on vegetables. A total of 42 samples were collected from fields and markets and multiple pesticide residue analysis was done by Gas Chromatography (GC) with Flame Thermionized Detector (FTD) and Electron Capture Detector (ECD). Out of 42 samples, 27 had pesticide residue. Among these 27 samples, 14 samples had pesticide residues above the Maximum Residue Limit (MRL). The detected pesticides were Diazinon, Malathion, Quinalphos, Fenitrothion, Cypermethrin, Fenvalerate and Propiconazole.

Hossain *et al*. (2014) conducted a study to determine the pre harvest interval (PHI) for cypermethrin and acephate in Yard long bean depending on Maximum Residue Limit (MRL) set by FAO/ WHO. Two supervised field trials were conducted and sprayed with the field dose (2 ml/L of water for acephate) and for cypermethrin, it was1 ml/L of water. Samples were collected at 0, 1, 3, 5, 7, 10, 12, and 15 days after spray. The collected samples were analyzed using Gas Chromatography (GC) coupled with Flame Thermionic Detector (FTD) and Electron Capture Detector (ECD). The level of residues were detected up to 10 DAS for cypermethrin (0.096 mg/kg), and 7 DAS for acephate (0.435 mg/kg), however, the level of detected residues for both of the pesticides were above MRLs up to 5 DAS. Therefore, The PHI was determined for both of the pesticides were 7 DAS.

Corteas *et al*. (2013) developed a method for the determination of organophosphorus pesticides in vegetables. Pesticide residues are extracted from samples with a small amount of ethyl acetate and anhydrous sodium sulfate. Analyses are performed by large volume GC injection using the through oven transfer adsorption desorption (TOTAD) interface. The calculated limits of detection for each pesticide injecting 50 µL of extract which is much lower than the maximum residues levels (MRLs). Repeatability studies yielded a relative standard deviation lower than 10% in all cases. The method was applied to the analysis of eggplant, lettuce, pepper, cucumber, and tomato.

Panhwar and Sheikh (2013) carried out a study to assess the pesticide residues of bifenthrin, endosulfan, profenofos, emamectin benzoate, imidacloprid and diafenthiuron in cauliflower through gas Chromatography-µECD and high 44 performance liquid Chromatography (HPLC) analysis. The results revealed that the residual level of pesticides in unwashed unprocessed cauliflower samples are beyond their recommended MRLs, the amount of bifenthrin, endosulfan, profenofos, emamectin benzoate, imidacloprid and diafenthiuron and the respective values were 0.151, 0.671, 0.172, 1.04, 1.011 and 0.052 ppm respectively.

Neetu (2013) conducted a research in order to investigate the chlorinated pesticide in vegetables, cereals and pulses by Gas Chromatography in East National Capital Region, Delhi, India. Most of the collected samples were found to be contaminated with residues of DDT. In some of detected samples DDT exceeded the limit of tolerance prescribed by WHO and FAO.

Chauhan (2012) estimated the residues of five commonly used pesticides (endosulfan, carbendazim, chlorpyrifos, cypermethrin and imidacloprid) in different vegetables collected from Uttarakhand, India. Out of the five pesticides, four of them were insecticides belonging to organochlorine, organophosphate, pyrethroid and nicotine based groups respectively and one was fungicide belonging to the benzimidazole group**.** The analysis revealed that most of the vegetables have endosulfan residues above MRL (maximum residue limit) values followed by carbendazim, chlorpyrifos, imidachloprid and cypermthrin, respectively. Amongst the samples, cauliflower and tomato had carbendazim residues higher than the recommended MRL's.

Kanda *et al.* (2012) conducted a research using Gas Chromatography on the extracts from soil, water and vegetable samples. In soil samples, the concentrations of pesticide residues are lower than 20 µg/kg of dry material. For water samples, contamination levels vary from 0.02 to 1.1 µg/L of dry material with the highest levels for metalaxyl M (1.1 µg/L) and for dimethoate (1 µg/L). In vegetables, the concentrations measured are between 0.01 and 0.1 mg/kg of dry material. All these concentrations are affected by a positive factor of the maximum limits of residues.

Parveen *et al.* (2011) monitored pesticide residues in 120 sample of different fruits including apple, apricot, persimmon, chiku, citrus, grapes, guava, mango, papaya, peach, pulm and pomegranate procured from different selling point of Karachi,

Pakistan. They analyzed the samples for multiple pesticide residue using GC/FID and HPLC/UV. They found that 62.5% of samples contained residues of pesticide while 22% exceeded the maximum residue limit (MRL) according to FAO/WHO.

Bagyalakshmi *et al.* (2011) carried out a research on the determination of dimethoate residue in leafy vegetables (Spinach) using HPLC. They carried out the quantification using RP-HPLC for unprocessed (fresh) and processed (washed, washed and cooked) spinach samples. The extraction was carried out with benzene. They found that unprocessed spinach contains dimethoate residue which was slightly more than the tolerance limit, while the processed sample contains dimethoate residue within the tolerance limit. The washed samples (3 times under ordinary tap water) showed 28-50 % reduction while the cooked samples (boiling for 15 min) showed 58-71 % reduction compared to the unprocessed samples. They said that in case of greens, it should be washed three times with ordinary water and then cooked (cooked for fifteen minutes) so that the pesticide residue is very much reduced and it will be well within the tolerance limit as specified by EPA (Environmental Protection Agency).

Sahoo *et al.* (2011) estimated the propamocarb residues in tomato (*Lycopersicon esculentum*) and soil using QuEChERS method and gas chromatograph-mass spectrometry (GC-MS). They found that propamocarb presented a distinct peak at retention time of 8.962 min. They also determined the limit of quantification (LOQ) of their method which was 0.10 mg/kg.

Prodhan *et al*. (2010) has been developed an analytical method for the determination of pesticide residues in fish using Gas Chromatogarphy-Mass Spectrometry (GC-MS). A total of 125 samples of fish covering Rui, Carp, Shrimp, White fish, and Fin fish were collected from Dhaka, Khulna and Chittagong region of Bangladesh and carried to the Pesticide Analytical Laboratory (PAL), Entomology Division, Bangladesh Agricultural Research Institute (BARI), Gazipur. All samples were extracted and prepared for injection using the standard protocols for multi-residue analyses during September, 2007 to April, 2008. The samples were injected into either GCMS-EI or GCMS-NCI depending on the nature of target insecticides. Results revealed that among 125 samples, 49 had insecticide residues. Out of seven samples from Dhaka, 2 had multiple insecticide residues and 1 had single insecticide residue. The detected carbofuran residues ranged from 0.29 to 1.13 ppm, the residues of diazinon, carbaryl and fenvalerate were 1.38, 0.35 and 0.009 ppm, repectively. Out of 60 samples from

Chittagong, 8 had multiple insecticide residues and 15 had single insecticide residue. The detected residues of carbofuran, diazinon, fenvalerate, chlorpyrifos, heptachlor and DDT ranged from 0.17-0.89 ppm, 0.03-2.75 ppm, 0.01-0.03 ppm, 0.005 ppm, 0.19-1.15 ppm and 0.06-0.52 ppm, repectively. Out of 58 samples from Khulna, 9 had multiple insecticide residues and 14 had single insecticide residue. The detected residues of carbofuran, diazinon and DDT ranged from 0.03-1.25 ppm, 0.02-1.03 ppm and 0.04-0.12 ppm respectively. Thus 22 samples contained insecticide residue above ADI (Acceptable Daily Intake) referring to average body weight of 50 kg/person, of which 02 samples collected from Dhaka, 10 from Chittagong and 10 from Khulna region.

Prodhan *et al.* (2009) estimated organophosphorus and organochlorine insecticide residues in fish sample using electron capture detector (ECD) and flame thermionic detector (FTD) of Gas Chromatograph (GC). They collected sixty eight samples of fish (Rui, Shrimp and others) from Dhaka, Khulna and Chittagong offices of Department of Fisheries including different fish export companies during August, 2008 to July, 2009. The results revealed that among 68 samples, 13 had insecticide residues. For Dhaka, out of six samples 1 had DDT residue which was 0.28 ppm. For Chittagong, out of 23 samples 3 had Diazinon residue ranged from 0.03-0.120 ppm. For Khulna, out of 39 samples 9 had Diazinon residue ranged from 0.04-0.205 ppm.

Kabir *et al.* (2008) developed an analytical method for the determination of residue of diazinon and carbosulfan in brinjal and quinalphos in yard long bean under supervised field trial. The present study was undertaken to detect and quantify the left over residue of Diazinon and Carbosulfan in brinjal and Quinalphos in yard long bean and comparison between the detected residue level with maximum residue level (MRL) set by FAO (1970). Three supervised field trials (two for brinjal and one for yard long bean) were carried out sprayed with the field dose (1.5 ml/L of water) of Diazinon, Carbosulfan, and Quinaiphos. Samples were collected daily after spraying till residue were found. In case of Diazinon, left over residue was found upto 6 days after spray (DAS), and upto 3 DAS, the level of residue was above the MRL. Carbosulfan residue was detected till 7 DAS and the detected quantity of residue was above MRL upto 3 DAS. Left over residue of Quinalphos in yard long bean sample was detected upto 6 DAS and upto 4 DAS the level of residue was above the MRL.

