

# **Safety chemical residues of protected crops against infestation on health care delivery and environment, a review**

## **Abstract**

The use of pesticides for effective pests control has generated a lot of concerns relating to public health and environmental pollution. Nigerian cocoa production is still dependent on pesticides to attain acceptable levels of crop production. Kola nuts need no further processing before consumption hence little or no chemical residue therein will be acceptable, perhaps sparingly chemical should be used for storage pests. The screening of new insecticides, fungicides and herbicides, new spraying pumps are usually evaluated by the Cocoa Research Institute of Nigeria (CRIN), for their efficiency before they are recommended for use in the application of cocoa pesticides and spraying equipment in Nigeria. Accordingly, Article 12 of Regulation (EC) No 396/2005, EFSA has reviewed the maximum residue levels (MRL s) currently established at European level for the pesticide active substance like chlorpyrifos. Therefore, to assess the occurrence of chlorpyrifos residues in plants, processed commodities and rotational crops EFSA considered the conclusions derived in the framework of Directive 91/414/EEC, the MRL s established by the Codex Alimentarius Commission. However, the indices to national Maximum Residue Limit (MRL) regulations on pesticides affecting agricultural and food trade were employed using a science-based criteria embodied in Codex Alimentarius international standards. The consumer risk assessment is considered and measures for reduction of the consumer exposure were also considered.

**Keywords:** Chemical; Residues; EFSA; MRL; Consumer; Risk; Assessment

## **Introduction**

Pesticide use in Nigeria has been on the increase ever since its introduction in early fifties for cocoa production. Most of the cash crops are exposed to attack by highly complex insect populations. Several workers identified some of the species as major insect pests which caused economic damage (Daramola, 1978; NRI, 1996; Ofuya and Bamigbola, 1991). Few of the common insect species are shown in Table 1. Most of the cash crops are vulnerable to attacks of insect pests at different stages of growth e.g. cocoa, kola, cowpea etc. Consequently, an attempt was made to control insect infestation both in the fields and stores, hence farmers employed different types of insecticides without recourse to approved insecticides (Asogwa, 2014a). Insecticides, herbicides and fungicides constitute the major pesticides used in Nigeria. The bulk of the pesticide is used in respect of agricultural production for the control of noxious weeds, insect pests and diseases of crops; and as the agricultural production system moves more and

more from subsistence market-oriented large scale farming, a concomitant increase in pesticide usage seems inevitable (Asogwa and Dongo, 2009).

A pesticide uses of which have been prohibited by final regulatory action, in order to safeguard human health or the environment against the hazard (Conway and Pretty, 1991). Pesticide residue limits in feed, food and food products are set by the Environmental Protection Agency (EPA), as required by the Federal Food, Drug, and Cosmetic Act amended to include the Food Quality Protection Act. These limits are known as tolerances, and are set to protect the nation's food supply and its consumers from harmful levels of pesticide residues. Food products must be traceable throughout the entire supply chain to guarantee food safety, to allow appropriate action in cases of unsafe food and to limit risks of contamination. An important aspect to control food safety hazards is defining critical control points (HACCP) by implementing food management principles. Subjecting food products to official controls is another important aspect. Products that are not considered safe will be denied access to the EU.

In the event of repeated non-compliance of specific products originating from particular countries stricter conditions, such as a health certificate and analytical test report, are required for import. Products from countries that have shown repeated non-compliance are put on a list included in the Annex of Regulation (EC) 669/2009. Due to problems with pesticide residues in tea from China, it is subject to an increased level of official controls. If nutrition or health claims are made, these have to be approved in advance by the European Food Safety Agency (EFSA). In December 2014 Regulation 1169/2011 which involved pest control strategy used complementary strategies including growing practices and chemical management. However, specific product legislation was enacted for coffee extracts and cocoa and chocolate products (including cocoa butter), in addition to other legislation which sets common sales names. These also took into account composition, description and labelling on the packages and clearly communicate to buyer about these issues if the products listed are supplied in the respective EU Directives.

### **Food safety and EU legislation on contaminants**

Contaminants are substances that may be present as a result of the various stages of growing, processing, packaging, transport or storage. Threshold limits for several contaminants force to avoid negative impact on the quality of food and risks to human health. The different forms of contamination included pesticides which are the most common reason for border authorities to reject CTC. Green tea, is often refused for this reason. Therefore products containing more pesticides than allowed will be withdrawn from the EU market.

Also, Mycotoxic such as Moulds and fungi are another important reason for many border rejections (WHO, 2015). For roasted coffee beans and ground roasted coffee the maximum level of Ochratoxin A (OTA) is set at 5 µg/kg. There are no specific limits for green coffee beans as

they are not consumed as such. While in the case of cocoa, farmers in West-Africa should be aware especially of mycotoxins (Settimi *et al.*, 2003).

However, poly-aromatic hydrocarbons (PAH) can result from cocoa beans coming into direct contact with smoke, for example during artificial drying using badly designed or poorly maintained driers (Conway and Pretty, 1991). The limit for benzo(a)pyrene is 5.0 µg/kg of fat and 35 µg/kg for the total sum of PAHs. Furthermore, Salmonella is a very serious form of contamination and occurs occasionally as a result of incorrect harvesting and drying techniques (WHO, 2015). Tea, especially herbal and rooibos tea, is more prone to contamination than Cocoa and coffee beans which are considered low-risk commodities. In the current EU legislation no microbiological criteria for CTC have been set. Food safety authorities however can withdraw imported food products from the market or prevent them from entering the EU when Salmonella is found present. Irradiation is a way to combat microbiological contamination but this is not allowed by EU legislation for CTC. Finally, contamination by foreign matter like plastic and insects are a threat when food safety procedures are not carefully followed. Extraction solvents as contaminant can be used for decaffeination of coffee and tea and the production of cocoa butter. There are maximum residue limits restrictions for the extraction solvents such as methyl acetate (20 mg/kg in the coffee or tea), dichloromethane (2 mg/kg in the roasted coffee and 5 mg/kg in the tea) and Hexane (1 mg/kg in cocoa butter).

However, good agricultural practices address environmental, economic, and social sustainability for on-farm and post-production processes and result in safe and quality agricultural products.

Most chemicals used in Nigeria are imported through either the statutory channels or illegal means. According to FAO (2005), pesticide importation rises steadily from about 13 million dollars in 2001 to 28 million dollars in 2003 with insecticides accounting for about 32% of the imports. Thus the lists of these chemicals are in exhaustive because they come with different trade names. Among these synthetic insecticides only a few percentage are screened/passed through the regulatory agency (CRIN). Therefore this makes the bio-degradability and other inherent qualities of insecticides to be in doubt/suspect. The synthetic insecticides screened against kola weevils/mirids include Actara 25 WG, Esiom 150 SL, Proteus 170 O-TEC, Avestrin, Termicid, Capsida, Phostoxin, Zap and Confidor (Table 2). Among the chemical insecticides screened are Actara 25 WG, Esiom 150 SL and Proteus 170 O-TEC passed through all stages and considered effective, and thus recommended for use on Kola/cocoa tree or their products (Asogwa, 2014b).

However, Phostoxin is equally recommended as a fumigant in controlling storage pests. Other chemicals stated above aside recommended ones are still under investigation. Pesticide watchlist is composed of active ingredients that are not banned that have a potentially severe and/or cumulative risk for human health and/or the environment. According to Lale, (1995) who reported that beside Nigeria, other developing countries are fond of using adulterated and banned

insecticides due to high costs of approved insecticides and inadequate knowledge by farmers about proper use of pesticide products.

However, the following lists of insecticides are considered banned and farmers should be refrained from using it on kola/cocoa production. According to the report by several scientists in CRIN (Asogwa *et al.*, (2012); Asogwa (2014a); that the lists of insecticides included Acephate, Amitraz, Aldrin, Azinphos-methyl, Cabaryl, Cabofuran, Carbosulfan, Cartap, Terbufos, Cyhexatin, DDT, Dichlorvos, Dieldrin, Endosulfan, Lindane, Methyl-parathion, Methonmyl, Monocrotophos, Profenfos, Promecarb, Propoxur and others (Table 3). Thus, EU banned some Kola/Cocoa pesticides and approved some because of health and environmental concerns on pesticide residue on Kola/Cocoa products (Conway and Pretty, 1991; Mosudi *et al.*, 2008). Consequently, farmers were encouraged to use only recommended pesticides and adhere to use of optimum concentration of dosage on treated products.

