

Ramesh Kumar Sharma · Salvatore Parisi

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# Chapter 1

## Insecticides in Indian Food Products

**Abstract** The current usage of insecticides and pesticides in modern food industry and agriculture is surely evident in many industrialised countries such as India. Despite their use as crop protection chemicals, their toxic action has been remembered by environmentalists as well as food and agricultural scientists. Organic insecticides—mainly organochlorines, organophosphorus, carbamates and pyrethrins/pyrethroids—are extensively used for crop protection, but their limitation is correlated with safety consequences. Azadirachtin, the interesting natural insecticidal compound extracted from *neem* trees, may be a solution against environmental harms caused by synthetic insecticides and pesticides. Indian food business operators have to face consequences of the excessive use of insecticides in farms as rejections of export consignments. Countries possessing enough dense infrastructure, particularly European countries, are capable of keeping insecticide at minimum residue levels lower than India. Also for this reason, the Indian Country needs dense forestation.

**Keywords** Carbamate · Forestation · Insecticide · Organochlorine · Organophosphorous · Pyrethrin · Pyrethroid

### Abbreviations

BHC	Benzene hexachloride
DDT	Dichlorodiphenyltrichloroethane
FAO	Food and Agricultural Organization
FSSR	Food Safety and Standards Regulation
HCN	Hydrogen cyanide
LD <sub>50</sub>	Lethal dosage
MRL	Maximum residue level
UNEP	United Nations Environment Programme



## 1.1 Introduction

With the technical advancement in the twentieth century in the fields of food processing, agriculture, and public health, the control of pests and insects is now a part of human tasks instead of natural action. The food production and farming and hygiene techniques for the reduction of insect populations, originally limited to promotion of natural biocontrol (as lizards and the frogs eat up flies and mosquitoes), have got the new dimension of use of insect–pest controlling substances: insecticides and pesticides. Well known for the contribution to agricultural yield safety and chronic contagion control, man-made insecticides and pesticides are also known for polluting the environment, water and foods. Despite the fact that insecticides adversely affect the environment, their use as sprays is almost unavoidable in food industries (not in food, but in building) and agriculture farms for protection of foods and crops from microorganisms, pests and insects. The modern food industry and agriculture cannot exclude the use of different chemical substances with peculiar function including insecticidal function. This situation is surely evident in many industrialised countries such as India, where age-old insect controlling techniques like biocontrol promotion and use of natural insecticides—mainly *neem* (*Azadirachta indica*) leaves' juice—have been becoming obsolete since mid-twentieth century.

The use of synthetic pesticides in India commenced in 1948 when the country imported *p, p'*-dichlorodiphenyltrichloroethane (DDT) for the control of contagion malaria (Kumar and Kumar 2007). In the same year, benzene hexachloride (BHC) was imported to control insect locusts for safety of agricultural yield.

In 1952, India initiated the indigenous production of both insecticides; at present, approximately 145 pesticides are registered for use (Kumar and Kumar 2007). In India, forests and pastures, which had been publicly utilised and maintained too as a commonly shared property since centuries, were subjected to government acquisition followed by conversion for mining and housing purposes. This situation of deforestation led to decrease in soil conservation (or soil fertility) and compelled farmers, particularly belonging to non-irrigated lands, to use increasing amounts of fertilisers like urea and superphosphates in place of natural manures like animal dung and urine as well as synthetic insecticides in place of natural *neem* leaf juice.

Indian farmers have initially owned small farms. The big agriculture companies in India began to run with effect from commencement of nineteenth century when Dupont India and Rallis India Limited were established in 1802 and 1815 at Gurgaon (Haryana) and Bangalore (Karnataka), respectively. Both these companies deal in agro-based products including synthetic insecticides. On the other hand, Foabs Organic Estates—established in 1889 at Tiruvalla (Kerala) dealing in tea, coffee and spices—has till-to-date maintained traditional farming employing the ways of natural manure insecticide applications. Recently, from 2000 onwards, Insecticides India Ltd. emerged as one of the fastest growing agrochemicals manufacturing companies with insecticides based on chlorpyrifos, monocrotophos, imidacloprid and other active principles. Nowadays (from 2000 onward) it may be estimated that

more than half of food commodities in India are contaminated with insecticide–pesticide residues; out of these one-fifth cross allowed maximum residue levels (MRL) (Kumar and Kumar 2007), when the dire need of traditional (or organic) farming is underlined by both environmentalists and food scientists.