Kabir *et al.* (2008a) conducted an experiment to quantify the purity in respect of active ingredient (AI) of some common insecticides used against vegetable insect pests. A series of analyses were made at Pesticide Research Laboratory, Pesticide Research & Environmental Toxicology Section, Entomology Division, BARI, Gazipur using GC-2010 (with FID and ECD detectors) and HPLC- 20A Prominence (with PDA detector). Tested insecticides were 9 brands of Carbofuran 5G and 3G, 2 brands of Carbaryl 85SP, 3 brands of Carbosulfan 20EC, 8 brands of Malathion 57EC, 9 brands of Diazinon 10G and 60EC, 3 brands of Quinalphos 25EC, 3 brands of Dimethoate 40EC, 4 brands of Cypermethrin 10EC and 7 brands of Chloropyrifos 20EC). These collected insecticides were collected from pesticide traders of Jessore and Tongi region of Bangladesh. Results of the present investigation clearly indicated that most of the marketed insecticides were found to have lower AI (≤60%) than that stated on the label. In some cases AI of unknown chemical was being used. Of 9 tested brands of Carbofuran, 3 were found to have 100% purity, 5 had 75-78% AI and 1 had nothing except carrier. Among 2 tested brands of Carbaryl none had >50% purity while all brands of Carbosulfan were found to have 78-98% purity. Eight brands of Malathion were tested, of which only 2 were found as pure, 5 had 80-90% AI and 1 had 60% AI. Of 9 tested brands of Diazinon, only 1 had >90 AI, while 3 had 80%, 4 had 40% and the other 1 had 10%. All 3 tested brands of Quinalphos provided 68-76% AI, while all Dimethoate had only 15-20% AI. Among 4 brands of Cypermethrin, 3 consisted of 100% AI, only 1 had 65%. Seven brands of Chloropyrifos were analyzed, 2 were found to have 100% purity, 3 had 91-97% and 2 had 85-87% AI.

Frenich *et al.* (2008) conducted a research work for the analysis of 53 pesticides in 200 samples of cucumber, orange, strawberry and olive by using ultra performance liquid chromatography (UPLC) coupled to triple tandem mass spectrometry (MS-MS). They found the mean recoveries ranged from 70-109% with relative standard deviation less than 20%. They also found imidacloprid was mostly used pesticide ranged from .01-1.00 mg/kg.

Kabir *et al.* (2007) conducted an experiment at the Regional Sugarcane Research Station, Gazipur in which carbofuran (2 kg AI/ha) was applied in sugarcane field to document the level of carbofuran residue left in soil and plant samples after different days of application (DAA). Plant and soil samples were analyzed by using GCMS-EI.

Carbofuran residues were found even at 90 DAA both in soil and plant. In case of soil, the amount of carbofuran residues were 24.84, 3.32, 2.12, 0.59, 0.035, 0.02 and 0.005 ppm at 0, 3, 7, 15, 30, 60 and 90 DAA, respectively. In case of plant samples, the lower residue (0.0035 ppm) was observed at 0 DAA compared to those at 3 DAA (0.075 ppm), 7 DAA (0.035 ppm) and 15 DAA (0.015 ppm). At 60 and 90 DAA, residues were the same (0.002 ppm) while at 30 DAA it was 0.0025 ppm. The highest level of carbofuran residue (0.075 ppm) in plant samples was found at 3 DAA which is lower than FAO/WHO recommended MRL (0.1mg/kg crop).

Khan and Ali (2005) investigated a study for the residue analysis of commonly used insecticides on fruits and vegetables grown in NWFP-Pakistan during crop season of 2000 using HPTLC. The initial residues of cypermethrin were 0.67 mg/kg. After 10 days, it was dissipated to 0.10 mg/kg, thus representing a loss of 85%. The samples did not contain any detectable residues 15 days after application. The year 2001, the initial residues of cypermethrin on tomato fruits were found to be 0.87 mg/kg which were reduced to 0.10 mg/kg after 15 days. The initial residue of chlorpyrifos 2.61 mg/kg degraded to 1.02 mg/kg 14 days after application. No residues were detected in the fruits 21 days after application.

Lee (2001) conducted a research to investigate the magnitude of the residue of carbofuran and 3-hydroxy carbofuran in rice in Brazil following furadan 50G insecticide treatment in South Korea. In one trials, total residues were 0.17 mg/kg and the two other trials, no residue was detected (<0.02 mg/kg) after 63 days of the last application.

Lehotay (2000) estimated 22 diverse pesticide residues in green bean and carrot extracts by bench top gas chromatography. The targeted pesticides which were incurred in the samples, included chlorpyrifos, azinphos-methyl, parathion-methyl, diazinon, terbufos, DDE, endosulfan sulfate, carbofuran, carbaryl, propargite, bifenthrin, dacthal, trifluralin, metalaxyl, pendimethalin, atrazine, piperonyl butoxide, diphenylamine, vinclozolin, chlorothalonil, quintozene, and tetrahydrophthelimide (the breakdown product of captan). Average recoveries of the pesticides were 103-107% with relative standard deviations of 14%.

Frank *et al.* (1990) conducted a research to investigate residues of insecticide (organophosphorus, synthetic pyrethroid N-methyl carbamate) and fungicide

(dithiocarbamate, dicarboximide and organochlorine) in 433 composite samples of eggplant, asparagus, carrots celery, cole crops, cucumbers, lettuce, onions, peppers, potatoes, radishes and tomatoes in Ontario, Canada. In 64% samples, no pesticide residues were identified to the limits of detection which ranged from 0.005 to 0.05 mg/kg.

CHAPTER III

MATERIALS AND METHODS

The samples of Green chili and Coriander leaf were collected from different markets of Gopalganj district. Five Upazila were considered for sample collection. Six samples were collected from each Upazila. The collected samples were carried to the Pesticide Analytical Laboratory, Entomology Division, Bangladesh Agricultural Research Institute (BARI), Joydebpur, Gazipur for pesticide residue analysis during November 2019 to February 2020. From sampling to final analysis, the required procedures are described below.

3.1 Study area

The study area included vital five markets of five Upazila (Kotalipara, Tungipara, Gopalganj Sadar, Kashiani and Muksudpur) of Gopalganj district. According to retailers openion, the spice crops come from various places of Gopalganj district and nearby districts also. The area of Gopalganj district is 1489.92 sq. km, located in between 22°50' and 23°01' north latitudes and in between 89°40' and 90°02' east longitudes. It is bounded by Faridpur district on the north, Pirojpur and Bagerhat districts on the south, Narail district on the west, Madaripur and Barisal districts on the east. Total no. of population is 1165273; male 592805, female 572468; Muslim 779962, Hindu 371629, Buddhist 13401, Christian 17 and others 264. Madhumati river flows through the Gopalganj district.

3.2 Sample collection

A total of 60 samples (30 green chili and 30 coriander leaf) were collected for this study. Six samples of green chili and six samples of coriander leaf were collected from each market. Each sample was 0.5 kg for both green chili and coriander leaf. Transparent airtight clean polyethylene bags were used to collect samples and each bag was properly labeled with source, sample number, sample ID, collection date, location etc. To avoid cross contamination each sample was collected in a separate polyethylene bag.

