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Assessment of pesticides residues in Nigerian honey and their potential health risk on consumption

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Abstract
Residual level and dietary health risk assessment of pesticides were measured in raw (RHS) and commercial (CHS) honey samples from Hong and Mubi municipal area of Adamawa State, Nigeria. The concentrations of the pesticides were observed to be significantly ($p<0.05$) higher in the CHS when compared to the RHS. More so high in samples from Mubi than samples from Hong. All the pesticides (Except cypermethrin, and permethrin in RHS from Hong) were detected in both the RHS and CHS from Mubi and Hong. Higher concentrations of about 0.430±0.014 mg/kg, 0.240±0.014 mg/kg, and 0.230±0.011 mg/kg above the maximum residual level (MRL) were observed for aldrin, metachlor, and atrazine in the CHS from Mubi. The dietary exposure were observed to be significant ($p<0.05$) in children compared to adults. The Target hazard quotient (THQ) and the Health Index (HI) for the pesticides in the RHS and CHS from both Hong and Mubi were all less than (<1) one for both age categories. Exposure to the pesticides through the consumption of the CHS is however higher than the THQ values recorded for the RHS and were also observed not to pose any HR. When compared between the two locations, the probability for non-carcinogenic risk is much higher for sample from Mubi compared to the same samples from Hong and much higher for children than adults. The results indicate that aldrin, and heptachlor are of particular concern because of their high THQ and HI levels in the CHS.

Keywords: Honey, pesticides, health risk, acceptable daily intake

Introduction
Conceptually, human being amidst all odds stands out as the most destructive pathogen the earth is fighting assiduously to resist. Living behind destructive footprint from agrochemicals in her wake to meet food security and sufficiency. Several efforts were made to tame the onslaught engineered by the indiscriminate use of pesticides by human in agricultural enterprise. These efforts due to several environmental, social, economic and cultural ubiquity were observed to differ amongst regulatory jurisdictions (Li, 2017) [1]. Migration of pesticides and other agrochemicals into the foods chain were observed to infringe on the right to health and quality food and stood against environmental best practices (Gerage *et al.*, 2017) [2]. These unwholesome agricultural practices were reported to leads to several health-related complication from pesticides poisoning. An estimated 3 million cases of pesticides poisoning and about 250,000 pesticides-poisoning related death cases are reported globally (https://www.bbc.com/news/world-africa-32372502) [3]. From the figures presented, most of the reported cases were observed to emanate from the developing countries. The UN report shows only 35% of the developing countries had regulatory guidelines on pesticide (Project Syndicate, 2017) [4]. In 2015, pesticides poisoning were implicated as the probable cause of 18 mysterious deaths in the south-western Nigeria. The symptoms recorded within 24 hours from the exposure includes blurred vision, headaches, and loss of consciousness (https://www.bbc.com/news/world-africa-32372502; Project Syndicate, 2017) [3, 4]. Even though these pesticides are widely regulated globally, same were rarely monitored in most developing economies. (Al-Waili *et al.*, 2012; Korta *et al.*, 2002; Mullin *et al.*, 2010; Frazier *et al.*, 2008; Martel *et al.*, 2007; Codex Alimentarius, 1998) [5-10]. Thus, compromising the much desired needs for food security in such countries.
Relative to crops, meats, water and other edible plants, pesticides residues have been found in bee's colony samples, beeswax, pollen, and bee bread (Eissa and Zidan, 2014; Essa *et al.*, 2014) [11, 12]. In addition to their roles in establishing a crosstalk between pollinating plants, honey bees are considered a social species with great economic values (Toledo *et al.*, 2018) [13]