In this context, it is worthwhile to get an overview of insecticides in Indian food products in terms of chemistry, and limits and consequences for food business operators. It is worth mentioning that Indian agrichemicals market, which was stood at 641 million \$ in 2000, is currently poised at over 2.5 billion \$ (Kaki 2015).

## 1.2 Chemistry of Insecticides

### 1.2.1 Definitions

A substance used for reducing or controlling insect/pest populations is called insecticide or pesticide. The elimination of insect populations is mainly required for disease prevention, crop protection, wood/paper/cloth preservation and people/animals injury reduction. Insecticides and pesticides are broadly defined as follows (Carter 1976): *‘Insecticides and pesticides are the substances used to kill insects and pests or affect their life cycle to reduce and control their populations’*.

Insecticides and pesticides are well known for contributing to agricultural yield safety and chronic contagion control and, at the same time, for environmental pollution. These substances are normally classified by (a) the selective toxic effect, or (b) the way to target a particular insect, or (c) the chemical structure, or (d) the toxicity level or the health hazards for humans.

At present, environmentalists worldwide suggest alternative insect-control techniques (Agriculture Census 2016):

- (a) Physical control (infested parts of vegetables have to be destroyed/removed)
- (b) Cultural control (after harvest operations, remaining insects have to be eliminated with profound ploughing; in addition, sowing times can be defined preventively with the aim of reducing insect incidence)
- (c) Biological control (natural enemies of crop insects can be favoured).

On the other hand, Indian farmers still prefer chemical control techniques to protect crops from the attack of insects and pests due to immediate action. Insecticides are popularly known as crop protection chemicals in India and applied in farms with proper equipment. From the agricultural viewpoint, an appropriate definition of insecticides and pesticides is as follows (Carter 1976): *‘Insecticides and pesticides are crop protection chemicals applied to control different insects-pests as dusts, sprays or granules on the crops and/or incorporated into the soil for the control of soil-inhabiting insects’*.

The toxic action of pesticides, mostly organic molecules that are released intentionally in the environment against pests and different disease agents, has been recently described (WHO-UNEP 1990).

## 1.2.2 Chemical Structures of Insecticides

Insecticides belong to both categories of chemicals: inorganic and organic substances. Nowadays, inorganic insecticides—arsenicals, fluorosilicates, hydrogen cyanide, hydrogen phosphide, bromides, etc., are not much used in India. However, hydrogen cyanide (HCN) occurs in crops as natural toxin. At the same time, agricultural scientists feel that arsenicals could be advantageously used against leaf-feeding insects, particularly parasites or predators. The Food Safety and Standards Regulation (FSSR) 2011 permits MRL of 37.5 and 25 ppm in food grains for HCN and inorganic bromide, respectively. At the same time, FSSR 2011 does not permit hydrogen phosphide even in residual traces (FSSR 2011). Among the organic insecticides, there are a few substances of bacterial and plant origin: nicotine sulphate, pyrethrins, rotenone, etc., as well as a large number of synthetic insecticides. Organic insecticides are categorised as hydrocarbons, organo-nonmetallics and organometallics.

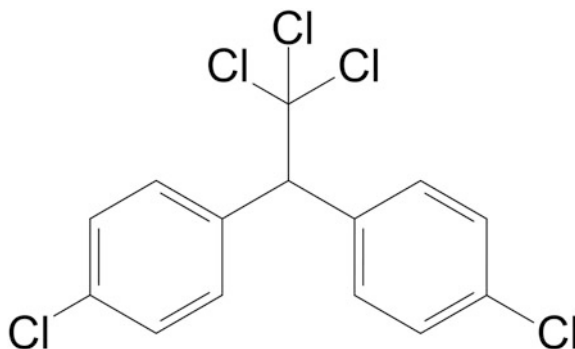
In general, organo-nonmetallic insecticides include organophosphorus and organosulphur compounds, while organometallic insecticides include mercury, tin, copper and zinc compounds. The FSSR 2011 permits 148 insecticide residues with prescribed MRL or tolerance limits for particular food articles. However, it also mentions hydrogen phosphide with nil tolerance limit (FSSR 2011).

