**Short Communication** 

# Analysis of banned organochlorine pesticide residues in fruit and vegetable samples from a market in shillong, Meghalaya, using gas chromatography – mass spectrometry

# Balawan Joshua Mawrie\* and Dipyendu Paul

Department of Environmental Studies, North Eastern Hill University, Shillong-793022., Meghalaya, India joshuamawrie@gmail.com

### Available online at: www.isca.in, www.isca.me

Received 26<sup>th</sup> October 2017, revised 23<sup>th</sup> November 2017, accepted 1<sup>th</sup> December 2017

### Abstract

In this study, six fruits and vegetable products (apple, grapes, tomato, lettuce, potato and cabbage) were collected from lewduh market in Shillong which is the state capital of Meghalaya, India, and were tested to reveal whether they were contaminated with organochlorine pesticides which are banned under Indian Law for posing a threat to the health of humans as well as the environment. For the purpose of this study three banned organochlorines: dieldrin, heptachlor and endosulfan were chosen. A standard operating procedure using Gas Chromatography – Mass Spectrometry was employed for their analysis.

Keywords: Banned, Organochlorine, Pesticide, Fruit, Vegetable, Chromatography, Mass spectrometry.

# Introduction

Pesticides are heavily used in the agricultural and health sector for control of pest and vector borne diseases. They play an important role in reducing crop loss due to pest infestation and help boost crop production which is important to sustain the food security of any nation. Although, pesticides have proven themselves very useful in the agricultural sector but they bring along with them a host of disadvantages. Due to their toxic nature, these chemicals have harmful effects on animals as well as humans. Commonly, pesticides are divided into four main groups: organochlorines, organophosphates, carbamates and pyrethroids<sup>1</sup>. Of these four groups, organochlorines are highly persistent in nature and can cause poisoning and even death. It is for this that they are banned in most countries; however, they are still produced and used in many developing countries as a cheap source of insecticide<sup>2</sup>. In India a number of organochlorines are banned and the Central Insecticide Board and Registration Committee, Ministry of Agriculture and Farmer Welfare, has enlisted pesticides which have been banned or restricted for use by the government of India<sup>3</sup>. Pesticide based studies have been carried out in many places in India but the states in the North Eastern Region still lack behind in this regard. Therefore, it is of utmost importance to initiate research work on pesticide residues especially in food products in this region. The main objective is to analyze a few fruit and vegetable samples collected from a market in Shillong for any possible contamination by three banned organochlorine pesticides in India and these are as follows:

**Dieldrin:** As per the toxicological profile provided by the U.S. Department of Health and Human Services, it is a white powder

and it is scientifically named as 1,2,3,4,10,10-hexachloro-6,7-epoxy 1, 4,  $4\alpha$ , 5, 6, 7, 8,  $8\alpha$ -octahydro-1, 4-endo, exo-5, 8-dimethanonaphthalene. It was used extensively as an insecticide. Dieldrin is also breakdown product of aldrin and can be found in areas where aldrin was used. It is highly persistent, toxic and classified by the Environmental Protection Agency as a probable human carcinogen<sup>4</sup>.

**Heptachlor:** is an organochlorine pesticide which is persistent in nature and can transform into 1- hydroxychlordene or heptachlor epoxide. Its scientifically represented as 1, 4, 5, 6, 7, 8, 8-heptachloro-3a, 4, 7, 7atetrahydro- 1H-4, 7-methanoindene. The Environmental Protection Agency and the International Agency for Research on Cancer have classed heptachlor as a possible human carcinogen (ATSDR 2007)<sup>5</sup>.

**Endosulfan:** Endosulfan (6,7,8,9,10,10-hexachloro-1, 5, 5a, 6, 9, 9a-hexahydro- 6, 9-methano-2,4,3-benzadioxathiepin-3-oxide) is a persistent organochlorine used as a pesticide in many parts of the world and has been linked with severe health effects<sup>6</sup>. In 2011, a report on health effects of Endosulfan by the Department of Health and Family Welfare, Government of Kerela, stated that by the 1990s health disorders due to endosulfan use came into light with children being the most affected. Congenital anomalies, mental retardation, physical deformities, cerebral palsy etc. were a few of the health impacts which were attributed to the use of Endosulfan<sup>7</sup>.