Demonstrate their social skills during foraging activity by maintaining colony food reserve by means of food recruitment communication (Toledo *et al.*, 2018; Wilms *et al*., 2010; Mitchell *et al*., 2017, Tosi *et al*., 2018; Calatayud-Vernich *et al*., 2016; Pettis *et al*., 2012) [13-18]. Several studies have documented the presence of pesticide residues of various classes (Organochlorine, (OCs), organophosphates (Ops), pyrethroids, organonitrogen, and carbamates) in honey (Eissa and Zidan, 2014, Eissa *et al*., 2014; Naggar *et al*., 2017; Claeys *et al*., 2011; Blanco *et al*., 2011; Barganska *et al*., 2013; Gallagher *et al*., 2015) [11, 12, 19-23]. Hives foraging on pesticides cultivated crops could through several pollinating mechanism transfer the metabolites into the honey with a potential risk to the consumer through the food chain (Eissa *et al*., 2014) [12]. Relative to other insect genomes, the honey bee genome is markedly deficient in the number of genes encoding detoxification enzymes, including cytochrome P450 monooxygenases (P450s), glutathione-S-transferases, and carboxylesterases (Claudianos *et al*., 2006) [24]. Thus, pesticides residues taken up by the bees during foraging, based on this incomplete detoxification processes transfer most of the active metabolites into the honey products. Therefore, the determination of pesticides residues in honey and other bee products has become a growing concern considering the growing popularity of honey in human daily diets. Studies shows that, diet especially for non-occupational exposed individuals contributes an average of five times more pesticides to the body burden than all other routes of exposure, such as air and drinking water (Claeys *et al*., 2011; Naggar *et al*., 2017) [19, 20],. This route were also observed to aid exposure to multiple pesticides residues, compounding the toxicological mechanism and increasing the risk to a level of concern (Gallagher *et al*., 2015; Judge *et al*., 2016) [23, 25]. Some effort were made to evaluate honey in Nigeria. These effort tends toward evaluating the physicochemical, microbial and biochemical properties of the honey (Ndife *et al*., 2014; Buba *et al*., 2013; Lullah-Deh *et al*., 2018; Adebiyi *et al*., 2004; Lawal *et al*., 2009. Oladipupo *et al*., 2009, James *et al*., 2009; Lawal, *et al*., 2010) [26-33]. In all these efforts, no related information involving dietary risk assessment of pesticides residues in Nigerian honey samples.

In light of these concerns, dietary human health risk assessment of both raw (RHS) and commercially (CHS) obtained honey samples collected from Hong and Mubi local government area of Adamawa were appraised and presented in this study in other to establish their potential risk to public health. The outcome is to savor policy program that will foster health-based best practices in the honey market with the view to ensure sustainable food security.

Materials and Methods

The RHS used in this study were obtained from beehives at the local bee farms in Hong and Mubi municipal area of Adamawa State, Nigeria. Similarly, the CHS were purchased in the market in the same municipal area. The extraction and purification of the pesticides (Aldrin, heptachlor, chlorpyrifos, fenthion, cypermethrin, permethrin, metachlor and atrazine) residues from the honey samples prior to instrumental analysis were carryout based on the procedure described by Lehotay *et al., (*2005) [34]. The respective honey samples, approximately 5g of was taken and dissolved in 10 ml de-ionized water. Followed by the addition of acetonitrile acidified with acetic acid (10 ml), 1.0 g sodium acetate and 4.0 g anhydrous magnesium sulphate after shaking for at least a minute. And further shacked vigorously for another minute. Following the centrifugation at 4,000 rpm for two minutes, 6 ml of the extract were transferred into 15 ml glass tube containing 0.4 g primary secondary amine (PSA) sorbent and 0.6 g anhydrous magnesium sulphate. The formed mixture were then vigorously shaken for one minute and centrifuged at 4000 rpm for 2 minutes. The residue was dissolved in 2 ml of injection standard and passed through a 0.50 µm sized pore filter and quantified using HPL. The data obtained from all the analysis were statistically integrated and presented as mean ± S.D of three replicate analysis using Graph phard-prism (version 6.0), One-way ANOVA and students T-test. The level of significance was sets at $P<0.05$.

Health risk characterization

To characterize the potential HR of the pesticides residue in the honey, some assumption were factored into the mix to help in computing the HR indices. The Estimated Daily Intake (EDI) for the respective pesticides were estimated using the description in equation 1 (Forkuoh *et al*., 2018; USEPA, 1997) [35, 36]. The potential non-carcinogenic risk determined using the Target Hazard Quotient (THQ) and the health index (HI) expressions described in equation 2 (USEPA, 1997) [36].

$$\text{EDI} = \frac{Ch\ x\ H_{\text{IR}}}{BW} \qquad (1)$$

The C_h is the pesticides concentration (mg/kg) in the honey, H_{IR} represents the average honey consumption rate or intake rate for an average child and adults. The *BW* is the average body weight of children (15 kg) and adults (60 kg) (USEPA, 2000; Akbari *et al*., 2012) [37, 38].