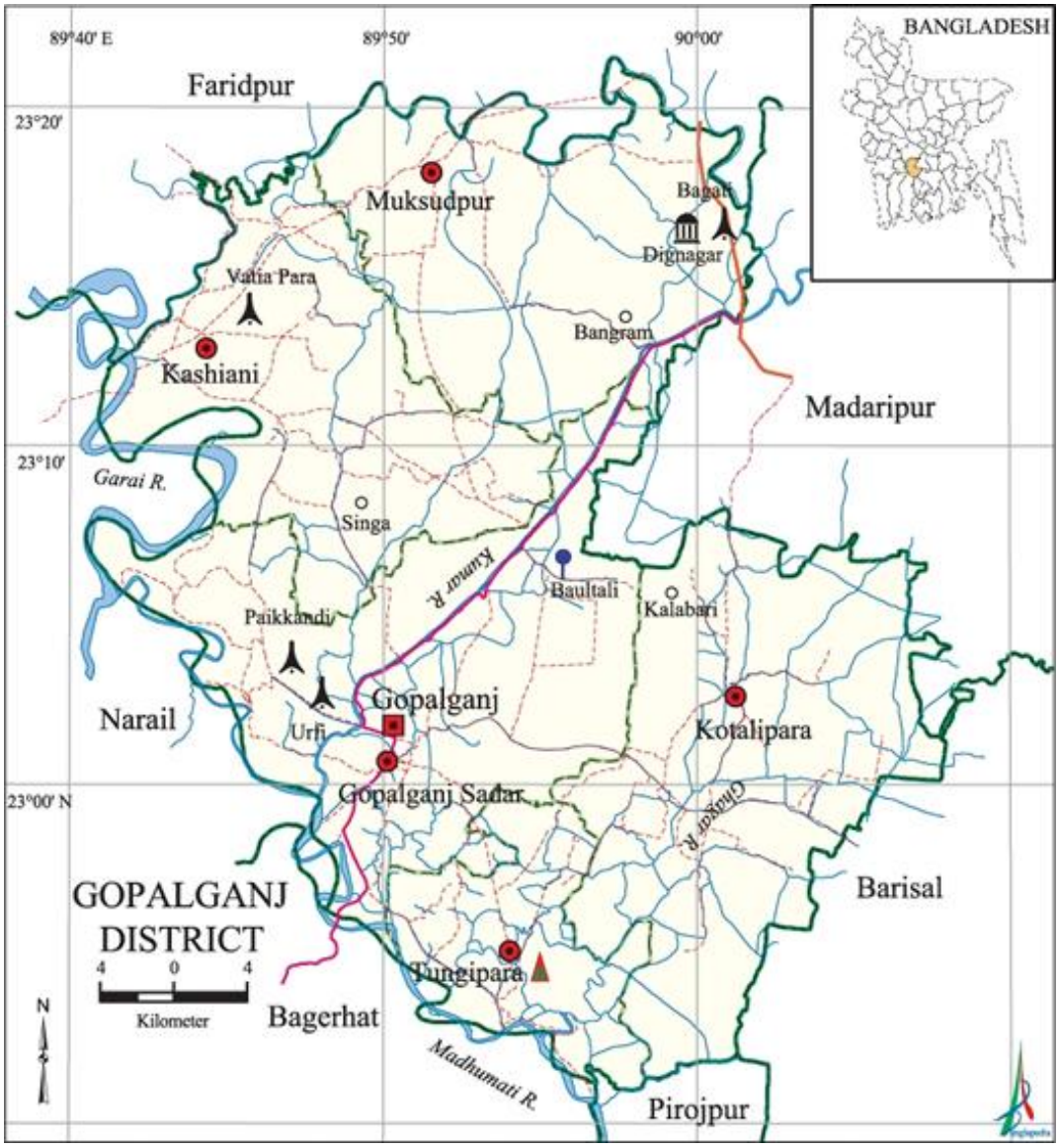


Figure 1. Map showing the places of sample collection

Table 1: Sources and places of collection of green chili samples

Area of collection	Sample ID	Source
Kotalipara	GgChi-01	Ghagar
	GgChi-02	Nazirpur
	GgChi-03	Kandi
	GgChi-04	Radhaganj
	GgChi-05	Agailjhara
	GgChi-06	Kalabari
Tungipara	GgChi-07	Mollahat
	GgChi-08	Chitalmari
	GgChi-09	Dumuria
	GgChi-10	Patgati
	GgChi-11	Nazirpur
	GgChi-12	Faridpur
Gopalganj Sadar	GgChi-13	Chitalmari
	GgChi-14	Dumuria
	GgChi-15	Nazirpur
	GgChi-16	Mollahat
	GgChi-17	Faridpur
	GgChi-18	Korpara
Kashiani	GgChi-19	Nazirpur
	GgChi-20	Faridpur
	GgChi-21	Chitalmari
	GgChi-22	Dumuria
	GgChi-23	Patgati
	GgChi-24	Mollahat
Muksudpur	GgChi-25	Faridpur
	GgChi-26	Chitalmari
	GgChi-27	Dumuria
	GgChi-28	Patgati
	GgChi-29	Agailjhara
	GgChi-30	Nazirpur

* According to the retailer's opinion

Table 2: Sources and places of collection of coriander leaf samples

Area of collection	Sample ID	Source
Kotalipara	GgCo-01	Kalabari
	GgCo-02	Nazirpur
	GgCo-03	Kandi
	GgCo-04	Radhaganj
	GgCo-05	Agailjhara
	GgCo-06	Ghagar
Tungipara	GgCo-07	Nazirpur
	GgCo-08	Chitalmari
	GgCo-09	Dumuria
	GgCo-10	Patgati
	GgCo-11	Mollahat
	GgCo-12	Faridpur
Gopalganj Sadar	GgCo-13	Chitalmari
	GgCo-14	Dumuria
	GgCo-15	Nazirpur
	GgCo-16	Mollahat
	GgCo-17	Faridpur
	GgCo-18	Korpara
Kashiani	GgCo-19	Faridpur
	GgCo-20	Nazirpur
	GgCo-21	Chitalmari
	GgCo-22	Dumuria
	GgCo-23	Patgati
	GgCo-24	Mollahat
Muksudpur	GgCo-25	Agailjhara
	GgCo-26	Chitalmari
	GgCo-27	Dumuria
	GgCo-28	Patgati
	GgCo-29	Faridpur
	GgCo-30	Nazirpur

* According to the retailer's opinion

3.3 Sample preparation for analysis

The collected samples were carried to the Pesticide Analytical Laboratory, Entomology Division, Bangladesh Agricultural Research Institute (BARI), Gazipur on the day of collection. The whole unit of each sample cut into small pieces and mixed properly. Clean air tight polythene bags were used to store chopped sample in refrigerator at - 20°C until extraction and cleanup process started.

3.4 Chemicals and reagents

The standard of cypermethrin, acetamiprid, lambda-cyhalothrin and thiram were obtained from Sigma-Aldrich Laborchemikalien (St Louis, MO, USA) via Bangladesh Scientific Pvt. ltd. Dhaka, Bangladesh. Standards of all the pesticides contained >99.6% purity. Methanol, acetone, HPLC grade acetonitrile (MeCN), sodium chloride (NaCl), anhydrous magnesium sulphate ($MgSO_4$) and Primary Secondary Amine (PSA) were purchased from Bangladesh Scientific Pvt. ltd. Dhaka, Bangladesh.

3.5 Analytical apparatus used

a. Centrifuge machine, Model: Sigma 3k 30, Germany (Plate 1).

b. Electric balance, Model: AY- 220, Shimadzu Corporation, Japan (Plate 2)

c. Vortex mixer, Model: Maxi max ii, USA (Plate 3)

d. GC-2010, Shimadzu corporation, Japan (Plate 4)

Plate 1. Centrifuge Machine

Plate 2: Electric Balance

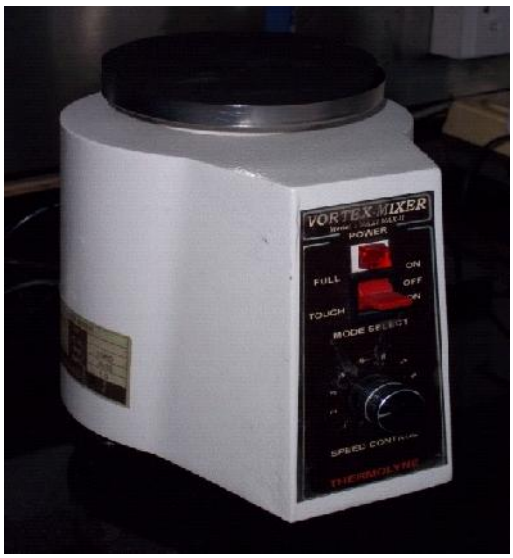


Plate 3: Vortex Mixer

Plate 4: Gas Chromatograph (GC)

In addition to the above instruments the following accessories were also used:

- Centrifuge tube
- Conical flask
- Scissors
- Measuring cylinder
- Volumetric flask
- Tray
- Knife
- Spatula
- Funnel
- Test tube
- Micro pipette
- Aluminum foil
- Para film

3.5.1 Some pictorial view related to sample preparation:

Plate 5: Chopping of Sample

Plate 6: Homogenization of collected Sample

Plate 7. Weighing of Salt (NaCl and anhydrous $MgSO_{4)}$

Plate 8: Adding of Salt (NaCl and anhydrous $MgSO_{4)}$

Plate 9: Shaking of sample

Plate 10. Centrifuging the sample

Plate 11: Filtration through PTFE Filter

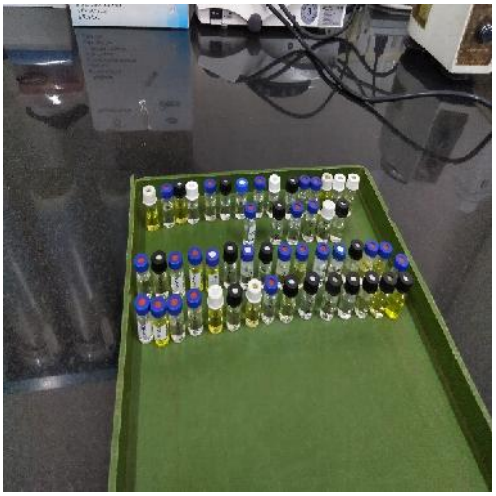


Plate 12. Sample extract ready for injection

3.6 Preparation of pesticide standard solution

Pesticide standard stock solutions of cypermethrin, lambda-cyhalothrin, acetamiprid and thiram were prepared separately in acetonitrile (ACN) at a concentration of 1000 mg/L and stored at -20°C until use. A mixed standard solution of 50 mg/L in ACN containing all the aforementioned pesticides was prepared by adding the appropriate volume of each individual stock solution in a 50 ml volumetric flask and made to volume by addition of acetone. An intermediate mixed standard solution of 10 mg/L in ACN was prepared from the mixed standard solution of 50 mg/L. Then working standard solutions of 0.1, 0.2, 0.5, 1.0, 2.0, 3.0, and 5.0 mg/L in ACN were prepared by transferring the appropriate amount from 10 mg/L intermediate mixed standard solution into ten separate 10-mL volumetric flasks. All the standard solutions were kept in a freezer at -20°C until use.

3.7 Extraction and clean up

QuEChERS extraction method is one of the latest extraction and clean up techniques for pesticide residue analysis in food matrices which is an anagram for Quick, Easy, Cheap, Effective, Rugged and Safe. This techniques was first introduced by Anastassiades *et al*. (2003), which is gaining popularity day by day compared to the other existing techniques such as Supercritical Fluid Extraction (SFE), Liquid-liquid extraction (LLE), Solid phase extraction (SPE), Solid phase micro extraction (SPME), Stir bar sorptive extraction (SBSE), and Microwave assisted extraction (MAE). The technique uses a single extraction in acetonitrile and requires a very small amount of (10-15 gm.) sample. A large excess of salts or buffers are added to extract to aid in the extraction of both polar and non-polar pesticide. This sample initial step simultaneously extract the pesticide from the samples and prepares it for the next dispersive solid phase extraction (d-SPE), the salts and SPE sorbents chosen for the d-SPE step serve to remove residual water and further remove matrix interference from the sample. The resulting acetonitrile extract is typically analyzed directly by gas chromatography-electron capture detector (GC-ECD) with proper dilution.