Furthermore, the above listed pesticides have been recorded and used on cocoa (e.g. by the ECA/CAOBISCO project), but have been rejected by major importing countries (usually for toxicological/eco-toxicological reasons) and have no residue tolerance in major markets. Therefore, in view of the development farmers are strongly advised to stop using any of the products containing any of the active ingredients (Table 3). Paradoxically, farmers are still fond of using DDT on their cocoa plantation because DDT is cheap, they could procure the banned chemicals easily because it is readily available in the market, no adequate awareness on the status of the banned insecticides, and they do not have information on detrimental effect of the banned insecticides (Uwagboe *et al.*, 2010; Azeez, 2015a).

Consequently, the adulterate chemical/banned insecticides are sold in the market by accessible retailers with unapproved graduated measurements (Azeez, 2015a). However, farmers/traders are ignorant of the side/detrimental effects of the chemical used on their health and the environment (Azeez, 2015b). Moreover, Azeez, (2015b) reported that there is dearth of information on the efficient and economical methods of handling pests problems facing kola traders in Nigeria. Farmers are ignorant of the side/ detrimental effects of the chemicals used on their health and the environment. Since kola nut is consumed in its raw form without further processing, chemical control of weevils and other insect pests of kola must therefore take into consideration the level of residue of the pesticide in the treated nuts and the possible long-term effect of residue on consumers who are mainly Nigerians (Banerjee *et al.*, 1999; Settimi *et al.*, 2003). Adedire and Ajayi, (1996) reported that developing countries like Nigeria experienced additional problems which included use of adulterated and banned insecticides, non-availability of suitable application equipment, supply uncertainties, high costs and inadequate knowledge by farmers about proper use of pesticide products.

## **EU ban on exports of high farm product residues and knock on Nigeria economy**

National Bureau of Statistics (2015) showed that crude oil and gas accounted for 89.2 percent of Nigeria total export of #3.23 trillion with other exports constituting only 19.8 per cent in the first quarter. The nation imported goods and services worth #1.64 trillion within the same period. There is need to reverse this dependency on imports and harness our natural resources to become self-reliant in food production including cash crops for exports. Nigeria's economy is going through a turbulent period from reduction in oil income and this was further worsen due to recent ban placed on exported farm products from Nigeria. The European Union has just suspended some agricultural food exports from Nigeria; hence the food items banned hitherto from Europe are beans, sesame seeds, melon seeds, dried fish and meat, peanut chips and palm oil (Anonymous, 2015). This is a setback for a nation that desperately needs to expand its export basket to boost domestic agricultural activities and create jobs. This serves as stumbling block to the drive for economic diversity and sustainability.

According to the European Food Safety Authority (2015), the rejected beans were found to contain between 0.03 mg per kilogramme to 4.6 mg/kg of dichlorvos pesticide, when the acceptable maximum residue limit is 0.01 mg/ kg. This is the global standard set to adhere to at the international level (Anonymous, 2015). Overturning the ban requires a firm approach to enforcing standards at all times, though the EU has been warning Nigeria that the items constitute danger to human health because they contained a high level of unauthorized active ingredient. The pesticide is applied for the treatment of the farm products against pest infestation to extend their shelf life for export (Mosudi *et al.*, 2008). After several warnings and notifications by the EU and United Kingdom issued to Nigerian beans exporters. It is baffling that the Nigerian authorities did not take any significant steps to reverse the situation (Anonymous, 2015). Likewise, the United Kingdom also issued 13 border rejection alerts to Nigerian beans exporters between January and June, 2015. However, the laxity and lethargy of scheduled officers in the system might continue to hamper the economy from appropriating the benefits derivable from a revived export programmes.

Hitherto, 24 commodities of Nigerian origin exported to the UK were rejected, while the figure climbed to 42 food products in 2014. Some of the items were said to have been contaminated by aflatoxins, making them unfit for consumption (Anonymous, 2015; Conway and Pretty, 1991). It confounds many that this problem has been with us for some time and nothing strategic has been done to deal with the situation. The relevant government agency, like National Agency for Food and Drug Administration and Control (NAFDAC) and Ministry of Agriculture should be more proactive and enforced sanctions on exporters that caused the problem by not complying with regulatory requirements for semi-processed and processed commodities thus any further excuse is untenable. NAFDAC has not conducted its regulatory oversight properly and needs to put stringent measures in place to monitor our products and guarantee them as safe for export before the next EU review in future.

Nigerian consumers might be susceptible to poisonous food imported from Overseas. Take for example, the imported semi-processed poultry products and meats; several studies conducted by researchers and public agencies in markets in Lagos, Abuja and Port Harcourt are revealing. A study by Okiomah (2015), a nutritional enzymologist reported that poultry products imported into the country contain toxic heavy metals that can worsen the occurrence of food borne diseases based on the combination of feeds the animals eat. According to Ayoola (2015) the President of the Poultry Association of Nigeria, based on personal communication who reported that smuggled poultry products contained high residues of toxic chemicals and solvents used in the preservation of the products against infection by bacteria. This was done to extend the shelf life of the products and in anticipation for super profit at the expense of the consumers.

World Health Organisation (2015) reported that Food contaminants, such as harmful parasites, bacteria, viruses, prions, chemical or radioactive substances, caused more than 200 diseases, ranging from infectious diseases to cancers. The global health body added that unsafe food is linked to the death of about 2 million people annually. This however gingered Ministry of Agriculture to order the destruction of a large consignment contaminated imported frozen fish stored in a warehouse in Lagos (Anonymous, 2015). In the same vein, the Nigerian Customs Service had recently started enforcing the ban on imported poultry products, which are massively smuggled into the country. But government at the three tiers should also make policies to boost poultry and fish farming in the country to meet local demand and for export. However, the Ministry of Health, NAFDAC, the Standard Organisation of Nigeria and the newly inaugurated National Food Safety Management Committee should see the EU ban as a wake up to sanitise food imported into Nigeria, and those being consumed at home. The EU action suggests that Nigeria unfavourable balance of trade position with the international partners will worsen due to sanction against export of agricultural goods (Anonymous, 2015).

As a way forward, we could follow the standard practice in other climes like India, the UK, China and the United States, which operate effective food safety and regulatory agencies that monitor products stringently. US authorities are still battling China, South Korea, Mexico and South Africa to review a ban placed on American poultry and egg imports over the avian flu scare that broke out in December 2014. Recently, the Food Safety and Standards Authority of India ordered Nestle, the Swiss multinational, to withdraw its instant noodles from the market over safety concerns.

**Regulations on plant residues and commission implementing regulations of European Union (EU) countries**

Regulation (EC) No 396/2005<sup>1</sup> establishes the rules governing the setting and the review of pesticide maximum residue levels (MRLs) at European level. Article 12(2) of that Regulation stipulates that the European Food Safety Authority (EFSA) shall provide by 1 September 2009 a reasoned opinion on the review of the existing maximum residue levels (MRLs) for all active substances included in Annex I to Directive 91/414/EEC<sup>2</sup> before 2 September 2008. As chlorpyrifos was included in Annex I to Council Directive 91/414/EEC on 1 July 2006 by means of Commission Directive 2005/72/EC,<sup>3</sup> and has been deemed to be approved under Regulation (EC) No 1107/2009<sup>4</sup>, in accordance with Commission Implementing Regulation (EU) No 540/2011<sup>5</sup>, as amended by Commission Implementing Regulation (EU) No 541/2011<sup>6</sup>, EFSA initiated the review of all existing MRLs for that active substance.

As chlorpyrifos was approved before the entry into force of Regulation (EC) No 396/2005 on 2 September 2008, EFSA is required to provide a reasoned opinion on the review of the existing maximum residue limits (MRLs) for that active substance in compliance with Article 12(2) under Regulation (EC) No 1107/2009, in accordance with Commission Implementing Regulation (EU) No 540/2011, as amended by Commission Implementing Regulation (EU) No 541/2011 to complete the Pesticide Residues Overview File (PROFile) and to prepare a supporting evaluation report. Based on the conclusions derived by the European Commission in the framework of Directive 91/414/EEC, the conclusion on the peer review of the pesticide human health risk assessment of chlorpyrifos carried out by EFSA, the MRLs established by the Codex Alimentarius Commission and the additional information provided by the RMS. According to the legal provisions, EFSA shall base its reasoned opinion in particular on the relevant assessment report prepared under Directive 91/414/EEC. It should be noted, however, that, in the framework of Directive 91/414/EEC, only a few representative uses are evaluated, whereas MRLs set out in Regulation (EC) No 396/2005 should accommodate all uses authorised within the European Union (EU), and uses authorised in third countries that have a significant impact on international trade. The information included in the assessment report prepared under Directive 91/414/EEC is therefore insufficient for the assessment of all existing MRLs for a given active substance.