The ADI values used in equation 2 represents the reference oral dose values set as an estimate for the tolerable daily intake of pollutants that will pose no health risk during a lifetime (FAO/WHO, 2002, 2010; USEPA, 1996; Bwatanglang and Magili, 2019; Bwatanglang, 2019) [39, 40-43].

$$\text{THQ} = \frac{EDI}{ADI} \qquad (2)$$

The cumulative effects (HI) of the individual pesticides to induced potential non-carcinogenic risk were estimated using the description in equation 3 (USEPA, 1997) [36]. The HI, expressed as the sum of THQ is the cumulative effect pose by the combination of the individual pesticides presents in the honey (Forkuoh *et al*., 2018; Reffstrup *et al*., 2010; USEPA. 2016) [35, 44-45].

$$HI = \frac{EDI_1}{ADI_1} + \frac{EDI_2}{ADI_2} + \frac{EDI_3}{ADI_3} + \ldots.. \frac{EDI_i}{ADI_i} = \sum_{i=1}^{n} \frac{EDI_i}{ADI_i} \qquad (3)$$

Were EDI_i and ADI_i are respectively the estimated and acceptable daily intake dose of the individual pesticides in the honey.

Results and Discussion

Concentration of pesticide residues detected in honey from Hong and Mubi

The average mean concentration of pesticide residues in the honey samples (both raw and commercial) collected from Hong and Mubi are presented in figure 1. The results were compared with the maximum residue limits (MRLs) for pesticides in honey (EC, 2005, Darko *et al*., 2017) [46, 47]. The MRLs for pesticides in honey were adopted from European MRL due to lack of available MRL for honey in Codex

Alimentarius Commission. An MRL of 0.01 mg kg^{-1} set by the European regulation 396/2005 EC were adopted for substances for which no MRL were allocated (EC, 2005) [46]. The pesticides with the highest concentration in the raw honey samples (RHS) collected in Hong as shown in Fig.2a is metachlor (0.064±0.002 mg/kg) follow by heptachlor (0.039±0.005 mg/kg) and atrazine (0.044±0.001 mg/kg). As observed in the figure, the concentration of the pesticides were found to be above the MRL except for chloropyrifos, cypermethrin, and permethrin whose concentrations were found to below the MRL. Furthermore, when compared to the RHS, higher concentration of the pesticides were detected in the commercial honey samples (CHS) obtained in Hong (Fig. 2b). All the pesticides (except cypermethrin, in the RHS) investigated in this study were detected in both the RHS and CHS from Mubi. The concentrations were observed to be higher compared to the values detected in samples from Hong. Higher concentrations of about 0.430±0.014 mg/kg, 0.240±0.014 mg/kg, and 0.230±0.011 mg/kg were observed for aldrin, metachlor, and atrazine in the CHS from Mubi. These values were observed to be significantly ($p<0.05$) higher compared to the concentrations in the RHS. Similarly, when compared to both the RHS and the CHS, the concentrations of the pesticides residues in the samples in Mubi as shown in the figures were all above the MRL and much higher than the values recorded in samples from Hong.

The results from this study agrees with several studies detecting residual level of pesticides in honey. In a study conducted by Kumar and Bidi, (2018) [48], about 72% of market honey sample from northern India were found to be positive for both organophosphates and organochlorines pesticides. According to the study, the concentration of the pesticides in most of the uncertified branded and unbranded honey were found to be above the MRL. In another related study, about 88.44% and 93.33% of honey samples, and 22.22% and 100% of pollen samples of *S. mexicana* and *A. mellifera*, respectively, were reported to be positive and above the EU safety and legal levels for at least one organochlorine with heptaclor accounting for almost 44% of the samples detected (Ruiz-Toledo *et al*., 2018) [13]. Other study reported that 56% (Balayiannis and Balayiannis, 2008; Pohorecka *et al*., 2012) [49, 50], 60 % (Mullin *et al*., 2010) [7], and 94% (Panseri *et al*., 2014) [51] of their honey samples contain at least two organophosphorous insecticides. About 0.013, 0.012 and 0.005 mg/kg of deltamethrin, chlorpyrifos and endosulfan residues were detected in raw honey samples from Pakistan (Farooqi *et al*., 2015) [52]. Endosulfan, chlorpyrifos, and cypermethrin with an average mean concentration of 0.01-0.092 mg/kg above the MRLs were detected in honey samples from Brazil (Fell and Cobb, 2009) [53]. Presence of pesticide residues in honey samples and bee products were also reported by several researchers (Bermejo *et al*., 2010; Ivana *et al*., 2010; Johnson *et al*., 2010; Peres *et al*., 2010) [54-57]. Orso *et al*., (2014) [58] using Modifed QuEChERS Method detected 0.03 mg kg^{-1}, 0.03 mg kg^{-1}, and 0.09 mg kg^{-1} of chlorpyrifos ethyl, chlorothalonil and malathion in three honey samples from Branzil. The concentration of the pesticides were observed to be above the MRLs allowed by the Brazilian and European legislations. Pesticide residues following various incidences in percentage were identified in 32 honey samples from different geographic regions of Colombia. In the study, chlorpyrifos, profenofos, DDT, HCB and fenitrothion were detected with 36.1%, 16.4%, 6.6%, 4.9%, and 1.6% incidence respectively with just 4.9% of the samples exceeded the MRL (López *et al*., 2014) [59].