In this study, the QuEChERS extraction technique was used for the extraction and clean-up of samples which was modified by Prodhan *et al*. (2015). The chopped

samples were grounded thoroughly with the fruit blender. A representative 10g portion of thoroughly homogenized sample was weighted in a 50 mL polypropylene centrifuge tube. Then 10 mL of acetonitrile (MeCN) was added into the centrifuge tube. The centrifuge tube was closed properly and shaken vigorously for 30 s by the use of a vortex mixer. Then, 4 g of anhydrous $MgSO_4$ and 1 g of NaCl were added into the centrifuge tube, and it was shaken immediately by the vortex mixer for 1 minute to prevent the formation of magnesium sulfate aggregates. Afterwards, the extract was centrifuged for 5 min at 5000 rpm. An aliquot of 3 mL of the MeCN layer was transferred into a 15 mL micro centrifuge tube containing 600 mg anhydrous $MgSO_4$ and 120 mg Primary Secondary Amine (PSA). Then it was thoroughly mixed by vortex for 30 s and centrifuged for 5 minutes at 4000 rpm (Laboratory Centrifuges, Sigma-3K30, Germany). After centrifuge, a 1 mL supernatant was filtered by a 0.2 µm PTFE filter, and then it was taken in a clean GC vial for injection.

3.8 Detection and quantification of pesticide residue in samples

The concentrated extracts were subjected to analysis by GC-2010 (Shimadzu) with Electron Capture Detector (ECD) for the detection of cypermethrin, lambda-cyhalothrin, thiram and acetamiprid. The capillary column was Rtx-CLPesticides2, length was 30m, ID was 0.32mm and film thickness was 0.25µm. Nitrogen gas was used as carrier and make up gas for ECD. The identification of suspected pesticide was performed by peak retention times in samples to those of peaks in the pure analytical standards. A typical chromatogram containing selected insecticides is presented in Figure 2 to Figure 5. The instrument conditions are described in Table 3 to Table 7.

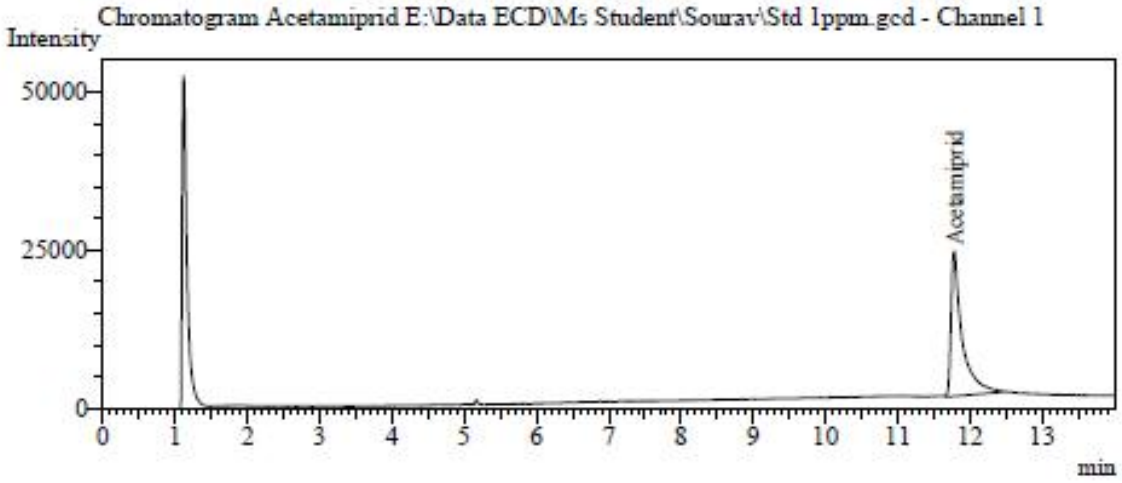


Figure 2: Typical chromatogram of acetamiprid standard run by GC-ECD.

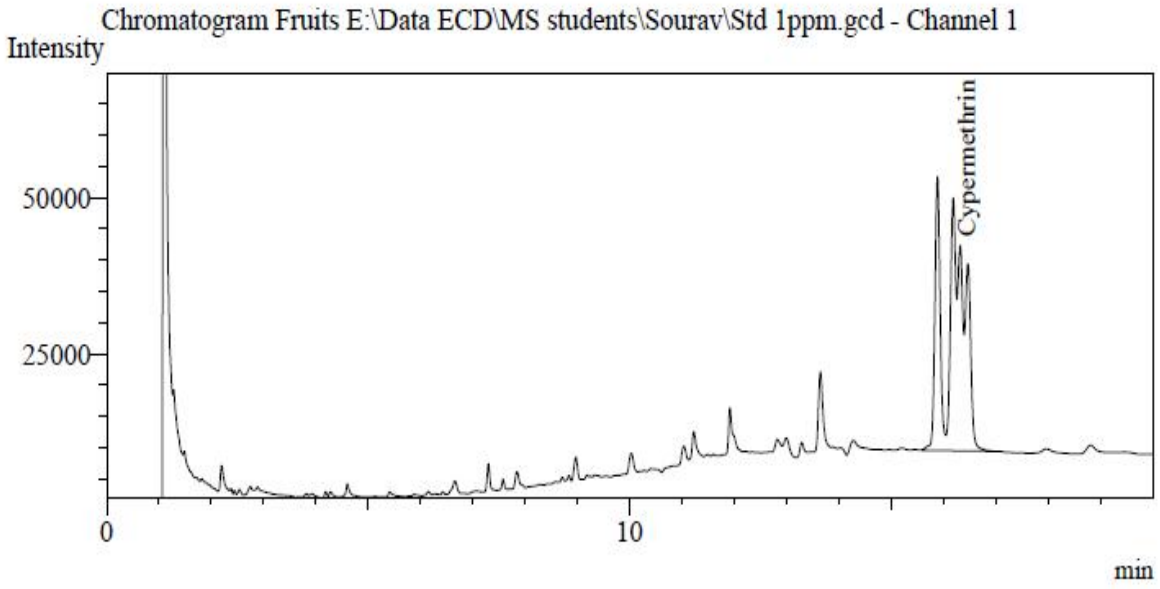


Figure 3: Typical chromatogram of cypermethrin standard run by GC-ECD.

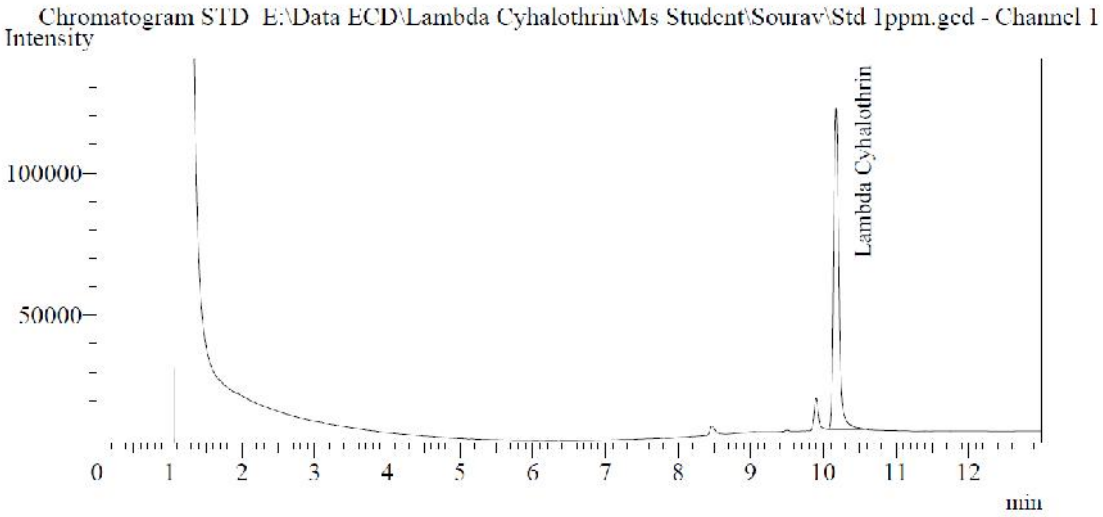


Figure 4: Typical chromatogram of lambda cyhalothrin standard run by GC-ECD

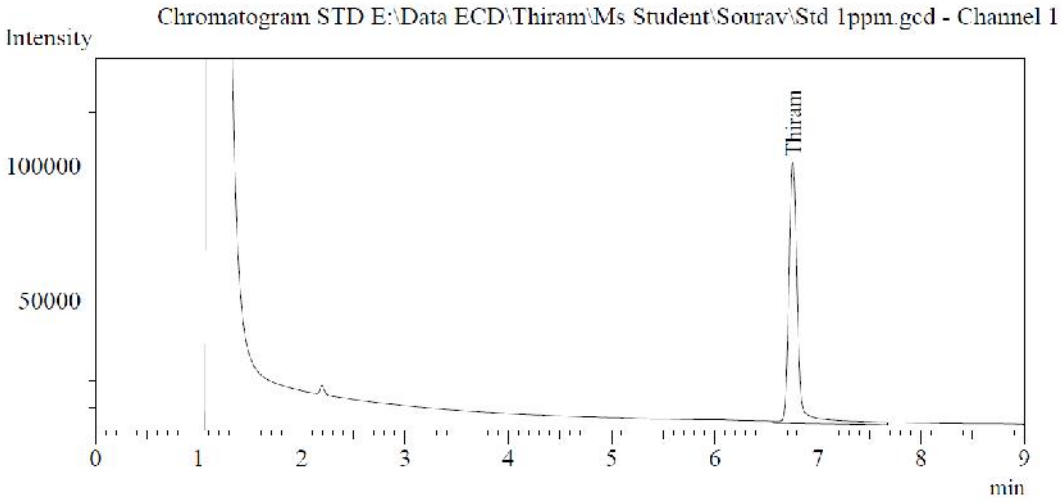


Figure 5: Typical chromatogram of thiram standard run by GC-ECD

Table 3. The instrument parameters for GC-ECD

Instruments	**Conditions**
Injection port SPL	Injection mode: split; temperature:280°C; flow control mode: linear velocity; split ratio: 10:0
Detector channel 1 ECD	Temperature:300°C; current: 0.50 Pa; make up flow: 30 ml/min;