### **The active substance (Chlorpyrifos) of plant residues**

Chlorpyrifos (ISO common name for *O,O* -diethyl *O* -3,5,6-trichloro-2-pyridyl phosphorothioate) belongs to the group of organothiophosphate compounds (Fig.1) which are used as acaricide, insecticide or nematicide. Chlorpyrifos acts against the pest through the inhibition of acetyl-cholinesterase (AChE inhibitor) and the subsequent accumulation of acetylcholine in the nerve endings. It is used in a wide range of crops against sucking and biting pests, including pests belonging to the Coleoptera, Diptera, Homoptera and Lepidoptera.

Chlorpyrifos was evaluated under the first stage of the review programme of Directive 91/414/EEC when EFSA was not yet in charge of the risk assessment of active substances. The

evaluation resulted in the inclusion of the substance in Annex I to Directive 91/414/EEC on 1 July 2006 by Commission Directive 2005/72/EC, and has been deemed to be approved under Regulation (EC) No 1107/2009, in accordance with Commission Implementing Regulation (EU) No 540/2011, as amended by Commission Implementing Regulation (EU) No 541/2011. The European Commission launched in June 2012 a toxicological review of chlorpyrifos under Article 21 of Regulation (EC) No 1107/2009 in the light of new toxicological studies.

The EU MRLs for chlorpyrifos are established in Annexes II and IIIB of Regulation (EC) No 396/2005 and CXL(s) for chlorpyrifos were also established by the Codex Alimentarius Commission (CAC). Following a refined risk assessment regarding certain maximum residue levels (MRLs) of concern (EFSA, [2015](#)), MRLs for apples, pears, peaches, blackberries, raspberries, pineapples, potatoes, tomatoes, peppers, melons, watermelons, and sugar beet were lowered by means of Commission Regulation (EU) No 2016/60, which entered in application on 10 August 2016. An application to modify the existing EU MRLs for chlorpyrifos was assessed by EFSA ([2012](#)). However, it was preferred to await the outcome of the MRL review before implementing these MRLs into the EU legislation.

Based on the conclusions derived by the European Commission in the framework of Directive 91/414/EEC, the specific conclusion on chlorpyrifos as regards the pesticide human health risk assessment finalised by EFSA on 7 April 2014 (EFSA, [2014](#)), the MRLs established by the Codex Alimentarius Commission (codex maximum residue limit; CXLs) and the additional information provided by Member States, EFSA prepared in October 2016 a draft reasoned opinion, which was submitted to Member States for commenting via a written procedure. All comments received by 21 November 2016 were considered by EFSA during the finalisation of the reasoned opinion. Also, the chronic and acute exposure calculations for all crops reported in the framework of this review performed using the EFSA Pesticide Residues Intake Model (PRIMo) and the PROFile are key supporting documents and made publicly available.

### **Metabolic pathway of Chlorpyrifos in plant and processed farm products**

The metabolism of chlorpyrifos was sufficiently investigated for foliar treatment in oranges, radishes, head cabbage and peas. These studies allowed depicting a general metabolic pathway of chlorpyrifos in plant. The parent compound, the metabolite 3,5,6-trichloropyridinol otherwise known as 3,5,6-TCP and its conjugates (Fig.1) are the main components of the residues after foliar applications. The parent compound is mainly degraded into desethyl chlorpyrifos when subjected to standard hydrolytic conditions and was considered as toxic as the parent compound.

However, different toxicological reference values are available for chlorpyrifos and for its metabolite 3,5,6-TCP. Based on this information, two separate residue definitions for enforcement and risk assessment were proposed by EFSA. The first residue definition (specific to chlorpyrifos) includes the parent compound (in raw commodities) and its desethyl metabolite

(in processed commodities only); chlorpyrifos can be enforced in plant commodities with a limit of quantification (LOQ) of 0.01 mg/kg. The second residue definition is the sum of 3,5,6-TCP and its conjugates, expressed as 3,5,6-TCP. Since this compound is not a specific metabolite of chlorpyrifos, the first residue definition remains the most relevant for enforcement purpose but, as risk managers may consider that enforcement of metabolite 3,5,6-TCP is also necessary, an optional separate list of MRLs was also derived for the second residue definition. An analytical method is validated for analysis of 3,5,6-TCP and its conjugates with a LOQ of 0.01 mg/kg in plant commodities. Based on the same trials an optional list of MRLs, reflecting the use of chlorpyrifos, was also derived for the sum of 3,5,6-TCP and its conjugates. However, the final list of MRLs proposed for this residue definition also accommodates the use of chlorpyrifos-methyl (other source of 3,5,6-TCP in plant commodities)

Chronic and acute consumer exposure resulting from the authorised uses reported in the framework of this review was calculated for chlorpyrifos, using revision 2 of the EFSA Pesticide Residues Intake Model (PRIMo). Apart from the MRLs evaluated in the framework of this review, internationally recommended codex maximum residue limits (CXLs) have also been established for chlorpyrifos. Additional calculations of the consumer exposure, considering these CXLs, were therefore carried out and exceedances of the ARfD were identified for the existing CXLs in examined crops.

As different toxicological reference values were derived for the metabolite 3,5,6-TCP, a separate consumer risk assessment for 3,5,6-TCP and its conjugates was performed. In order to carry out a comprehensive consumer exposure calculation for metabolite 3,5,6-TCP, EFSA took into account residues arising from chlorpyrifos-methyl, chlorpyrifos and triclopyr. These chronic and acute exposure calculations were also performed using revision 2 of the EFSA PRIMo and the exposures calculated were compared with the toxicological reference values derived for the metabolite 3,5,6-TCP. Major uncertainties remain due to the data gaps identified for the metabolite 3,5,6-TCP but, this indicative exposure calculation did not indicate a risk to consumers and considering the large margin of safety, there are indications that metabolite 3,5,6-TCP is not of concern with regard to the use of triclopyr, chlorpyrifos and chlorpyrifos-methyl.

### **Nature of residues in primary crops**

In oranges and head cabbage, the parent compound, the metabolite 3,5,6-trichloropyridinol (referred to as 3,5,6-TCP) and polar metabolites represented the main part of the residues. The polar fraction, which represented the majority of the radioactivity in cabbage (75% of the total radioactive residues (TRR) at 42 days after treatment (DAT)), was characterised as 3,5,6-TCP conjugated mainly with glucose and malonic acid. In oranges, 99% of the TRR remained associated with the peel, mostly as the parent compound. Residues in pulp were < 0.01 mg eq/kg at any preharvest interval (PHI). The parent compound remains a good marker in radish roots

(41–80% TRR; > 0.91 mg/kg) and peas with pods (4–33% TRR; up to 0.2 mg/kg) and the polar metabolites represent an important part of the residues at harvest (44.7% TRR at 35 DAT in radish roots; 42.5% TRR at 28 DAT in pods). No other metabolite was present at significant level in any of the four crop groups investigated.

The metabolic pattern after foliar application is similar in the four different crop groups which were investigated. It involves the hydrolysis of the thiophosphate group to form 3,5,6-TCP, which is then readily conjugated. It is also highlighted that the results of these studies performed with chlorpyrifos are consistent with the studies performed with foliar application of chlorpyrifos-methyl in tomatoes (EFSA [2017b](#)). Therefore, it is concluded that these two substances share a similar metabolic pattern when applied as foliar spraying.

### **Nature of residues in rotational crops**

Some of the crops authorised within the EU can be grown in rotation with other plants. The soil degradation studies demonstrated that the degradation rate of chlorpyrifos and its metabolite is moderate, with a maximum DT<sub>90f</sub> of 248 and 319 days, respectively (European Commission, [2005](#)). Hence, assessment of the possible occurrence of residues in succeeding crops resulting from the use on primary crops is relevant.

Rotational field crop studies performed with chlorpyrifos were assessed in the DAR (Spain, [1999](#)). In the main study, wheat, carrots and lettuce were planted 30 and 132 DAT in a soil previously treated with <sup>14</sup>C-chlorpyrifos at a rate of 5.4 kg a.s./ha. Other studies were performed with the rate of 2.24 kg a.s./ha and 5.6 kg a.s./ha with wheat, lettuce, spinach, turnips, soybean and sugar beets but they were considered supportive only. The investigated crops were analysed at various times and at maturity for uptake of the <sup>14</sup>C activity.