The most probable explanation to high level of the pesticides residues in the honey samples recorded in this study could be from the bioaccumalation processes in the plants. The pesticides residues bioaccumulated in flowering plants, and through possible foraging activities by the bees on the contaminated pollens and nectars could transfer same into the honey product. According to Panseri *et al.*, (2014) [51]. organochlorines pesticides in contaminated soils may possibly enter into the human food web through non-fatty food products like honey. The lipophilic nature of organochlorine pesticides make it readily soluble and stable in beeswax, in this form could be easily made available in the honey (Blasco *et al*., 2003) [21]. The concentration of the pesticides residue in sample from Mubi were higher probable due to the differences in population, farming activities and locations. Hong municipal area is largely rural and most of the bees farming activities are relatively far from farmlands. However, Mubi is a densely populated area compared to Hong, a community heightened by commercial activities, with the biggest cattle market in West Africa (Bwatanglang *et al*., 2019) [60]. The proximity of the beehive farms to agricultural farmlands also contributes to the likelihood of the bees foraging on pesticides contaminated pollen and nectar from the nearby crops and plants (De Oliveira *et al*., 2016) [61]. It was estimated that 10000–25,000 honey bee workers make 10 round trips to cover an area up to 10 km^2 around the colony during foraging (Kumar *et al*., 2018; Sherry and Mitchell 2007) [48, 62], increasing the likelihood of carrying pesticides from the pollen and nectar to the honey product (Krupke *et al*., 2012) [63]. Studies shows that the concentration, and types of pesticides in honey bee colonies and products are observed to be positively correlated to the distance from the contaminated source and the duration of the exposure (De Oliveira *et al*., 2016, Chauzat *et al*., 2011; Chauzat *et al*., 2009; Malhat *et al*., 2015) [61, 64-66].

Besides the above reasons, Mubi being a community striving with commercial activities, will experience inflow of honey from different geographical locations in to the market increasing the level of contaminated honey in circulation. Thus, the high concentration of the pesticides residue in the CHS implies possible adulteration or rather emanating from the mixture of different honey samples from different location during the processing. This implies higher possible health risk to honey consumers from Mubi compared to consumers from Hong.