Table 4. Conditions for column oven temperature for acetamiprid determination

Column oven	Rate	Temperature (°C)	Hold time (min)
Initial temperature: 160°C	-	160	0
	15.0	220	0
	10.0	250	0
	5.0	270	3

Table 5. Conditions for column oven temperature for cypermethrin determination

Column oven	Rate	Temperature (°C)	Hold time (min)
Initial temperature: 160°C	-	160	1
	10	270	8

Table 6. Conditions for column oven temperature for thiram determination

Column oven	Rate	Temperature (°C)	Hold time (min)
Initial temperature: 220°C	-	220	0
	5.0	250	3

Table 7. Conditions for column oven temperature for lambda-cyhalothrin determination

Column oven	Rate	Temperature (°C)	Hold time (min)
Initial temperature: 160°C	-	160	0
	15	220	0
	10.0	270	4

3.9 Preparation of Calibration Curve

Prior to the injection of the sample extract, standard solutions of different concentrations of each pesticide group were prepared and injected with suitable instrument parameters. The samples were calibrated (retention time, peak area etc.) against five pointed calibration curve of standard solution of concerned pesticide (Figure 6 to Figure 9). Each peak was characterized by its retention time. Sample results were expressed in mg/kg automatically by the GC software.

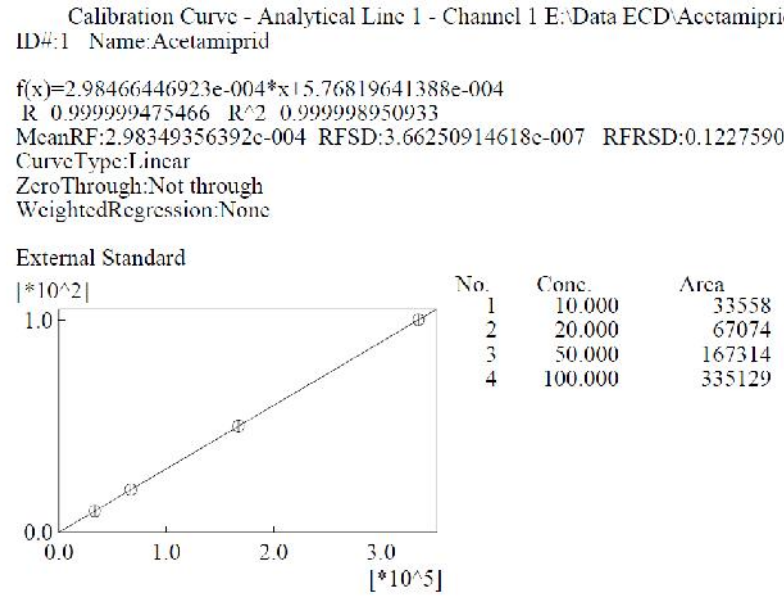


Figure 6. Calibration curve prepared for acetamiprid made with different concentrations ranging from 10 µg/L to 100 µg/L

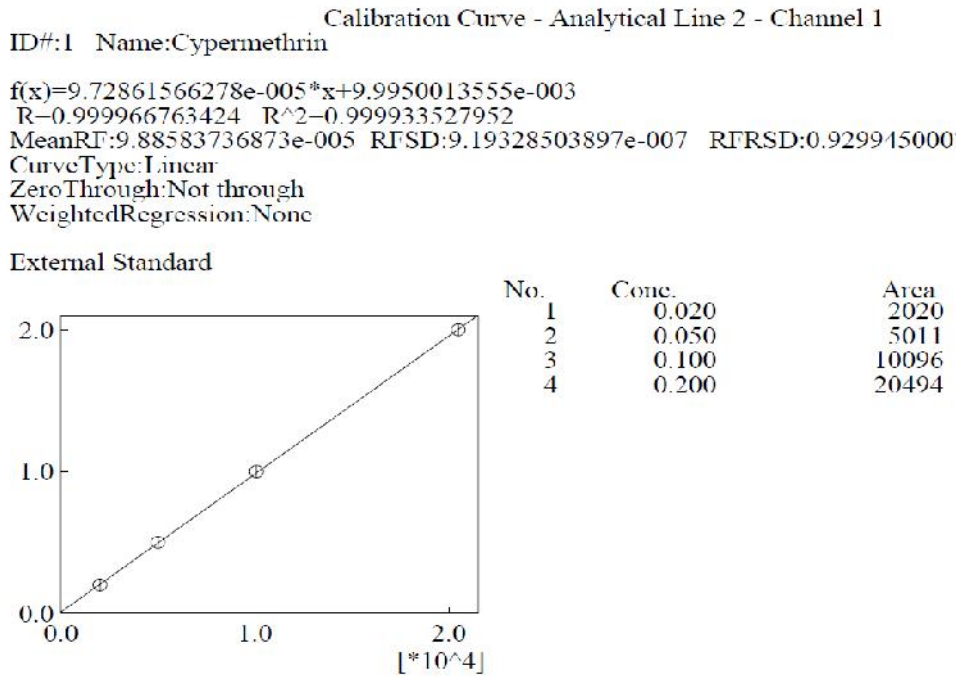
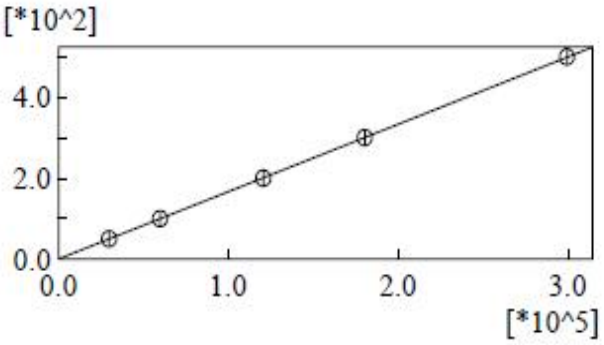


Figure 7. Calibration curve prepared for cypermethrin made with different concentrations ranging from 20 µg/L to 200 µg/L

Calibration Curve - Analytical Line 1 - Channel 1
ID#:1 Name:Lambda Cyhalothrin

f(x)=1.66809373616e-003*x-0.347455682203
R=0.999991464358 R^2=0.999982928789
MeanRF:1.66651033293e-003 RFSD:6.5983219302e-006 RFRSD:0.395936454746
CurveType:Linear
ZeroThrough:Not through
WeightedRegression:None

External Standard



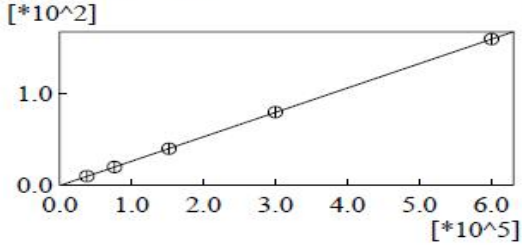
No.	Conc.	Area
1	50.000	29871
2	100.000	59888
3	200.000	120696
4	300.000	180415
5	500.000	299581

Figure 8. Calibration curve prepared for lambda cyhalothrin made with different concentrations ranging from 50 µg/L to 500 µg/L

Calibration Curve - Analytical Line 1 - Channel 1
ID#:1 Name:Thiram

f(x)=2.67125267516e-004*x-0.358918911314
R=0.999994312816 R^2=0.999988625665
MeanRF:2.63979444472e-004 RFSD:2.38482372024e-006 RFRSD:0.903412659654
CurveType:Linear
ZeroThrough:Not through
WeightedRegression:None

External Standard



No.	Conc.	Area
1	10.000	38131
2	20.000	76418
3	40.000	152201
4	80.000	300024
5	160.000	600448

Figure 9. Calibration curve prepared for thiram made with different concentrations ranging from 10 µg/L to 160 µg/L

CHAPTER IV
RESULTS AND DISCUSSIONS

Sixty (60) samples of green chili (30 samples) and coriander leaf (30 samples) were collected from five major markets of five upazila (Kotalipara, Tungipara, Gopalganj Sadar, Kashiani and Muksudpur) of Gopalganj to detect and quantify pesticide residues. The results obtained from this study are presented and described in this chapter using figures and tables.

4.1 Pesticide Residues in green chili

The concentrated extracts of green chili samples collected from different markets of Gopalganj were analyzed by GC-2010 (Shimadzu) with Electron Capture Detector (ECD) with the pre-set parameters. Figure 10-14 shows the chromatograms of the injected extracts of green chili sample containing detected pesticides.