According to the available data, very low amounts of chlorpyrifos (< 0.01 mg eq/kg) or 3,5,6-TCP were observed in carrots, lettuce and wheat (≤ 0.05 mg eq/kg). Another component was identified (3,5,6-trichloro-2-methoxy pyridine), but also at trace levels. The main portion of the residues appeared to be the result of incorporation into natural plant components, such as starch, cellulose and lignin. Based on this information, it is expected that relevant residue levels of chlorpyrifos and its soil metabolites will not occur in rotational crops, but this should still be confirmed by a fully validated study.

### **Nature of residues in processed commodities**

A study investigating the effect of processing on the nature of residues was provided to the RMS in the active substance renewal dossier. This study has been evaluated by the RMS and is therefore considered in the framework of the present review (Spain, [2016b](#)). It covers the representative hydrolytic conditions for pasteurisation (20 min at 90°C, pH 4),

boiling/brewing/baking (60 min at 100°C, pH 5) and sterilisation (20 min at 120°C, pH 6). This study clearly demonstrates that chlorpyrifos is readily degraded when subject to hydrolytic conditions. The level of degradation increases with temperature: 24% degradation under pasteurisation, 70% degradation under boiling/brewing/baking and 98% degradation under sterilisation. The main degradation product is desethyl chlorpyrifos (19.8–80.7% of the applied radioactivity (AR)). The metabolite 3,5,6-TCP is also observed in lower proportions: 5–10% of the AR. Therefore, the desethyl metabolite of chlorpyrifos needs to be considered in processed commodities.

### **Methods of analysis in plants**

An analytical method using gas chromatography with mass spectrometric detection using negative chemical ionisation (GC-NCI-MS) was validated for enforcement of chlorpyrifos in high water and high acid content commodities with a LOQ of 0.01 mg/kg (Spain, [2002](#)). An independent laboratory validation (ILV) was used and the method was validated for two different mass transitions though a confirmatory method was not necessary. The multiresidue QuEChERS method in combination with gas chromatography with tandem mass spectrometry (GC-MS/MS), as reported by the EURLs ([2016](#)), is sufficiently validated for analysis of chlorpyrifos in high water content commodities, high acid content commodities and dry commodities with the LOQ of 0.01 mg/kg. A high-performance liquid chromatography with tandem mass spectrometry (HPLC-MS/MS) was used for the enforcement of chlorpyrifos in high oil content commodities as reported by the RMS. This method is fully validated with a LOQ of 0.01 mg/kg hence, it is concluded that chlorpyrifos can be enforced in the four main commodity groups with a LOQ of 0.01 mg/kg. However, an ILV for high oil content commodities is still required.

However, an analytical method using HPLC-MS/MS was used for metabolite 3,5,6-TCP and the EURLs. This method is validated for analysis of 3,5,6-TCP and its conjugates with a LOQ of 0.01 mg/kg in the four main commodity groups. It is highlighted that this method used a radio labelled internal standard. In the same report, an ILV is also available for dry and high water content commodities. However, in order to release the conjugates of 3,5,6-TCP, this method involves a hydrolysis step, which is also expected to convert chlorpyrifos (and desethyl chlorpyrifos) into 3,5,6-TCP. Therefore, this method is not specific enough to enforce 3,5,6-TCP and its conjugates separately.

### **Stability of residues in plants**

In the framework of this review, the RMS has provided several studies covering the storage stability of chlorpyrifos and its metabolites. Storage stability of chlorpyrifos was demonstrated in high water content commodities, high acid content commodities and high oil content commodities for a period of 18 months as well as in dry commodities and cereal straw for a period of 24 months. The storage stability of metabolite 3,5,6-TCP was also evaluated in the

same matrices and identical results as for the parent compound were demonstrated. This study is also considered adequate to address storage stability of 3,5,6-TCP conjugates because a possible decline of such conjugates is only expected to release 3,5,6-TCP.

### **Magnitude of residues in primary crops**

All residue trial samples considered in this framework were stored in compliance with the storage conditions for which stability of residues was demonstrated. Since chlorpyrifos and its metabolite 3,5,6-TCP should be assessed separately, the residue levels related to their corresponding residue definitions were reported in two separate tables. In all trials considered in this review, analysis were carried out for the parent compound and for the total residue hydrolysed to 3,5,6-TCP. As the parent compound can be hydrolysed to 3,5,6-TCP, the absolute levels of 3,5,6-TCP and its conjugates were calculated by subtracting the contribution of the parent compound to the total residue.

It is highlighted that chlorpyrifos can be used under different types of formulations, one of them being Pyrinex 25 CS. As chlorpyrifos residues are expected to behave differently when applied with Pyrinex 25 CS formulation, the GAPs with Pyrinex 25 CS formulation and the GAPs with the other formulations were assessed independently. The number of residue trials and extrapolations were evaluated in accordance with the European guidelines on comparability, extrapolation, group tolerances and data requirements for setting MRLs (European Commission, [2016](#)). GAPs supported by trials were identified and used to derive MRL and risk assessment values from available data. However, residue trials supporting the most critical GAPs reported during this review should still be provided by Member States where these GAPs are authorised.

### **Magnitude of residues in processed commodities**

All studies investigating the magnitude of chlorpyrifos residues in processed commodities were assessed by the RMS in the framework of a former MRL application. Since Chlorpyrifos and its metabolite 3,5,6-TCP should be assessed separately, the processing factors related to the respective residue definitions were reported in two independent tables.

It is highlighted that the available processing studies do not contain analysis of the desethyl metabolite, which is relevant in many of the processed commodities that involve one of the standard hydrolysis. Therefore, robust processing factors are only derived for peeled citrus and peeled bananas where the same residue definitions as for raw commodities apply. The situation is identical for wheat bran, white flour and whole-meal flour but, due to the limited number of data, only tentative processing factors were derived for these items. For citrus (juice), plums (canned), wine grapes (juice, dry pomace, must, red wine heated and unheated, white wine), barley (beer, brewing malt and pot/pearl) and wheat (white bread and whole-meal bread), the

analysis for the desethyl metabolite was not available. Therefore, only tentative processing factors were derived.

For the main residue definition (chlorpyrifos in raw commodities and sum of chlorpyrifos and its desethyl metabolite in processed commodities), EFSA proposed to derive tentative processing factors considering the available results for total 3,5,6-TCP residue levels in processed commodities, instead of considering the sum of chlorpyrifos and its desethyl metabolite. This approach is expected to overestimate the calculated processing factors because the total 3,5,6-TCP residues include chlorpyrifos, desethyl chlorpyrifos and 3,5,6-TCP instead of considering only chlorpyrifos and its desethyl metabolite. However, most of these processing factors are supported by a very limited set of studies, especially citrus (juice), plums (canned) and wine grapes (juice, red wine heated and white wine), which are supported by one study only.

For the second residue definition (sum of 3,5,6-TCP and its conjugates, expressed as 3,5,6-TCP), processing factors were also derived. However, as the levels of desethyl metabolite was not available, it was only possible to subtract the contribution of the parent compound to the total residue hydrolysed as 3,5,6-TCP. Therefore, the absolute levels for 3,5,6-TCP and its conjugates may also be overestimated in processed commodities.

## **Assessment**

The assessment is performed in accordance with the legal provisions of the uniform principles for evaluation and authorisation of plant protection products as set out in Commission Regulation (EU) No 546/2011<sup>7</sup> and the currently applicable guidance documents relevant for the consumer risk assessment of pesticide residues (European Commission, [1996](#), [1997a-g](#), [2000](#), [2010a,b](#), [2016](#); OECD, [2011](#)).

## **Consumer risk assessment**

As different toxicological reference values were derived for chlorpyrifos and for its metabolite 3,5,6-TCP, EFSA performed separate consumer risk assessments for chlorpyrifos (resulting from the use of chlorpyrifos only) and for 3,5,6-TCP and its conjugates (resulting from the use of chlorpyrifos, chlorpyrifos-methyl and triclopyr) (FAO, [2004](#), [2006b](#)).