Organic insecticides, extensively used for crop protection, cover the wide range of chemical structures, including hydrocarbons, carboxylic acid derivatives, alcohols, aldehydes, ketones, amines, nitro compounds, quinones, thiocyanates, mercaptans, heterocyclic compounds, etc. However, a four-class subdivision may be proposed: organochlorines, organophosphorus, carbamates and pyrethrins/pyrethroids (Buchel 1983), despite a wide range of chemical structures.

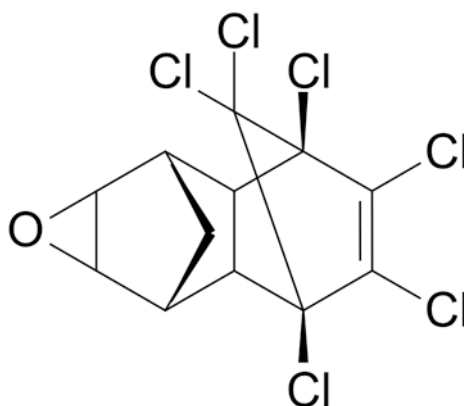
### 1.2.2.1 Organochlorides

The first important synthetic organic insecticide, DDT, was synthesised by the German scientist Ziedler in 1873 (Kroschwitz 1998). The Swiss scientist Paul Muller first noticed its insecticidal property in 1939. It was obviously the first discovered organochlorine insecticide and the first synthetic organic insecticide to be used in crop protection and contagion controls: this compound was hailed as a miracle for its broad-spectrum activity, low cost, and easy use (Kenneth 1992).

Organochlorine insecticides with four or more chlorine atoms are known for disrupting nervous system of insects and thus paralysing them. On the other side, they are resistant to chemical and microbial degradation; therefore, these compounds remain in the environment for a long time and adversely affect biodiversity and public health (Kumar and Kumar 2007). Two of the most important organochlorine insecticides, with prevalent name and molecular formula/structures are shown in Figs. 1.1 (DDT) and 1.2 (dieldrin).



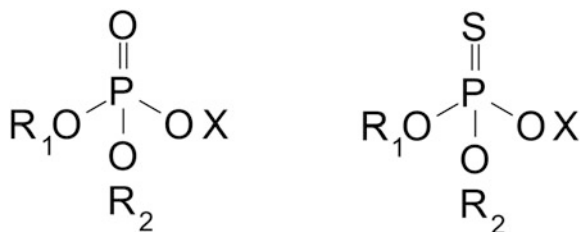
**Fig. 1.1** The molecular structure of *p,p'*-dichlorodiphenyltrichloroethane (DDT). BKchem version 0.13.0, 2009 (<http://bkchem.zirael.org/index.html>) has been used for drawing this structure



**Fig. 1.2** The molecular structure of the insecticide dieldrin, also named 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. BK chem version 0.13.0, 2009 (<http://bkchem.zirael.org/index.html>) has been used for drawing this structure

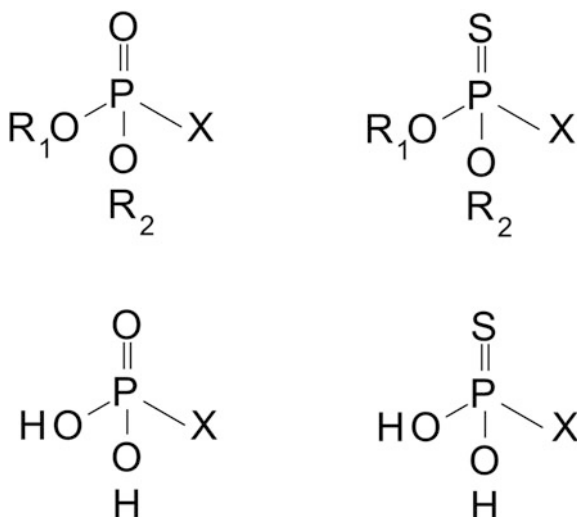
### 1.2.2.2 Organophosphorus Insecticides