Dieldrin, Heptachlor were enlisted by the Central Insecticide Board and Registration Committee as banned pesticides whereas endosulfan fall under the restricted for use list. The Honourable Supreme Court of India passed an ad-interim order on May, 2011 banning production, sale and use of Endosulphan in the country<sup>8</sup>. This study is important in order to reveal if these banned pesticides are still being detected in food products which may pose a threat to the population of Shillong and also as an indicator on whether the ban on the concerned organochlorine pesticides is working or not. The food products which were subjected to this study included apple, grapes, tomato, lettuce, potato and cabbage.

Area of sampling: This study was carried out in Shillong which is the capital of Meghalaya, a state that is located in the North Eastern Region of India with a population of 143229 as per the 2011 census<sup>9</sup>. The samples were collected from a market called Iewduh or Bara Bazaar and it is the single largest market in Shillong City which supplies the other markets within the city with almost all kinds of food products. It is also a market where farmers from different regions of the state usually bring their produce for sale. Major portion of the food products that come in from different places of the country also end up here. This market is highly significant as it is a very convenient study area to carry out any food product based study relating to the city of Shillong.

# Methodology

For analysis of pesticide residues the use of Chromatography-Mass Spectrometry was employed. Gas Chromatography-Mass Spectrometry is a widely use technique for qualitative and quantitative analysis especially for volatile compounds. It has also been successfully put to use in the field of pesticide study in a number of works carried out worldwide. This technique as described by Sneddon et al. involves two major methods wherein separation of mixtures into different components is carried out by Gas Chromatography and the qualitative and quantitative determination of the separated compounds is done by Mass Spectrometry<sup>10</sup>. Sample preparation is carried out before introducing the sample to the GCMS and this can be achieved through a number of methods such as QuEChERs, Solid Phase Microextraction, Liquid-liquid exraction etc. After this the sample is introduced into the Gas Chromatogrphy through the injection port where it volitalises and is carried by a carrier gas which is inert in nature through columns packed with a fine solid material like silica particles representing the stationery phase. Here, separation of the sample into different components takes place. The sample exist the GC separated and enters the Mass Spectrometer where detection and quantification of the separated fragments of the sample takes place as described by Hussain and Magboo<sup>11</sup>. Bhuvaneshwari and Rajendran in 2012 employed a similar method using Gas Chromatography-Mass Spectrometry in their study of organochlorine pesticides in fish that were collected from from River Cauvery and Veeranam Lake<sup>12</sup>.

**Sampling**: For the purpose of this study one kilogram of each sample was collected from 5 random areas within the market totaling to 5 kilograms each. A composite sample of 500gm was

then taken and used for analytical purposes. The samples were immediately packed in dry ice and sent to Institute of Pesticide Formulation, New Delhi, for analysis. An internal standard operating procedure of sample preparation and residue analysis using GC-MS was carried out for the determination of the pesticide residues of the three organochlorines which were chosen for this study.

# **Results and discussion**

The Gas Chromatography Mass Spectrometry analysis of the fruit and vegetable samples for the pesticide residues in question displayed expected results. As the pesticides chosen for this study are banned, it was anticipated that the food products should not contain any of their residues. The test results for the analysis of dieldrin, heptachlor and endosulfan in the six different fruit and vegetable samples from the market in Shillong showed that in all the samples the amount of pesticide residues were Below Detection Limit which is < 0.1 mg/kg as depicted in the Table-1.

**Table-1:** Pesticide Residues (mg/kg) in Fruits and Vegetable Samples.

Sample	Pesticide Residues (mg/kg)		
	Dieldrin	Heptachlor	Endosulphan
Apple	< 0.1	< 0.1	< 0.1
Grapes	< 0.1	< 0.1	< 0.1
Tomato	< 0.1	< 0.1	< 0.1
Lettuce	< 0.1	< 0.1	< 0.1
Potato	< 0.1	< 0.1	< 0.1
Cabbage	< 0.1	< 0.1	< 0.1

From the outcome of this study it can be mentioned that the food samples that were studied do not contain any harmful amounts of the banned pesticides that were analysed.