## **Consumer risk assessment for chlorpyrifos without consideration of the existing CXLs**

Chronic and acute exposure calculations for all crops reported in the framework of this review were performed using revision 2 of the EFSA PRIMo (EFSA, [2007](#)). Input values for the exposure calculations were derived in compliance with the decision tree. Hence, for those commodities where a (tentative) MRL could be derived by EFSA in the framework of this review, input values were derived according to the internationally agreed methodologies (FAO,

[2009](#)). For all commodities of plant origin, input values refer to the raw agricultural commodities, except for citrus fruits, bananas and wine grapes. For citrus fruits and bananas, the peeling factors derived in this review are taken into account. For wine grapes, a limited refined approach could be proposed by correcting the consumption data in the acute exposure. Indeed, it is noted that the consumption of wine grapes by children refers to grape juice while the consumption of wine grapes by adults exclusively refers to wine. In the consumption database, the consumption is expressed as raw wine grapes equivalent but 1 kg of wine grapes does not exactly produce 1 kg of wine or juice. To take this into account, the consumption data can be corrected by using a yield factor (0.7 for wine and 0.75 for juice). These considerations allowed EFSA to propose refined input values for wine grapes for British infants and adults which are the worst conservative acute diets for wine grapes. It is acknowledged that the processing factors for heated red wine and for grape juice could have been used to further refine the input values. However, the available data on the nature and magnitude of residues in processed commodities, which were evaluated in the present review, indicate that these processing factors are subject to uncertainty. The hydrolysis studies indicate that the parent compound is degraded to desethyl chlorpyrifos in processed commodities.

The exposures calculated were compared with the toxicological reference values for chlorpyrifos, derived by EFSA ([2014](#)). A chronic intake concern was identified as the highest chronic exposure represented 199% of the acceptable daily intake (ADI) (FR all population). Moreover, with regard to the acute exposure, an exceedance of the acute reference dose (ARfD) was identified for apples, pears, peaches, wine grapes (adults and children), peppers and quinces, representing 783.7%, 728.6%, 605.2%, 464.9%, 163.2%, 126.0 and 117.3% of the ARfD, respectively. A second exposure calculation was therefore performed, considering fall-back GAPs for these crops. For apples, pears and wine grapes, all the other foliar treatments (close to harvest) reported in this review would also lead to exceedances of the ARfD. According to the results of this second calculation, the highest chronic exposure was then calculated for the Dutch children and declined to 79.8% of the ADI; the highest acute exposure is then calculated for plums, representing 98.7% of the ARfD. Based on these calculations, a potential risk to consumers was identified for the most critical GAPs of chlorpyrifos on apples, pears, peaches, wine grapes, peppers and quinces (i.e. foliar treatment close to harvest). However, fall-back GAPs were identified for these crops, for which a second risk assessment did not indicate risk to consumers. For the remaining commodities, although some major uncertainties remain due to the data gaps identified in the previous sections, the indicative exposure calculation did not indicate a risk to consumers.

### **Consumer risk assessment for chlorpyrifos with consideration of the existing CXLs**

To include the CXLs in the calculations of the consumer exposure, CXLs were compared with the EU MRL proposals and all data relevant to the consumer exposure assessment have been collected from JMPR evaluations. For citrus fruits and bananas, the peeling factors derived in

this review are taken into account. For wine grapes, the same approach as explained was applied to the risk assessment values derived from CXLs.

Chronic and acute exposure calculations were also performed using revision 2 of the EFSA PRIMo and the exposures calculated were compared with the toxicological reference values derived for chlorpyrifos. The highest chronic exposure was calculated for Dutch children, representing 508% of the ADI. A second exposure calculation was therefore performed, excluding the CXLs for these crops. According to the results of this second calculation, the highest chronic exposure was then calculated for the UK infant and declined to 92.5% of the ADI; the highest acute exposure is then calculated for plums, representing 98.7% of the ARfD.

Based on these calculations, a potential risk to consumers was identified for the CXLs of chlorpyrifos on potatoes, apples, peppers, pears, tomatoes, plums and wine grapes, and no further refinements of the risk assessment were possible. For the remaining CXLs, although uncertainties remain due to the data gaps identified for some of them, the indicative exposure calculation did not indicate a risk to consumers.

### **Consumer risk assessment for the metabolite 3,5,6-trichloropyridinol**

Metabolite 3,5,6-TCP is not specific to chlorpyrifos as it is also a major metabolism product of two other active substances: chlorpyrifos-methyl and triclopyr. Hence, in order to carry out a comprehensive consumer exposure calculation for metabolite 3,5,6-TCP, EFSA took into account residues arising from chlorpyrifos, chlorpyrifos-methyl and triclopyr. In plant commodities, this metabolite is mainly expected to occur following the use of chlorpyrifos or chlorpyrifos-methyl. For these two compounds, the consumer risk assessment of the parent compounds already revealed a possible risk to consumers which could not be further refined by EFSA, and several fall-back GAPs were suggested by EFSA (2017b,c). Hence, for each plant commodity, the input value for 3,5,6-TCP is based on the highest residue level observed following the use of either chlorpyrifos or chlorpyrifos-methyl, assuming that the fall-back GAPs suggested by EFSA are implemented and that the two active substances are not used together on the same crop (EFSA, 2017).

### **Hazard of pesticides (active substances) exposure on human health and environment**

Pesticide usage generally is fraught with problems of undesirable side effects on food value chains. Many pesticides pose substantial short and long-term health risks (WHO,1990), which caused substantial risk/contamination to public health and the environment (Conway and Pretty, 1991). Uncontrollable usage of chemical pesticides in pest control disrupted the biochemical and physiological functions of erythrocytes and lymphocytes (Banerjee *et al.*, 1999). The adverse health effects include a series of chronic terminal diseases or end-points such as cancer (Settimi

*et al.*, 2003); Alavanja *et al.*, 2004), neurotoxic (Kamel and Hoppin, 2004), immunotoxic (Galloway and Handy, 2003) developmental (Colborn, 2006), endocrine (Barlow, 2005) and reproductive (Garcia *et al.*, 1999; Yucra *et al.*, 2006) and neurobehavioural effects (Amr *et al.*, 1993). This has led to the prescription of tolerances {maximum residue level (MRL)} and acceptable daily intake (ADI) as well as no observable adverse effect level (NOAEL) for various pesticides in food and water, especially by the Codex Alimentarius Commission (CODEX, 2004), and other designated authorities in several developed countries of the world like the US Environmental Protection Agency (EPA) (EPA, 1997).

Mosudi *et al.*, (2008) reported that cacao farmers in Southwestern Nigeria may have been occupationally exposed due to insecticide application for mirid control in their cacao/kola plantations; and the exposure at times is of such magnitude as to be hazardous to the farmers and their respective communities. Hence, the cacao farmers have been occupationally exposed due to the use of insecticide for mirid control in their plantations. In view of the types of insecticides commonly used and the residues detected in their blood serum and domestic water sources, there is a need to revitalize the pesticide regulation in Nigeria. Therefore, with the new EU legislation on Maximum Residue Levels (MRLs) allowed on cocoa beans and products, some of the pesticides still undergoing screening in appropriate agency (i.e CRIN) though the previously recommend pesticides were included in banned lists. This new regulation, which came into effect on 1<sup>st</sup> September, 2008, has left very few pesticides of tolerable active substance for use on cocoa both on farm and post farm activities in Nigeria (Asogwa and Dongo, 2009).

### **Government agency and other regulatory bodies intervention**

The regulatory Agency needs to become more alive to its responsibilities in enforcement and the prescription of standard safety measures such as acceptable daily intake (ADI) and no observable adverse effect level (NOAEL) for various pesticides being used in the country.

The Ministry of Agriculture, Ministry of Health, NAFDAC, the Standard Organisation of Nigeria (SON) and the newly inaugurated National Food Safety Management Committee should see the EU ban as a wake-up call to sanitise food exported and those being consumed at home. They need to conduct their regulatory oversight properly and need to put stringent measures in place to monitor Nigerian products and guarantee them safe for export.

Asogwa and Dongo (2009) suggested that a joint pesticide monitoring and regulatory task force should be set up to enforce the removal and disposal of banned chemicals from circulation. The government and agrochemical companies also should ensure the constant availability in the markets of those active ingredients that are within the new class of allowed pesticides at reasonable costs. It is only when that is done that the non proliferation of banned agrochemicals outside the “EU Regulation 149/2008/EC” can be guaranteed amongst the cocoa farmers in Nigeria.

Synthetic insecticides such as Actara 25 WG, Esiom 150 SL, Proteus 170 0-TEC, Avesthrin, Termicid, Capsida, Phostoxin, Zap, Confidor etc. were screened against attacks of kola weevils/mirids. This is to determine biodegradability of the insecticides with little or no residue effect that may pollute the environment. Among the chemical insecticides screened Actara 25 WG, Esiom 150 SL and Proteus 170 0-TEC passed through all the stages and considered effective, and thus recommended for use on Kola/Cocoa tree or their products.