Since 1968, when Martin stated that unlike organochlorines, organophosphorus insecticides are easily decomposed in the environment by various chemical and biological reactions (Martin 1968), the latter have been much more used for crop protection. The structural formula for organophosphorus insecticides, as initially proposed by Schrader, is shown in Fig. 1.3, where  $R_1$  and  $R_2$  substituents are usually alkyl groups (particularly methyl or ethyl groups), alkoxy, alkylthio or amino groups, whereas X might be an aliphatic, homo- or heterocyclic substituent. The basic formula may be partially modified as shown in Fig. 1.4 (Festa and Schmidt 1982), where the X group is one of the following substituents: halides,



**Fig. 1.3** The structural formula for organophosphorus insecticides, as initially proposed by Schrader.  $R_1$  and  $R_2$  substituents are usually alkyl, alkoxy, alkylthio or amino groups, whereas  $X$  might be an aliphatic, homo- or heterocyclic substituent. BKchem version 0.13.0, 2009 (<http://bkchem.zirael.org/index.html>) has been used for drawing this structure

**Fig. 1.4** The structural formula for organophosphorus insecticides, as proposed by Schrader and modified by Festa and Schmidt.  $R_1$  and  $R_2$  substituents are usually alkyl, alkoxy, alkylthio or amino groups.  $R_1$  and  $R_2$  can be also replaced by hydrogen atoms. This figure displays four possibilities only. BKchem version 0.13.0, 2009 (<http://bkchem.zirael.org/index.html>) has been used for drawing this structure

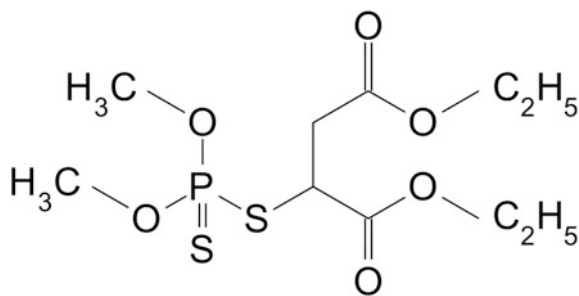


cyano, phenolic, aryloxy, etc. Some of the extensively used organophosphorus insecticides with prevalent name and molecular formula/structures are also displayed in Figs. 1.5 (malathion) and 1.6 (glyphosate).

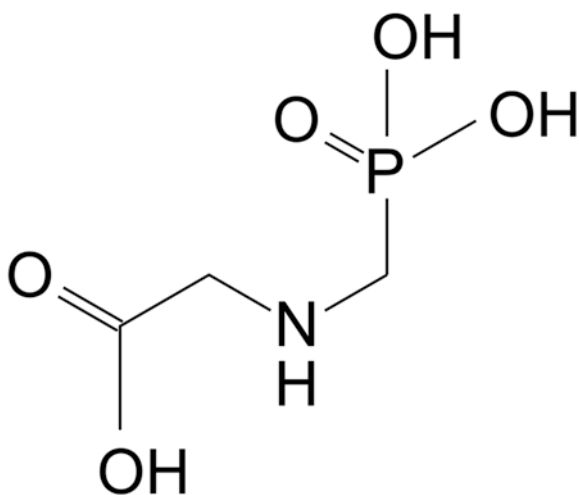
### 1.2.2.3 Carbamates

Carbamates are used because of their high insect toxicity as a result of cholinesterase inhibition. At the same time, these insecticides inhibit cholinesterase in humans and other mammals. Drum stated in 1980 that the cholinesterase inhibition of carbamates differ from that of organophosphorus insecticides because of their species specificity and reversibility (Drum 1980). The word 'carbamate' stands for an ester formed by an alcohol, general formula: ROH, and carbamic acid, usual formulas:

**Fig. 1.5** The molecular structure of malathion, also named *S*-1,2-bis(ethoxycarbonyl) ethyl-*O,O*-dimethylphosphorodithioate. BKchem version 0.13.0, 2009 (<http://bkchem.zirael.org/index.html>) has been used for drawing this structure



**Fig. 1.6** The molecular structure of glyphosate. BKchem version 0.13.0, 2009 (<http://bkchem.zirael.org/index.html>) has been used for drawing this structure



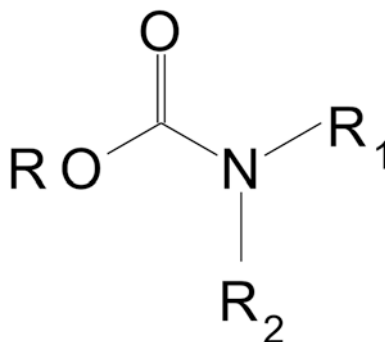
$\text{NH}_2\text{COOH}$  or  $\text{NR}_1\text{R}_2\text{COOH}$  ( $\text{R}_1$  and  $\text{R}_2$  are different substituents). The general structure for carbamates insecticides with  $\text{R}_1$  and  $\text{R}_2$  groups is shown in Fig. 1.7.