**Discussion:** The study to shed light upon the level of contamination of the food products in question by dieldrin, heptachlor and endosulphan showed that residual levels of these banned pesticides were below detectable limits. The findings of this study indicates that at least six fruits and vegetable products (apple, grapes, tomato, lettuce, potato and cabbage) which are sold in the Iewduh Market of Shillong may be free from the three organo-pesticides that have been banned under the Indian law. The initiative of the Indian Government to protect its population from being exposed to harmful organochlorine pesticides seems to be working for the city of Shillong as revealed by the study. Although, this study showed the desired outcome but there is still a number of other banned

pesticides and food products that are yet to be studied in order to come up with a concrete risk assessment for the people of Shillong City when it comes to contamination of food products by harmful pesticides. Apart from the pesticides that have been banned there are also other harmful pesticides that are still legally being use in the agricultural sector in our country. As of December 2016, an article published in The Hindu, Business Lines, informed that there are 51 pesticides that have been banned by other countries but still in use in India<sup>13</sup>. Therefore, there is a collective need for different sections of our society, from the farmers to the consumers and the government to work together to ensure that we are not consuming toxic pesticides along with our food.

# Conclusion

In conclusion, this kind of study is essential in order to keep the people informed about food safety and take necessary precautions to ensure what they eat is toxin free. There is little to no information about pesticides in food in the state of Meghalaya, especially in Shillong City. Therefore, such studies are important to add to the information pool and also aid the research community in carrying out further investigation in this field of study.

# Acknowledgement

We would like to express our profound gratitude to the Institute of Pesticide Formulation Technology for sample analysis.

## References

- 1. Garcai P.F., Ascencio C.Y., Oyarzun G.C.J., Hernandez C.A. and Alavarado V.P. (2012). Pesticides: classification, uses and toxicity. Measures of exposure and genotoxic risks. *Journal of Research in Environmentl Science and Toxicology (ISSN: 2315-5698)*, 1(11), 279-293.
- 2. Dent D. (1991). Insect Pest Management: Insecticides. C.A.B International, 146-147. ISBN: 0-85198-667-6.
- **3.** http://cibrc.nic.in/list\_pest\_bann.html. Retrieved on 10.10.2017.

- **4.** Agency for Toxic Substances and Disease Registry (2002). Toxicological Profile for Aldrin and Dieldrin. *U.S. Department of Health and Human Services*, 1-4.
- 5. US Department of Health and Human Services (2005). Agency for Toxic Substances and Disease Registry, 2007. Case Studies in Envionmental medicin-Lead toxicity. ATSDR Publication ATSDR-HE-CS-2001-0001.
- **6.** Naiar S.A., Tom T.T. and Pradeep T. (2003). Detection and Extraction of Endosulfan by Metal Nanoparticles. *J. Environmenta Monit.*, 5(2), 363-365.
- 7. Department of Health and Family Welfare, Report on Health Effects of Endosulfan and Progress of Rehabilitation Activities in Kerala (2011). Endosulfan The Kerela Story. Department of Health and Family Welfare, Government of Kerala, 9.
- **8.** Press Information Bureau (2014). Ban on Endosulphan. Ministry of Health and Family Welfare, Government of India. Retrived on 19.10.2017 http://pib.nic.in/newsite/PrintRelease.aspx?relid=107462.
- **9.** Census (2011). Shillong City Census 2011 data. http://www.census2011.co.in/census/city/187-shillong.html.
- Sneddon J., Masuram S. and Richert C.J. (2007). Gas Chromatography-Mass Spectometry- Basic Principles, Instrumentation and Selected Applications for Detection of Organic Compounds. *Analytical Letters* (ISSN: 0003-2719), 40(6), 1003-1012.
- **11.** Hussain Z.S. and Maqbool K.M. (2014). GC-MS: Principle, Technique and its application in Food Science. *INT CURR SCI*, 13(E), 116-126.
- **12.** Bhuvaneshwari R. and Rajendran B.R. (2012). GCMS Determination of Organochlorine Pesticides (OCPS) in Fish from River Cauvery and Veeranam Lake. *E-Journal of Chemistry*, 9(4), 2346-2353.
- **13.** Press Trust of India. (2016). Use of pesticides elsewhere allowed in India. *Business Lines, The Hindu*, 1. Retrieve on 13.10.2017. http://www.thehindubusinessline.com/economy/agri-business/use-of-51-pesticides-banned-elsewhere-allowed-in-india-centre-tells-high-court/article 9416251.ece.