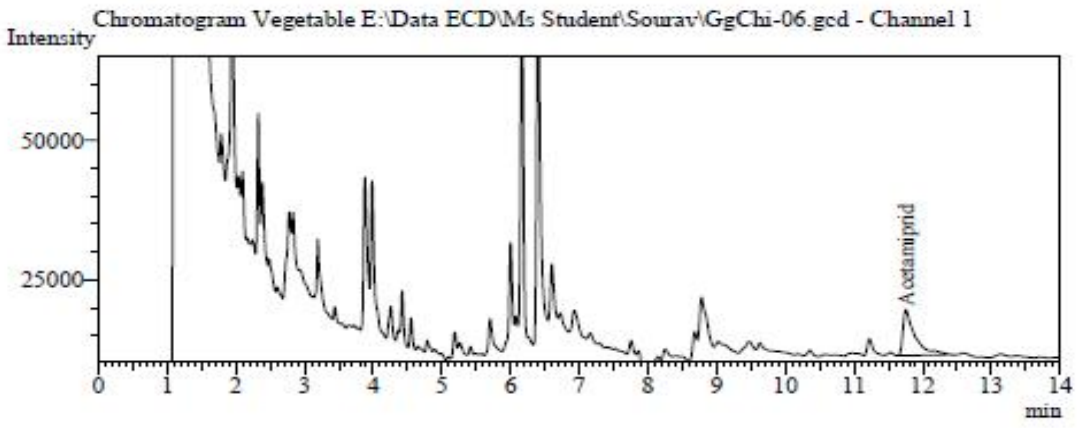


Figure 10. Chromatogram of acetamiprid found in one of the green chili sample (GgChi-06) collected from Kotalipara

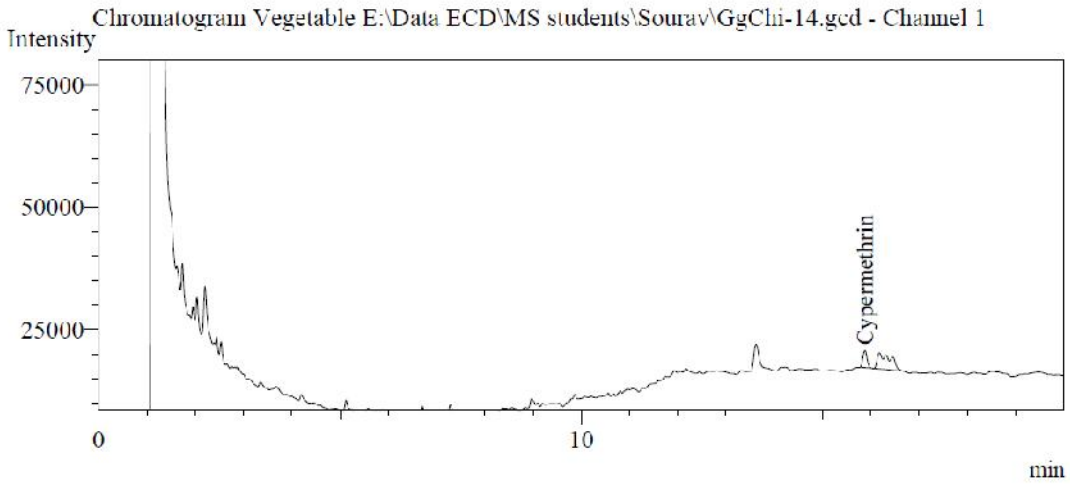


Figure 11. Chromatogram of cypermethrin found in one of the green chili sample (GgChi-14) collected from Gopalganj Sadar

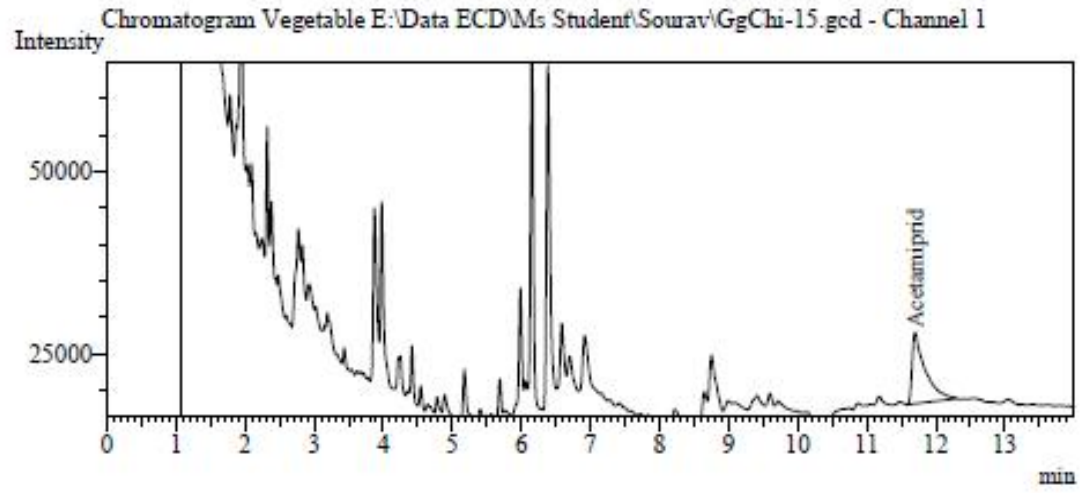


Figure 12. Chromatogram of acetamiprid found in one of the green chili sample (GgChi-15) collected from Gopalganj Sadar

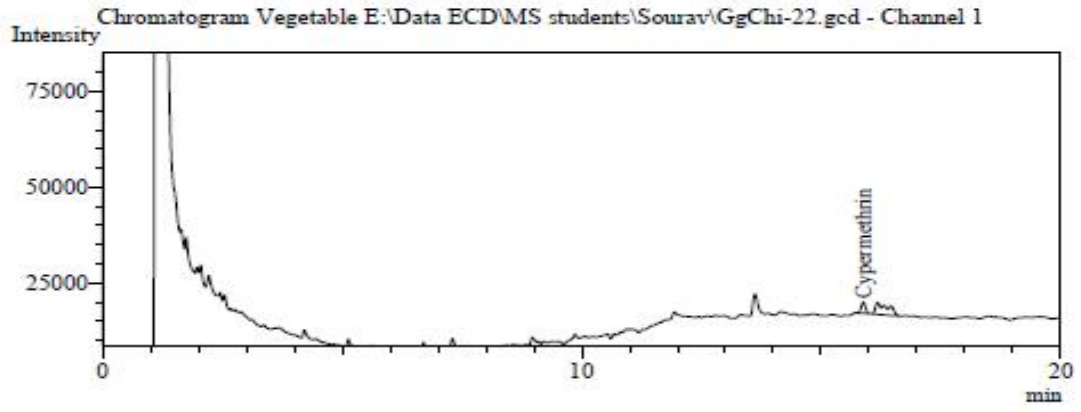


Figure 13. Chromatogram of cypermethrin found in one of the green chili sample (GgChi-22) collected from Kashiani

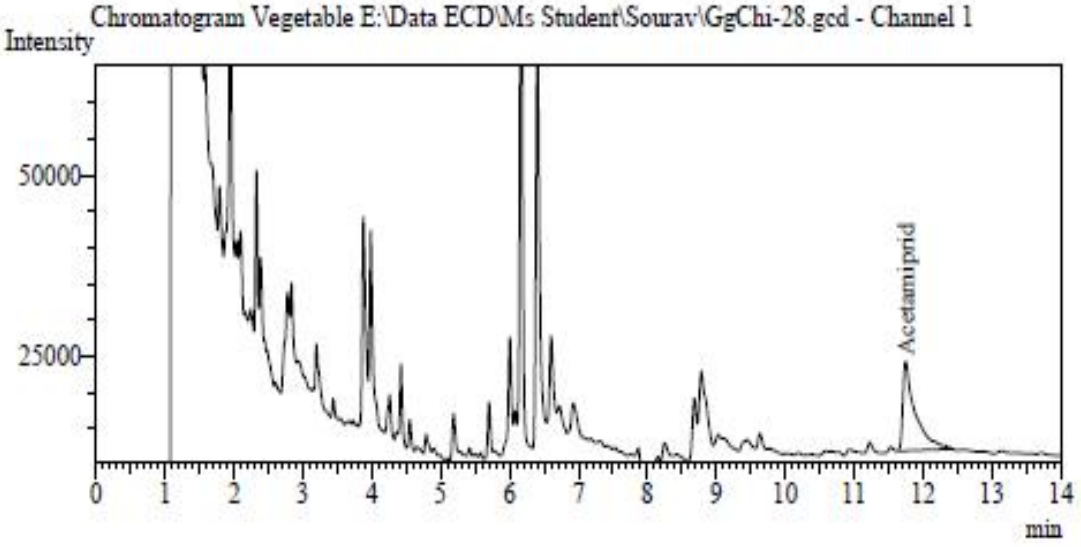


Figure 14. Chromatogram of acetamiprid found in one of the green chili sample (GgChi-28) collected from Muksudpur

Table 8. The level of residues (mg/kg) of different pesticides found in the analyzed green chili samples

Area of collection	Sample ID	Name of detected pesticide	Level of residue (mg/kg)	MRLs (mg/kg)
Kotalipara	GgChi-01	ND		
	GgChi-02	ND		
	GgChi-03	ND		
	GgChi-04	ND		
	GgChi-05	ND		
	GgChi-06	Acetamiprid	0.392	0.01*
Tungipara	GgChi-07	ND		
	GgChi-08	ND		
	GgChi-09	ND		
	GgChi-10	ND		
	GgChi-11	ND		
	GgChi-12	ND		
Gopalganj Sadar	GgChi-13	ND		
	GgChi-14	Cypermethrin	0.086	0.01*
	GgChi-15	Acetamiprid	0.424	0.01*
	GgChi-16	ND		
	GgChi-17	ND		
	GgChi-18	ND		
Kashiani	GgChi-19	ND		
	GgChi-20	ND		
	GgChi-21	ND		
	GgChi-22	Cypermethrin	0.077	0.01*
	GgChi-23	ND		
	GgChi-24	ND		
Muksudpur	GgChi-25	ND		
	GgChi-26	ND		
	GgChi-27	ND		
	GgChi-28	Acetamiprid	0.527	0.01*
	GgChi-29	ND		
	GgChi-30	ND		

*According to the EU Pesticide Database (European Commission-2017)

Thirty samples of green chili collected from major five markets of five Upazila (Kotalipara, Tungipara, Gopalganj Sadar, Kashiani and Muksudpur) of Gopalganj district and were analyzed to find out the presence of left over residue of four pesticides (cypermethrin, lambda-cyhalothrin, acetamiprid and thiram). Out of 30 samples of green chili, 5 samples (16.67% of the total number of samples) contained pesticide residues and 25 samples (83.33% of the total number of samples) contained no detectable residues of the sought pesticides.