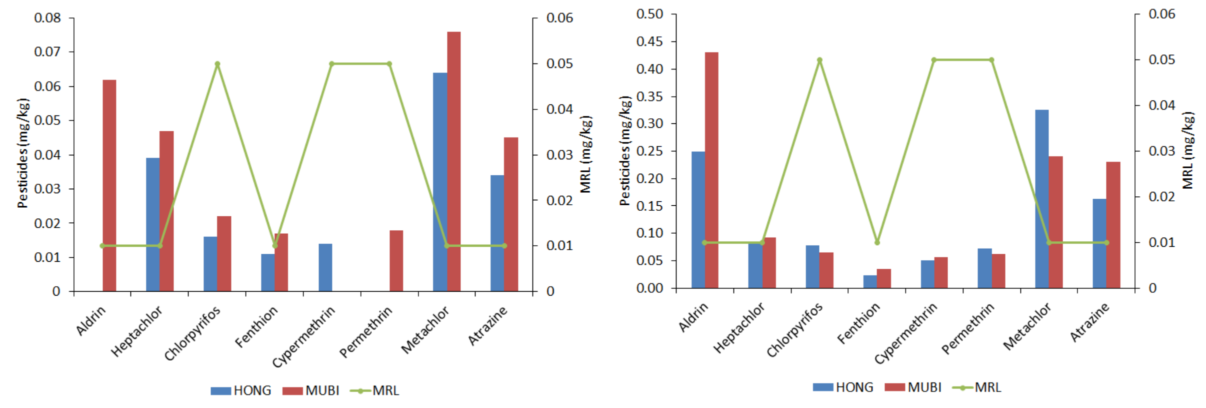

Fig 1: Showing the concentration of the pesticides in (a) Raw honey sample and (b) Commercial honey samples from Hong and Mubi municipal area. The result are presented as the mean ±SD of three triplicate analysis.

Estimated daily intakes (EDIs) of pesticides through the consumption of honey from Hong and Mubi

Though, figure 1 above might have projected a picture indicating a likely health risk for consumer in the study locations, estimating the average daily intake (EDI) of the pesticides through the consumption of the honey with the acceptable daily intake (ADI) values will bring into light a better quantifiable level of concern. The EDI of the pesticides through the consumption of honey from Hong and Mubi are summarized in Table 1. From the analysis, exposure to the pesticides from the honey samples from Mubi showed the highest contributions to dietary exposure. Similarly, dietary exposure to the CHS were significantly ($p<0.05$) higher to the exposure level from the RHS in both locations and highly significant ($p<0.05$) in samples from Mubi compared to samples from Hong. In general, dietary intakes of aldrin, metachlor, and atrazine were observed to be the major contributors to the exposure. The severity of the exposure were observed to be significant ($p<0.05$) in children compared to adults. The highest EDI for the RHS were observed through dietary exposure to metachlor by children in Hong (4.3E-06 mg/kg/bw) and Mubi (1.3E-06 mg/kg/bw). An EDI of 1.1E-05 mg/kg/bw, 1.7E-05 mg/kg/bw, and 2.2E-04 mg/kg/bw were recorded in children exposed to atrazine, aldrin, and metachlor respectively through the consumption of the CHS from Hong. These EDI were observed to be slightly lower than the EDI in children from Mubi. Even though the EDI for all the pesticides in Table 1 did not point toward a level of concern in comparison to their respective concentrations to the MRL in Fig.2. The dietary exposure to either the RHS or the CHS for the respective pesticides were observed to be far below the recommended ADIs to pose a health risk concerns (FAO/WHO, 2002, 2010; USEPA, 1996) [39-41]. Eissa *et al*., (2014) [12] detected organochlorine and Organophosphorus metabolites in 55.6% of the honey samples used in the study. Even though, 81.8% of the pesticides residue detected in the study falls above the MRL set by the European Union, the EDI calculated for each pesticides in the honey were observed to be much lower than acceptable daily intakes (ADIs).

Table 1: The estimated daily intakes (EDIs) and acceptable daily intake (ADI) of pesticide residues found in honey from Hong and Mubi

	Raw				Commercial				
	Hong		Mubi		Hong		Mubi		
	Adults	Children	Adults	Children	Adults	Children	Adults	Children	ADIs
Aldrin	ND	ND	1.0E-06	4.1E-06	4.2E-06	1.7E-05	7.2E-06	2.9E-05	1.0E-04
Heptachlor	6.5E-07	2.6E-06	7.8E-07	3.1E-06	1.4E-06	5.7E-06	1.6E-06	6.2E-06	1.0E-04
Chlorpyrifos	2.7E-07	1.1E-06	3.7E-07	1.5E-06	1.3E-06	5.2E-06	1.1E-06	4.3E-06	1.0E-02
Fenthion	1.8E-07	7.3E-07	2.8E-07	1.1E-06	3.8E-07	1.5E-06	5.8E-07	2.3E-06	1.0E-03
Cypermethrin	2.3E-07	9.3E-07	ND	ND	8.3E-07	3.3E-06	9.5E-07	3.8E-06	5.0E-02
Permethrin	ND	ND	3.0E-07	1.2E-06	1.2E-06	4.8E-06	1.0E-06	4.1E-06	5.0E-02
Metachlor	1.1E-06	4.3E-06	1.3E-06	5.1E-06	5.4E-06	2.2E-05	4.0E-06	1.6E-05	5.0E-03
Atrazine	5.7E-07	2.3E-06	7.5E-07	3.0E-06	2.7E-06	1.1E-05	3.8E-06	1.5E-05	3.5E-02

The EDI and ADI are expressed in mg/kg/bw. ND signified Not-detected.