However, Phostoxin is recommended as a fumigant in controlling storage pests. Other chemicals aside recommended ones are still under investigation (Table 2). Optimum dosage concentration of (0.25 ml/10 l) synthetic insecticide is recommended for use on tree crops and treated products. One quarter of phostoxin tablet is recommended for the preservation of kola nuts. However, the fragment of phostoxin will be enclosed in a perforated envelop and placed in a basket full of kola nuts. The preferable position of placing the perforated envelop is at the middle of the basket filled kola nut. Therefore this would enhance adequate diffusion of fumigant effect against kola weevil infestation. Hence, the perforated envelop avoided contact between the nuts and phostoxin tablet. However, phostoxin is poisonous and it is advisable farmers/traders should spread the stored kola nuts meant for sale in a ventilated environment so that the concentration of the chemical would diffuse into the air. Kola nuts should be handled with care because it does not require further processing before it is consumed and mindful of application rates (Azeez, 2015b).

#### **Use of bio-rational/botanicals as alternative method of control**

There has been a renewed interest in the use of natural products in the protection of stored agricultural products, following the growing awareness of the capacity of synthetic insecticides to contaminate stored commodities with harmful residues, caused health hazards to farm households and wide spread environmental pollutions. Plant products used as protectants of stored agricultural commodities are normally obtained from leaves, roots, flowers, fruits, seeds, bark and stems. In Nigeria, many local plant products such as peppers, ashes, vegetable oils, citrus peels, pawpaw leaves, cedar, neem tree products etc have been used successfully for the control of insect pests of stored products. These alternative bio-pesticides have been adjudged safe, biodegradable and environmental friendly (SU, 1977; Ofuya, 1986; Jackai, 1993).

The paradigm shift in global demand for environmentally sound pest management strategies necessitated the need to develop alternative pesticides with minimal or non-ecological hazards. Concerted effort so far on the development of botanicals has resulted in the commercialization of “azadirachtin”, a highly potent allelochemical from the tropical neem plant, *Azadirachta indica* A Juss. This is in agreement with the findings by Nisbet (1992) that compounds like aztin and azadirachtin, a limonoid extracted principally from the seeds of the neem and thus attributed the insecticidal properties to an ecdysone-like type of action. In the same vein, Enobakhare and Azeez, (2006) reported that there was an appreciable reduction in oviposition and adult bruchid emergence on treated cowpea seeds with powder and aqueous extracts of *A. indica* and *Nicotiana*

*tabacum*.

Natural chemicals (botanicals) prepared from parts of plant such as neem (*Azadirachta indica*), tea (*Cymbopogon citratus*), stool wood (*Alstonia boonei*), teak (*Tectonia grandis*) etc could be used in protecting kola nuts against devastating effect of kola weevils. The botanicals with any of the tested application strategies could provide effective control of kola weevils (Azeez, 2015c; 2016). However, the plant materials can serve dual purpose for protecting kolanuts as well as retaining the high percentage of moisture (Azeez, 2016). This agreed with findings reported by Azeez, (2016) that leaves pesticidal plants (*Azadirachta indica*, *Tectonia grandis*, *Musa paradisiacal* and *Alchornea cordifolia*) could serve dual purpose of protecting nuts as well as keeping the nut crispness during and after storage. Additionally, good hygiene (Cultural method) such as timely harvest, proper cleaning of the store, selection of good nuts etc should be practiced to have insect free kola nuts.

Asogwa *et al.*, (2009) reported that storage of *kola nitida* at  $2.5 \times 10^3$  ppm of *Cederela odorata*, *Khaya* spp, *Azadirachta indica*, *Chromolena odorata* and *Chrysophyllum albidum* ethanolic extracts were effective against damage of kola weevil. Therefore, the various extracts could be extended as alternatives to kola farmers, so as to reduce their total dependence on synthetic insecticides for kolanut storage. Oyedokun *et al.*, (2011) reported that the aqueous extracts of *Phyllanthus amarus*, *Acassia albida* and *Tithonia diversifolia* caused 40-56%, 24-60% and 42-88% mortality of termite, after 140 minutes of exposure (MOE) to the extracts. Similarly, ethanolic extracts of the *P. amarus*, *A. albida* and *T. diversifolia* resulted in a significantly ( $P < 0.05$ ) higher percentage mean mortality of 64-91%, 36.4-76% and 36-68% respectively. The findings could be the panacea to the devastating subterranean pest, *Macrotermes bellicosus* in Kola/Cocoa tree plantation. Termites feed on dead vegetation; their tunnels may weaken plant stems, causing lodging and secondary infections e.g. fungus and other diseases. It also causes damages on the field by attacking the trunks and pods of cocoa/kola tree causing the plant and the pods to dry up after severe infestations.

## Conclusions

Nigeria cash crop farmers are still dependent on pesticides to reduce insect infestation and increase productivity to attain desirable acceptable levels of self sufficiency and exports. The use of pesticides for effective pests control has generated a lot of concerns relating to public health and environmental pollution. However, cash crop like Kola nuts need no further processing before consumption hence little or no chemical residue therein will be acceptable, perhaps sparingly chemical should be used for storage pests. The screening of new insecticides, fungicides and herbicides, new spraying pumps are usually evaluated by the Cocoa Research Institute of Nigeria (CRIN), for their efficiency before they are recommended for use in the

application of cocoa pesticides and spraying equipment in Nigeria.

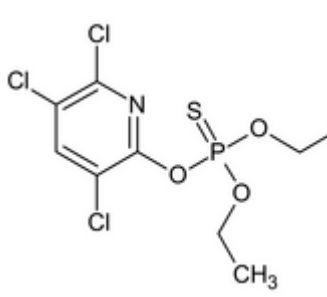
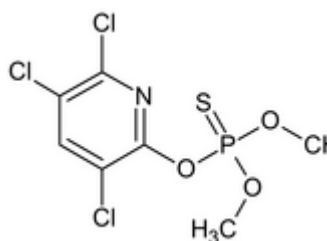
The recent ban on farm products export by EU due to unacceptable tolerable level of residues was of great concerns. Checklists of banned insecticides included Acephate, Amitraz, Aldrin, Azinphos-methyl, Cabaryl, Cabofuran, Carbosulfan, Cartap, Terbufos, Cyhexatin, DDT, Dichlorvos, Dieldrin, Endosulfan, Lindane, Methyl-parathion, Methonmyl, Monocrotophos, Profenfos, Promecarb, Propoxur etc. However some are on watchlist which composed of active ingredients that are not banned that have a potentially severe and/or cumulative risk for human health and/or the environment. Thus, EU banned some Kola/Cocoa pesticides and approved some because of health and environmental concerns on pesticide residue on Kola/Cocoa products.

The metabolism of active substance e.g. chlorpyrifos was sufficiently investigated as a major source of plant residue, since insecticides come in different trade names. The general metabolic pathway of chlorpyrifos in plant was properly reported. Thus, the parent compound, the metabolite 3,5,6-TCP and its conjugates are the main components of the residues after foliar applications. Furthermore, the nature of chlorpyrifos residues in rotational crops indicates that significant residues uptake was not significant compared with plants and processed products. It was reviewed that the parent compound is mainly degraded into desethyl chlorpyrifos when subject to the standard hydrolytic conditions. However, different toxicological reference values are available for chlorpyrifos and for its metabolite 3,5,6-TCP.