The present results can be compared to Islam *et al*. (2019a). They found that among the 65 analyzed samples, 8 (12.3% of the total number of samples) were contaminated with pesticide residues and all of the contaminated samples contained residues above Maximum Residue Limit (MRL) set by European Commission (EC) collected from different markets of Savar, Dhaka of Bangladesh. The results of this study are in a good agreement with Bempa *et al*. (2020). They have been collected 309 fruits and vegetable samples. Among the collected samples 39.2 % of the fruits and vegetable samples contained no detectable level of the monitored pesticides, 51.0 % of the samples gave results with trace levels of pesticide residues below the maximum residue limit (MRL), while 9.8 % of the samples were above the MRL.

The findings of the present study can also be compared with Akter *et al.* (2017). They have been monitored pesticide residues in eggplant collected from Mymensingh district and found that among the 50 analyzed samples, 11 (22% of the total number of the samples) contained pesticide residues of diazinon, dimethoate, quinalfos, and chlorpyrifos, of which, 2 had multiple pesticide residues and 5 samples contained residue above the European Union maximum residue limit (EU-MRLs). Chlorpyrifos was detected as the most used pesticide in eggplant in the studied area. The results of this study are in a good agreement with Hasan *et al*. (2017). They have been detected two types of insecticides (dimethoate and quinalphos) in country bean samples collected from different market places of Dhaka. Among the 50 analyzed samples of country bean, 10 samples (20%) contained residues of dimethoate and quinalphos, of which 5 samples were above the maximum residue limits (MRLs). Most of the contaminated samples (8 samples) contained residue of dimethoate.

From Kotalipara upazila, six green chili samples were collected, among them one samples (GgChi-06) contained acetamiprid at a level of 0.392 mg/kg, which was above the EU-MRL (0.01 mg/kg). The other 5 samples contain no detectable pesticide residues. In the case of Tungipara, six samples were collected. None of the samples contained detectable residues of the sought pesticides.

Among the six samples collected from Gopalganj Sadar, one sample (GgChi-14) contained cypermethrin (0.086 mg/kg) and one sample (GgChi-15) contained residues of acetamiprid (0.424 mg/kg). The level of detected pesticide residue was above EU-MRL (0.01 mg/kg).

One sample (GgChi-22) of green chili contained residue of cypermethrin (0.077 mg/kg) among the six samples collected from Kashiani upazila, which was above EU-MRL (0.01 mg/kg). The other 5 samples contain no detectable pesticide residues. From Muksudpur upazila, six samples were collected of which one sample (GgChi-28) contained acetamiprid residue (0.527 mg/kg). But other five samples contained no detectable pesticide residues. The level of detected acetamiprid residue was above EU-MRL (0.01 mg/kg).

4.2 Pesticide residues in coriander leaf

The concentrated extracts of coriander leaf samples collected from different markets of Gopalganj district were analyzed by GC-2010 (Shimadzu) with Electron Captured Detector (ECD) with the pre-set parameters. Figure 15-19 shows the chromatograms of the injected extracts of coriander leaf sample containing detected pesticides.

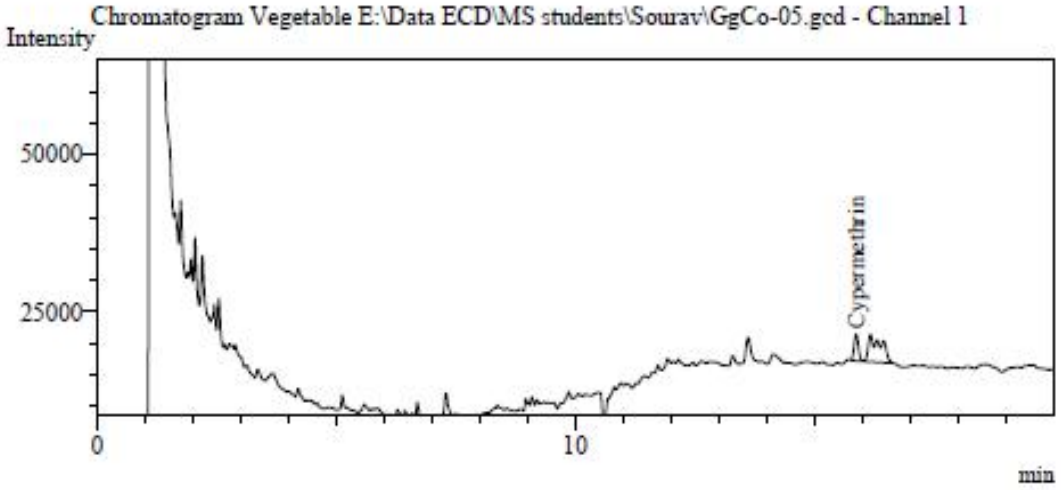


Figure 15. Chromatogram of cypermethrin found in one of the coriander leaf sample (GgCo-05) collected from Kotalipara

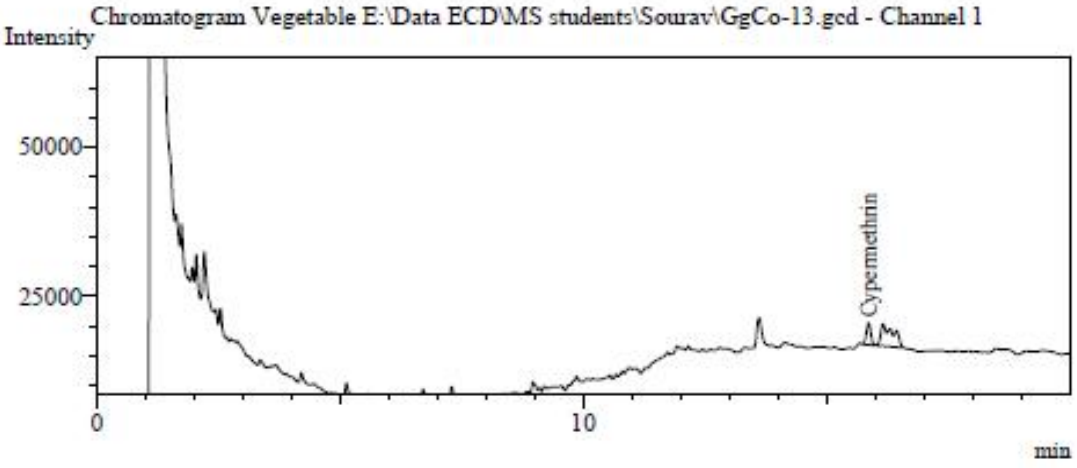


Figure 16. Chromatogram of cypermethrin found in one of the coriander leaf sample (GgCo-13) collected from Gopalganj Sadar

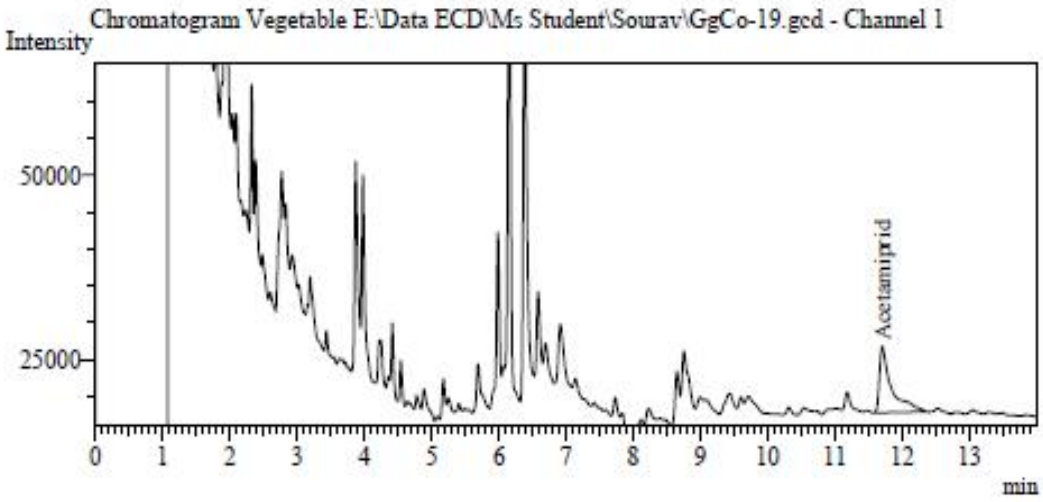


Figure 17. Chromatogram of acetamiprid found in one of the coriander leaf sample (GgCo-19) collected from Kashiani