#### 1.2.2.4 Pyrethrins and Pyrethroids

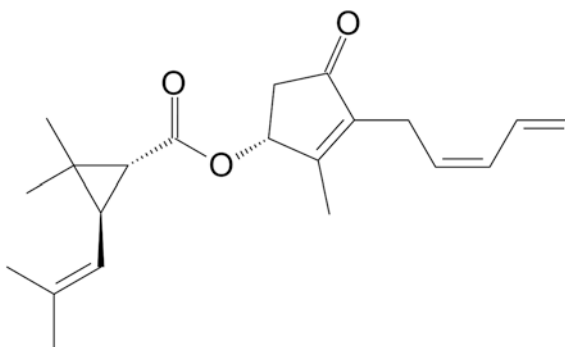
Pyrethrins I and II are two of the six naturally occurring, main insecticidally active ingredients of pyrethrum flowers (botanical names: *Chrysanthemum cinerariaefolium*, *C. coccineum* and *C. moshallii*; localisation: Africa, India and southern Europe). Pyrethrum flowers are also known as Persian insect flowers, because it is said that Persians (Iranians) initiated their use in killing insects. Pyrethrins I and II, cinerins I and II, and jasmolins I and II are the primary ingredients of pyrethrum flowers having insecticidal property. The molecular structure of one of these molecules, pyrethrin I, is shown in Fig. 1.8.

The FSSR 2011 of India considers collectively the term 'pyrethrins' (with relation to tolerance limits in food articles) as the sum of pyrethrins I and II and other structurally related insecticide ingredients of pyrethrum. In other terms, the

**Fig. 1.7** The general structure for carbamates.  $R_1$  and  $R_2$  are different groups. BKchem version 0.13.0, 2009 (<http://bkchem.zirael.org/index.html>) has been used for drawing this structure



**Fig. 1.8** The molecular structure of pyrethrin I, also known as the pyrethrolone ester of chrysanthemum monocarboxylic acid. BKchem version 0.13.0, 2009 (<http://bkchem.zirael.org/index.html>) has been used for drawing this structure



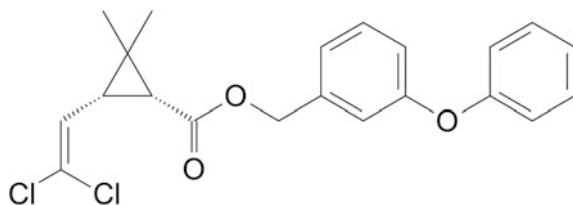
term ‘pyrethrins’ is tacitly referred to all the six insecticidally active pyrethrum ingredients. Pyrethrins have replaced organophosphorus and organochlorines insecticides to a great extent due to their considerable insect killing property as well as their low toxicity for humans and other mammals. In addition, it has to be observed that pyrethrins can aggravate pre-existing asthma in humans if these substances are not handled properly by the user, in particular when related concentration is 0.2 % or more, even in shampoos (Wagner 2000).

Pyrethroids are the synthetic analogues of pyrethrums and are obtained mostly by:

- (a) The introduction of a phenolic group, and
- (b) The substitution of some hydrogen atoms with halogens, and/or cyanide groups, and
- (c) The modification (increase or decrease) of the chrysanthemic or pyrethroic structural chain.

The molecular structure of one of these prominent pyrethroid insecticides, permethrin, is displayed in Fig. 1.9.