Based on this information, two separate residue definitions for enforcement and risk assessment were proposed by EFSA. The first residue definition (specific to chlorpyrifos) includes the parent compound (in raw commodities) and its desethyl metabolite (in processed commodities only); chlorpyrifos can be enforced in plant commodities with a LOQ of 0.01 mg/kg, while analytical methods are not available for its desethyl metabolite. The second residue definition is the sum of 3,5,6-TCP and its conjugates, expressed as 3,5,6-TCP. Since this compound is not a specific metabolite of chlorpyrifos, the first residue definition remains the most relevant for enforcement purpose but, as risk managers may consider that enforcement of metabolite 3,5,6-TCP is also necessary, an optional separate list of MRLs was also derived for the second residue definition. An analytical method is validated for analysis of 3,5,6-TCP and its conjugates with a LOQ of 0.01 mg/kg in plant commodities. Continuous usage of unapproved insecticides generally responsible for undesirable side effects on food value chains. Consequently, disrupted the biochemical and physiological functions of blood cells and malfunction of vital organs that caused health challenges like chronic terminal diseases

The regulatory Agency needs to become more alive to its responsibilities in enforcement and the prescription of standard safety measures such as acceptable daily intake (ADI) and no observable adverse effect level (NOAEL) for various pesticides being used in the country. They need to conduct their regulatory oversight properly and needs to put stringent measures in place to

monitor Nigerian products and guarantee them safe for export. The government and agrochemical companies also should ensure the constant availability in the markets of those active ingredients that are within the new class of allowed pesticides at reasonable costs. It is only when that is done that the non proliferation of banned agrochemicals outside the “EU Regulation 149/2008/EC” can be guaranteed amongst the cocoa farmers in Nigeria.

| Code/trivial name   | Chemical name/SMILES notation   | Structural formula  |
|---------------------|---|---|
| Chlorpyrifos        | <i>O,O</i> -Diethyl <i>O</i> -3,5,6-trichloro-2-pyridyl phosphorothioate<br><br>Clc1 cc(Cl)c(Cl)nc1OP(=S)(OCC)OCC |  |
| Chlorpyrifos-methyl | <i>O,O</i> -Dimethyl <i>O</i> -3,5,6-trichloro-2-pyridyl phosphorothioate<br><br>Clc1 cc(Cl)c(Cl)nc1OP(=S)(OC)OC  |  |

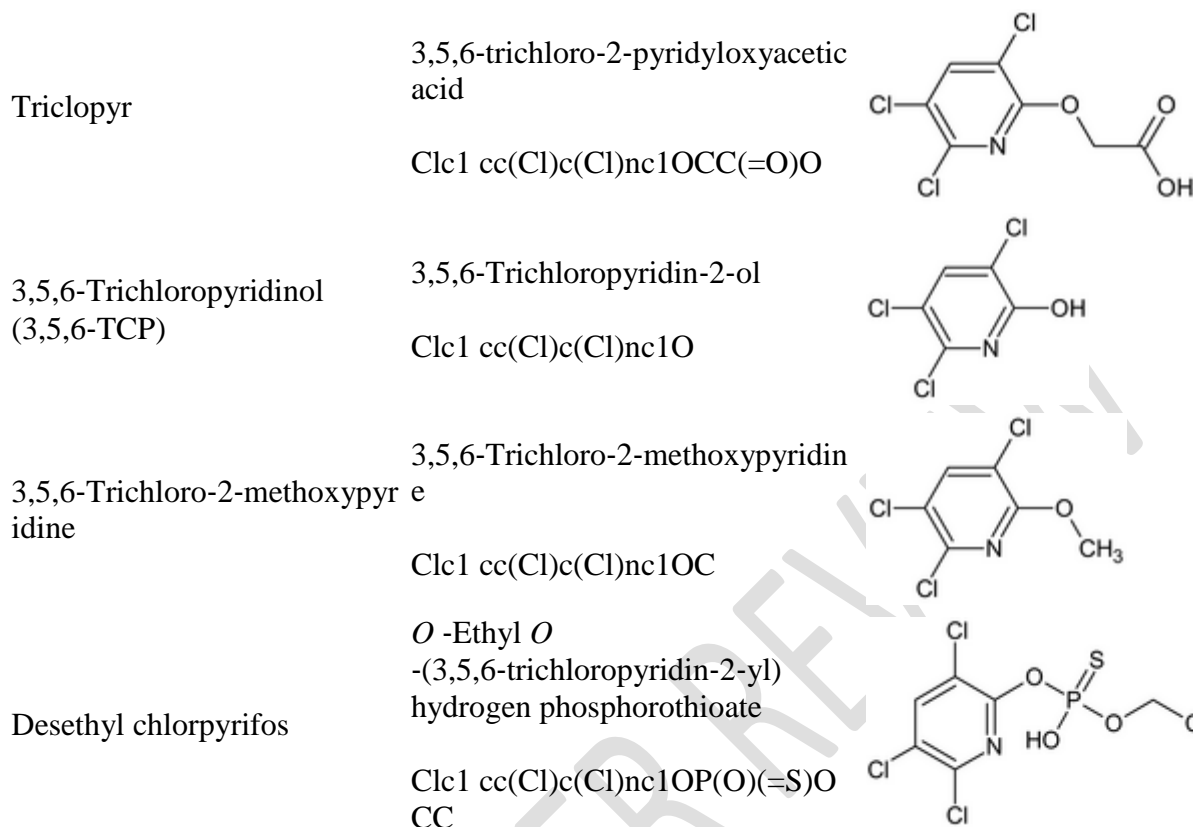


Fig 1: SMILES: simplified molecular-input line-entry system.

Table 1: Economic insect pests of cash crops

| S/N | Crops                                      | Scientific names of insect pests    | Common name | Parts attacked | Pest status    |
|-----|--|-------------------------------------|-------------|----------------|----------------|
| 1   | <i>Theobroma cacao</i> L                   | <i>Ephesttia cautella</i> Walker    | Moth        | Cocoa seed     | Store          |
|     |  | <i>Sahlbergella singularis</i> Hagl | Brown mirid | Pod/follicle   | Field          |
|     |  | <i>Characoma stictigrapta</i> Hmps  | Pod borer   | Pod/nut        | Field          |
|     |  | <i>Anaphe venata</i> Bulter         | Moth        | Leaves         | Field          |
| 2   | <i>Cola acuminata</i> ; <i>Cola nitida</i> | <i>Balanogastris kolae</i> Desbr    | Kola weevil | Pod/nut        | Field to store |
|     |  | <i>Phosphorus virescens</i> Oliver  | Stem borer  | Stem           | Field          |
|     |  | <i>Ceratitis colae</i> Silv         | Fruit fly   | Pod/nut        | Field          |
|     |  | <i>Sylepta derogata</i> Hmps        | Moth        | Leaves         | Field          |
| 3   | <i>Coffea Arabica</i> L                    | <i>Scephanoderes hampei</i>         | Fruit borer | Fruit          | Field          |
|     |  | <i>Cephanodes hylas</i>             | Leaf feeder | Leaves         | Field          |

|   |                            |                                    |               |             |                |
|---|----------------------------|------------------------------------|---------------|-------------|----------------|
|   |                            | <i>Leucoptera dohertyi</i>         | Leaf feeder   | Leaves      | Field          |
|   |                            | <i>Coccus viridis</i>              | Scale insect  | sucker      | Field          |
| 4 | <i>Camellia sinensis</i> L | <i>Dysdercus supersitiosus</i>     | Plant Bug     | sucker      | Field          |
|   |                            | <i>Helopetic schoutednii</i>       | Plant bug     | Sucker      | Field          |
|   |                            | <i>Lagris villosa</i>              | Leaf beetle   | Leaf        | Field          |
|   |                            | <i>Gryllus domesticus</i>          | Ground beetle | Root        | Field          |
| 5 | <i>Vigna unguiculata</i> L | <i>Callosobruchus maculatus</i> F. | Bean beetle   | Cowpea seed | Field to store |
|   |                            | <i>Maruca vitrata</i>              | Pod borer     | Pod/flower  | Field          |
|   |                            | <i>Megalurothrips sjostedti</i>    | Bud Thrips    | Flowers     | Field          |
|   |                            | <i>Mirperus jaculus</i>            | Pod bug       | Green pod   | Field          |

Source : Asogwa et al., 2006

Table 2: Pesticides currently approved for use on Kola/Cocoa farms

| S/N | Trade name         | Active ingredient                          | Distribution company in Nigeria | Target pest |
|-----|--------------------|--|---------------------------------|-------------|
|     | <b>Insecticide</b> |  |                                 |             |
| 1   | Actara 25 WG       | Thiamethoxam                               | SYNGENTA                        | Mirid       |
| 2   | Esiom 150 SL       | Acetamiprid + Cypermethrin                 | INSIS                           | Mirid       |
| 3   | Proteus 170 - Tec  | Thiacloprid 150 g/l + Delta methrin 20 g/l | SARO                            | Mirid       |

|   |                     |                              |              |              |
|---|---------------------|------------------------------|--------------|--------------|
|   | <b>Fungicide</b>    |                              |              |              |
| 1 | Funguran - OH       | Copper hydroxide             | INSIS        | Black pod    |
| 2 | Champ DP            | Copper hydroxide             | SARO         | Black pod    |
| 3 | Ridomil gold 66 WP  | Cuprous oxide + metalaxyl-M  | SYNGENTA     | Black pod    |
| 4 | Copper Nordox 75 WP | Cuprous oxide                | DIZZENGOFF   | Black pod    |
| 5 | Ultimax plus        | Metalaxyl + Copper hydroxide | HARVESTFIELD | Black pod    |
| 6 | Kocide 101          | Cuprous Oxide                | SARO         | Black pod    |
| 7 | Kocide 2000         | Cuprous hydroxide            | DUPONT       | Black pod    |
|   | <b>Herbicides</b>   |                              |              |              |
| 1 | Touch down          | Glyphosate                   | SYNGENTA     | Weed         |
| 2 | Clear weed          | Glyphosate                   | HARVESTFIELD | Weed         |
| 3 | Round up            | Glyphosate                   | CANDEL       | Weed         |
|   | <b>Fumigants</b>    |                              |              |              |
| 1 | Phostoxin           | Aluminum phosphide           | GONGONI      | Storage pest |