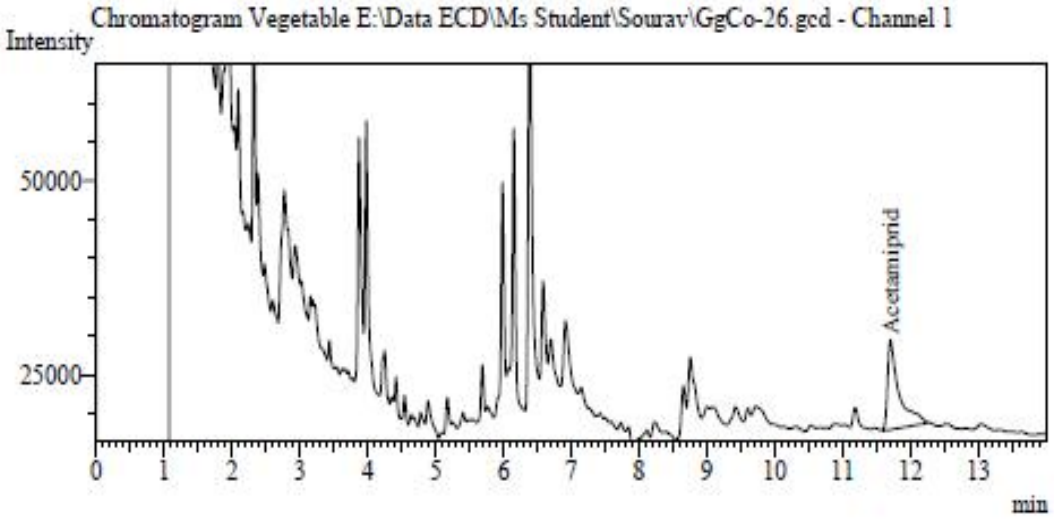


Figure 18. Chromatogram of acetamiprid found in one of the coriander leaf sample (GgCo-26) collected from Muksudpur

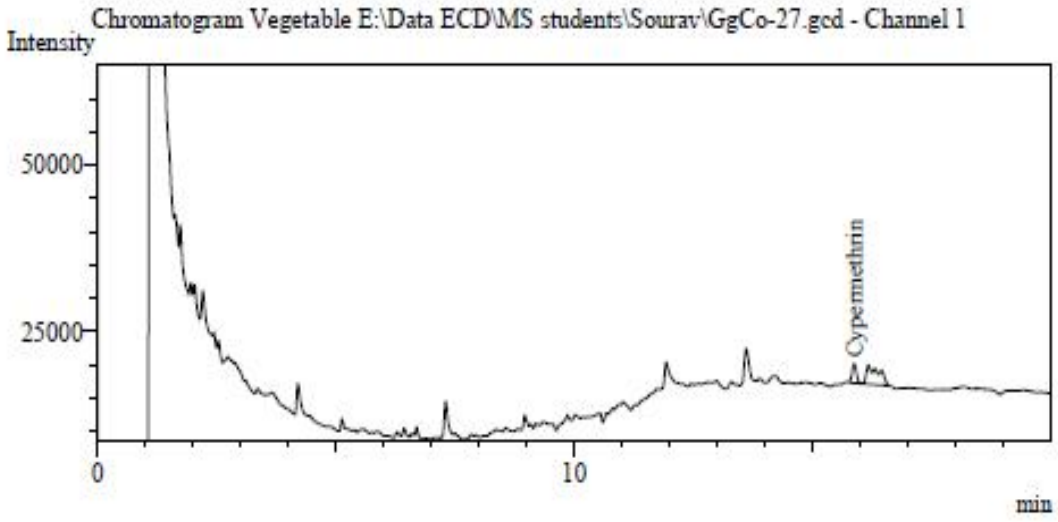


Figure 19. Chromatogram of cypermethrin found in one of the coriander leaf sample (GgCo-27) collected from Muksudpur

Table 9. The level of residues (mg/kg) of different pesticides found in the analyzed coriander leaf samples

Area of collection	Sample ID	Name of detected pesticide	Level of residue (mg/kg)	MRLs (mg/kg)
Kotalipara	GgCo-01	ND		
	GgCo-02	ND		
	GgC0-03	ND		
	GgCo-04	ND		
	GgCo-05	Cypermethrin	0.102	0.1*
	GgCo-06	ND		
Tungipara	GgCo-07	ND		
	GgCo-08	ND		
	GgCo-09	ND		
	GgCo-10	ND		
	GgCo-11	ND		
	GgCo-12	ND		
Gopalganj Sadar	GgCo-13	Cypermethrin	0.095	0.1*
	GgCo-14	ND		
	GgCo-15	ND		
	GgCo-16	ND		
	GgCo-17	ND		
	GgCo-18	ND		
Kashiani	GgCo-19	Acetamiprid	0.456	0.05*
	GgC0-20	ND		
	GgCo-21	ND		
	GgCo-22	ND		
	GgCo-23	ND		
	GgCo-24	ND		
Muksudpur	GgCo-25	ND		
	GgCo-26	Acetamiprid	0.489	0.05*
	GgCo-27	Cypermethrin	0.067	0.1*
	GgCo-28	ND		
	GgCo-29	ND		
	GgCo-30	ND		

*According to the EU Pesticide Database (European Commission-2017)

Thirty samples of coriander leaf collected from 5 major markets of five upazila (Kotalipara, Tungipara, Gopalganj Sadar, Kashiani and Muksudpur) of Gopalganj district and were analyzed to find out the presence of left over residues of four pesticides (cypermethrin, lambda-cyhalothrin, acetamiprid and thiram).

Out of 30 samples of coriander leaf, 5 samples (16.67% of the total number of samples) contained pesticide residues and 25 samples (83.33% of the total number of samples) contained no detectable residues of the sought pesticides. The results of the present study are also supported by the findings of Islam *et al.* (2014), who reported 15 out of total 42 samples (about 35.71%) of eggplant, cauliflower and country bean contained no residues of the sought pesticides collected from fields and markets in Narsingdi district of Bangladesh.

Six coriander leaf samples were collected from Kotalipara upazila, among them, one samples (GgCo-05) contained cypermethrin at a level of 0.102 mg/kg, which was above the EU-MRL (0.1 mg/kg). The other 5 samples contain no detectable pesticide residues. From Tungipara six samples were collected. None of the samples contained detectable residues of the sought pesticides. Six samples were collected from Gopalganj Sadar, one sample (GgCo-13) contained cypermethrin residue (0.095 mg/kg). The level of detected cypermethrin residue was below EU-MRL (0.1 mg/kg). The other 5 samples contained no detectable pesticide residues.

One sample (GgCo-19) of coriander leaf contained residue of acetamiprid (0.456 mg/kg) among the six samples collected from Kashiani upazila, which was above EU-MRL (0.05 mg/kg). The other 5 samples contained no detectable pesticide residues. From Muksudpur upazila, six samples were collected of which one sample (GgCo-26) contained acetamiprid residue (0.489 mg/kg) which was above EU- MRL (0.05 mg/kg). Another one sample (GgCo-27) contained cypermethrin (0.067 mg/kg) which was below EU- MRL (0.1 mg/kg). Rest of the four analyzed samples contained no detectable pesticide residues.

CHAPTER V
SUMMARY AND CONCLUSION

The present study was aimed to identify and quantify selected pesticide residues remain in the samples of green chili and coriander leaf collected from various local markets of Gopalganj district of Bangladesh. Regarding this, thirty (30) samples of green chili and thirty (30) samples of coriander leaf were collected from five major markets of five upazila (Kotalipara, Tungipara, Gopalganj Sadar, Kashiani and Muksudpur) of Gopalganj district and carried to the Pesticide Analytical Laboratory, Entomology Division, Bangladesh Agricultural Research Institute (BARI), Gazipur, Bangladesh. The QuEChERS extraction technique was applied for the extraction and cleanup of the collected samples. Gas chromatography associated with electron capture detector (ECD) was used to identify and quantify the level of pesticide residues present in the extracted samples. Four most commonly used pesticides i.e. cypermethrin, lambda-cyhalothrin, acetamiprid and thiram were selected for this study.

Among the thirty (30) analyzed samples of green chili, 5 samples (16.67% of the total number of samples) contained residues of cypermethrin and acetamiprid and 25 samples (83.33% of the total number of samples) contained no detectable residues of the sought pesticides. All the contaminated sample contained residues of cypermethrin and acetamiprid above the maximum residue limits. In case of coriander leaf, out of 30 analyzed samples, 5 samples (16.67% of the total number of samples) contained residues of cypermethrin and acetamiprid, and 25 samples (83.33% of the total number of samples) contained no detectable residues of the desired pesticides. Among the five contaminated samples, 3 contained residues of detected pesticide (cypermethrin and acetamiprid) above the maximum residue limits (MRLs) and 2 samples contained residues of detected pesticide (cypermethrin) below the maximum residue limits (MRLs).

The results of the present study indicate that the farmers of Gopalganj district are using acetamiprid and cypermethrin indiscriminately. Due to food safety issue, it is a great concern for the consumers. On the other hand, green chili and coriander leaf are almost directly consumed by the consumers, hence, it is necessary to account of these findings and it can be recommended that, the policy planners and the respective stakeholders may take proper action in order to control the indiscriminate use of pesticides for the management of insect pests and diseases of green chili and coriander leaf. It is also suggested that bio-pesticides can be used instead of toxic chemical pesticides for the control of insect pests and diseases.

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