Pyrethroids, similar to naturally occurring pyrethrum insecticide compounds, are well known for effective insect killing properties. However, differently from pyrethrins, they are susceptible to photochemical degradation (Linde 1994). Therefore,



**Fig. 1.9** The molecular structure of an important pyrethroid insecticide, permethrin. BKchem version 0.13.0, 2009 (<http://bkchem.zirael.org/index.html>) has been used for drawing this structure

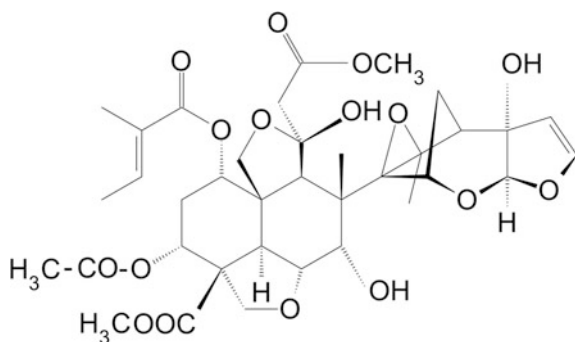
the real advantage of pyrethroids is that those are more effective insecticides than pyrethrins when speaking of farm application or crop protection. On the other side, their limitation is correlated with safety consequences (they are not as safe for the environment as pyrethrins are).

### 1.2.2.5 Miscellaneous Groups

Although organochlorines, organophosphorus, carbamates, pyrethrins and pyrethroids are the most extensively used organic insecticides for crop protection and contagion control applications, a wide range of peculiar substances such as phenoxyacetic acid, bipyridyls, tetranortriterpenoids, etc., are still considered in this ambit.

In particular, azadirachtin (Fig. 1.10) is an interesting insecticidal compound: it is extracted from *neem* (an Indian tree, Sect. 1.1) seeds. It has an oxidised tetranortriterpenoid molecular structure (Veitch et al. 2007) which includes enol ether, acetal, hemiacetal and tetra-substituted oxirane groups as well as a variety of carboxylic acid. With relation to natural insecticidal characteristics of *neem* tree seeds, it is the most distinguished example of oxygen functionalism. It is worth mentioning that Indian farmers have been applying *neem* leaf juice and *neem* seed extracts since centuries for crop protection. Azadirachtin is considered as very effective insecticide as well as safe for the environment.

**Fig. 1.10** The molecular structure of azadirachtin. BKchem version 0.13.0, 2009 (<http://bkchem.zirael.org/index.html>) has been used for drawing this structure





### 1.3 Toxicity of Selected Insecticides

The toxicity of insecticides is not only considered in context of its efficacy to kill the targeted insects which affect farm crop and public health, but also in relation to safety concerns for the biosphere, particularly humans and other mammals, or the entire environment.

For determining toxicity value of an insecticide, test animals such as rats, mice, or rabbits are employed. The calculated lethal dosage ( $LD_{50}$ ) is defined as the amount of insecticide at which 50 % of test animals expire.  $LD_{50}$  is expressed as insecticide dosage in milligrams per kilogram of body weight (of test animals).

A perusal of  $LD_{50}$  data of different insecticides leads to conclude that natural insecticides pyrethrins and azadirachtin are safe for mammals including humans, as compared to synthetic insecticides. At the same time, the comparison between  $LD_{50}$  values of two natural insecticides shows that azadirachtin is safer than pyrethrins and practically non-toxic to mammals. Azadirachtin is known for feeding inhibition activity towards over 200 insect species including *Schistocerca gregaria* (locust) and *S. littoralis* (Butterworth and Morgan 1968). A low  $LD_{50}$  value—15 mg/kg—has been recently reported (Boeke et al. 2004). It may be assumed that azadirachtin is safe for the entire biosphere, while pyrethrins are highly toxic for fish species such as trouts with  $LD_{50}$  values of 14 mg/kg (Coats et al. 1989).

### 1.4 Limits of Insecticides in Indian Food

Names of 149 insecticides are enlisted for protection of particular foods within the prescribed tolerance limits in the FSSR, 2011, Sect. 2.3: Residues. In detail, the FSSR states clearly, with relation to restriction on the use of insecticides (Sect. 2.3.1), that:

- (a) Insecticides cannot be used directly on articles of food; however, this declaration does not concern ‘*fumigants which are registered and recommended for use as such on articles of food by the Registration Committee, constituted under section 5 of the Insecticides Act, 1968 (46 of 1968)*’
- (b) Anyway, mentioned insecticides (annexed Table to Sect. 2.3) cannot exceed prescribed tolerance limits in relation to cited foods.