Source: Asogwa, 2014a

Table 3: List of pesticides that must not be used for Cocoa production

| S/N               | Active ingredient                | MoA group | EU, MRL status |
|-------------------|----------------------------------|-----------|----------------|
| Field application |                                  |           |                |
| 1                 | Acephate                         | 1B        | N              |
| 2                 | Amitraz                          | 19        | N              |
| 3                 | Aldrin                           | 2         | N Class 1      |
| 4                 | Azinphos-methyl                  | 1B        | N Class 1      |
| 5                 | Cabaryl                          | 1A        | N              |
| 6                 | Cabofuran (as spray formulation) | 1A        | N Class 1      |
| 7                 | Carbosufan                       | 1A        | N              |
| 8                 | Cartap                           | 4C        | N              |
| 9                 | Cyhalothrin (unresolved)         | 3         | N              |

|                          |                            |     |                             |
|--------------------------|----------------------------|-----|-----------------------------|
| 10                       | Cyhexatin (acaricide)      | 12B | N                           |
| 11                       | DDT (may be used for IRS)  | 3   | N                           |
| 12                       | Dichlorvos (DDVP)          | 1B  | N Class 1                   |
| 13                       | Dieldrin                   | 2   | N Class 1                   |
| 14                       | Dioxacarb                  | 1A  | N                           |
| 15                       | Endosulfan                 | 2   | N* Class1(MRL<br>0.1 mg/kg) |
| 16                       | Lindane, gamma BHC, HCH    | 2   | N*                          |
| 17                       | Meyhyl-Parathion           | 1B  | N*Class 1                   |
| 18                       | Methonmyl                  | 1A  | N Class 1                   |
| 19                       | Monocrotophos              | 1B  | N Class 1                   |
| 20                       | Profenfos                  | 1B  | N                           |
| 21                       | Promecarb                  | 1A  | N Class 1                   |
| 22                       | Propoxur                   | 1A  | N                           |
| 23                       | Terbufos                   | 1B  | N Class 1                   |
| <b>Store application</b> |                            |     |                             |
| 1                        | Allethrin (esbiothrin)     | 3   | N                           |
| 2                        | Fenitrothion               | 1B  | N                           |
| 3                        | Isoprocarb (MIPC)          | 1A  | Not listed                  |
| 4                        | Permethrin                 | 3   | N                           |
| 5                        | Resmethrin                 | 3   | N                           |
| 6                        | Tetramethrin               | 3   | N                           |
| <b>Fungicides</b>        |                            |     |                             |
| 1                        | Benomyl                    | B 1 | N                           |
| 2                        | Captafol                   | M 4 | N                           |
| 3                        | Hexaconazole               | G 1 | N                           |
| 4                        | Pyrifenox                  | G 1 | N                           |
| 5                        | Triadimefon                | G 1 | N                           |
| 6                        | Tridemorph                 | G 2 | N                           |
| 7                        | Zineb                      | M 3 | N                           |
| 8                        | Copper Sulphate            |     |                             |
| 9                        | Carbide                    |     |                             |
| <b>SHerbicides</b>       |                            |     |                             |
| 1                        | Ametryn                    | C 1 | N                           |
| 2                        | Atrazine                   | C 1 | N                           |
| 3                        | Diuron                     | 0   | N                           |
| 4                        | Fomesafen                  | E   | N                           |
| 5                        | MSMA (methyl arsenic acid) | Z   | N                           |
| 6                        | 2, 4, 5- T                 | 0   | N                           |

Source: Asogwa (2014b)

Notes:\*- High residue levels have been found within the last 5 years in imported produce to the EU and/or Japan

N – Not known to be on 9141/EC

P - Pending

Table 4. Residue definition of chlorpyrifos for recommended plants

| Code number  | Commodity    | Existing EU MRL (mg/kg) | Existing CXL (mg/kg) | MRL (mg/kg) | Outcome of the review<br>Comment |
|--|--------------|-------------------------|----------------------|-------------|----------------------------------|
| <b>Enforcement residue definition (existing):</b> chlorpyrifos <a href="#">F</a> |              |                         |                      |             |                                  |
| <b>Enforcement residue definition (proposed):</b> chlorpyrifos <a href="#">F</a> |              |                         |                      |             |                                  |
| 110020   | Oranges      | 0.3                     | 1                    | 1.5         | Recommended <a href="#">a</a>    |
| 163020   | Bananas      | 3                       | 2                    | 4           | Recommended <a href="#">a</a>    |
| 620000   | Coffee beans | 0.2                     | 0.05                 | 0.05        | Recommended <h href="#">h</h>    |

| Code number | Commodity             | Existing EU MRL (mg/kg) | Existing CXL (mg/kg) | Outcome of the review |                                       |
|-------------|-----------------------|-------------------------|----------------------|-----------------------|---------------------------------------|
|             |                       |                         |                      | MRL (mg/kg)           | Comment                               |
| 500050      | Oats grain            | 0.05*                   | –                    | 0.6                   | Recommended <u>c</u>                  |
| 500060      | Rice grain            | 0.05*                   | 0.5                  | 0.5                   | Recommended <u>h</u>                  |
| 500070      | Rye grain             | 0.05*                   | –                    | 0.15                  | Recommended <u>c</u>                  |
| 500080      | Sorghum grain         | 0.05*                   | 0.5                  | 0.5                   | Recommended <u>h</u>                  |
| 500090      | Wheat grain           | 0.05*                   | 0.5                  | 0.5                   | Recommended <u>j</u>                  |
| 300030      | Peas (dry)            | 0.05*                   | –                    | 0.01*                 | Further consideration needed <u>d</u> |
| 900010      | Sugar beet (root)     | –                       | –                    | 0.1                   | Further consideration needed <u>o</u> |
| 500040      | Millet grain          | 0.05*                   | –                    | 0.01*                 | Further consideration needed <u>d</u> |
| 610000      | Tea (dried leaves and | 0.1*                    | 2                    | 2                     | Further consideration                 |

| Code number | Commodity                   | Existing EU MRL (mg/kg) | Existing CXL (mg/kg) | Outcome of the review |   |
|-------------|-----------------------------|-------------------------|----------------------|-----------------------|---|
|             |                             |                         |                      | MRL (mg/kg)           | Comment                                   |
|             | stalks)                     |                         |                      |                       | needed <sub>e</sub>                       |
| 260040      | Peas (fresh, without pods)  | –                       | –                    | –                     | Further consideration needed <sub>r</sub> |
| 300010      | Beans (dry)                 | –                       | –                    | 0.01*                 | Further consideration needed <sub>o</sub> |
| 401050      | Sunflower seed              | –                       | –                    | 0.01*                 | Further consideration needed <sub>o</sub> |
| 401060      | Rape seed                   | –                       | –                    | 0.3                   | Further consideration needed <sub>o</sub> |
| 401070      | Soya bean                   | –                       | –                    | –                     | Further consideration needed <sub>r</sub> |
| 401080      | Mustard seed                | –                       | –                    | 0.3                   | Further consideration needed <sub>o</sub> |
| 401090      | Cotton seed                 | –                       | –                    | 0.09                  | Further consideration needed <sub>p</sub> |
| 620000      | Coffee beans                | –                       | –                    | –                     | Further consideration needed <sub>q</sub> |
| 810000      | Spices (seeds)              | –                       | –                    | –                     | Further consideration needed <sub>q</sub> |
| 820000      | Spices (fruits and berries) | –                       | –                    | –                     | Further consideration needed <sub>q</sub> |
| 840000      | Spices (roots and rhizome)  |                         |                      |                       | Further consideration needed <sub>q</sub> |

| Code number | Commodity | Existing EU MRL (mg/kg) | Existing CXL (mg/kg) | Outcome of the review MRL (mg/kg) | Comment |
|-------------|-----------|-------------------------|----------------------|-----------------------------------|---------|
|-------------|-----------|-------------------------|----------------------|-----------------------------------|---------|

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Authors have declared that no competing interests exist. The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

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