Risk characterization of pesticides through consumption of honey

The EDI calculated for each pesticides were further appraised for potential carcinogenic and non-carcinogenic related health risk (HR). The non-carcinogenic risk were evaluated by integrating the EDI with the ADI for the estimation of the target hazard quotient (THQ) and the health index (HI). The results are shown in Table 2. From the HR analysis, both the THQ and the HI for the pesticides in the RHS from Hong were all less than (<1) one for both each categories. This implies that consumption of RHS from Hong pose no health risk to the consumers. Similar trend were also observed in the RHS from Mubi for the adults populations. Furthermore, from the table, the exposure to the pesticides through the consumption of the CHS however higher than the THQ values recorded for the RHS were also observed not to pose any HR. A THQ <1 were also observed for both age categories consuming CHS from Hong and Mubi. When compared between the two locations, the probability for non-carcinogenic risk is much higher for sample from Mubi compared to Hong. The results indicate that aldrin, and heptachlor are of particular concern because of their high THQ and HI levels in the. Similar trend were observed in honey sample from Egypt. In the study, the following pesticides, diazinon, dicrotophos, profenofos, and chlorpyrifos with mean concentrations of 0.3, 0.34, 0.28, and

3.3 mg kg^1 respectively were detected (Al Naggar *et al*. 2015) [67]. Based on the worst-case scenario, exposure to these pesticides in the honey were observed to be 15-fold less than the HI value of 1.0 for adverse effects on humans [Al Naggar *et al*., 2017) [19].

Table 2: The target hazard quotient (THQ) and health index (HI) for non-carcinogenic risk from the pesticide residues found in honey from Hong and Mubi

	Raw				Commercial			
	Hong		Mubi		Hong		Mubi	
	Adults	Children	Adults	Children	Adults	Children	Adults	Children
Aldrin	ND	ND	1.0E-02	4.1E-02	4.2E-02	1.7E-01	7.2E-02	2.9E-01
Heptachlor	6.5E-03	2.6E-02	7.8E-03	3.1E-02	1.4E-02	5.7E-02	1.6E-02	6.2E-02
Chlorpyrifos	2.7E-05	1.1E-04	3.7E-05	1.5E-04	1.3E-04	5.2E-04	1.1E-04	4.3E-04
Fenthion	1.8E-04	7.3E-04	2.8E-04	1.1E-03	3.8E-04	1.5E-03	5.8E-04	2.3E-03
Cypermethrin	4.7E-06	1.9E-05	ND	ND	1.7E-05	6.7E-05	1.9E-05	7.6E-05
Permethrin	ND	ND	6.0E-06	2.4E-05	2.4E-05	9.6E-05	2.1E-05	8.3E-05
Metachlor	2.1E-04	8.5E-04	2.5E-04	1.0E-03	1.1E-03	4.3E-03	8.0E-04	3.2E-03
Atrazine	1.6E-05	6.5E-05	2.1E-05	8.6E-05	7.8E-05	3.1E-04	1.1E-04	4.4E-04
HI	6.9E-03	2.8E-02	1.9E-02	7.5E-02	5.8E-02	2.3E-01	8.9E-02	3.6E-01

ND signified Not-detected

Conclusion

The finding from this study show the concentrations of the pesticides to be significantly ($p<0.05$) higher in the CHS when compared to the RHS. Higher concentrations of aldrin, metachlor, and atrazine were observed to be above their MRL in the CHS from Mubi. The health risk index assessed for all the pesticides (both RHS and CHS) from Hong and Mubi were all less than (<1) one for all age categories. When compared between the two locations, the probability for non-carcinogenic risk is much higher for samples from Mubi compared to Hong and much higher for children than adults. The results indicate that aldrin, and heptachlor are of particular concern because of their high THQ and HI levels in the CHS. From the assessments conducted, it will suffice to say that continual monitoring of the residual level of pesticides and other agrochemicals in food for human consumption is not out of place.

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