The interested reader is invited to consult the FSSR 2011, Sect. 2.3 (Residues). For example purposes, it can be noted here that:

1. Insecticides aldrin and dieldrin (related values are expressed as dieldrin as single molecules or in combination) have different tolerance limits as mg/kg or ppm depending on foods. As an example, these values can be 0.01 mg/kg in food grains or 0.2 mg/kg in eggs.
2. Carbaryl is not allowed in milled food grains (tolerance limit: nil), while it could be permitted in okra and leafy vegetables up to 10.0 mg/kg.

## 1.5 Consequences for Food Business Operators

The Food Safety and Standards Act 2006 of India, Chap. 3—General Principles of Food Safety—part 3, states clearly that ‘*the provisions of the Act shall not apply to any farmer or fisherman or farming operations or crops or livestock or agriculture, and supplies by a farmer at farm level or fisherman in his operations*’. This statement of the Act should be discussed thoroughly when speaking of food safety. The procurement of safe raw material is quite essential for the production of a safe processed food article. For example, synthetic insecticides are used in farms where law of insecticide limitation is not applied. Consequently, the question obviously concerns the penalisation of a food business operator (FBO) with relation to the detection of insecticides above tolerance limits in his or her processed food article(s).

The Indian FBO have to face consequences for procurement of raw material indigenously grown in farms if found to exceed the prescribed insecticide tolerance limit or MRL mentioned in the FSSR 2011. This does not mean that Indian agricultural products contain high amount of insecticides. India is one of the countries which adhere to the Codex Alimentarius—International Food Standards (<http://www.fao.org/fao-who-codexalimentarius/en/>) with different aims, including the definition of MRL for insecticides. FSSR 2011 does not allow the presence of many insecticides like chlorpyrifos, cypermethrin, diazinon, dichlorvos, ethion, malathion, parathion phorate and phosalone in spices (FSSR 2011); India maintains normally insecticide amounts in foods much below Codex MRL. On the other side, European and other Western countries like United States of America and Canada are capable of keeping insecticide MRL in agricultural produce—due to cost-effective organic farming followed by dense forestations—even lower than India. This situation offers a tough challenge to Indian food exports, with particular reference to traditional spices. India is well known for the high quality of exported spices. Under the supervision of Spices Board in India, selective organic farming without use of synthetic insecticides is carried out for growing spices. However, Indian spices consignments still have to face some rejections at EU borders. As a consequence, India needs dense mountain forestation and cost-effective organic farming; fortunately, naturally grown *neem* tree insecticide products are abundant in the country.

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## Chapter 2

# Aflatoxins in Indian Food Products

**Abstract** Hepatitis cases have been reported in India in the past due to consumption of food contaminated by some aflatoxin, a mycotoxin produced by *Aspergillus flavus* and generally developed in food articles grown and/or stored in hot and moist environment. The main target organ of hepatic disease, caused by regular consumption of aflatoxin-contaminated foods, is liver which may suffer from jaundice and cancer in later stages. Milk is an ideal food for such patients provided it is free from aflatoxins. The Indian Food Safety and Standards Regulation, 2011 enlists aflatoxins among crop contaminants and naturally occurring toxins. In the European Union, food regulation ascertains much lower values for maximum aflatoxin contents in food articles than that the Indian food law does. Indian food business operators seldom have to face consequences due to high aflatoxin contents, particularly in samples of exported goods, despite the fact that detoxification (removal of aflatoxins from foods) to some extent is possible.

**Keywords** Aflatoxin • *Aspergillus* • Crop contaminant • Detoxification • Food rejection • Moist storage • Mycotoxin

### Abbreviations

AAA	Aromatic amino acid
BCAA	Branched-chain amino acid
DNA	Deoxyribonucleic acid
EU	European Union
FBO	Food business operator
FSSAI	Food Safety and Standards Authority of India
ICRISAT	International Crop Research Institute for the Semi-Arid Tropics
IUPAC	International Union of Pure and Applied Chemistry
mRNA	Messenger ribonucleic acid
ppb	Part per billion
ppm	Part per million
U.S. FDA	United States Food and Drug Administration