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## Evaluating the potential health risk of organochlorine pesticides in selected protein foods from Abeokuta southwestern Nigeria

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### ABSTRACT

This study evaluated the potential health risk of OCPs in selected protein foods. A total of 66 food samples were collected from selected areas in Abeokuta, southwestern Nigeria. Food samples were analyzed for OCPs using the standard method. OCP data were analysed for descriptive and inferential statistics. Health risk assessment was conducted for non-carcinogenic {Hazard Quotient (HQ), Hazard Index (HI) and Cancer Risk (CR)}. The  $\Sigma$ OCP concentration was observed highest in pork ( $42.20 \pm 33.33 \text{ mg kg}^{-1}$ ) of which methoxychlor and dieldrin represented 63 and 28%, respectively. The HIs of OCPs in protein foods were generally greater than 1.0 indicating adverse effects. The  $\Sigma$ CR data showed carcinogenic effects in protein foods with values greater than the acceptable limit of  $1.0 \times 10^{-4}$ .

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Organochlorine pesticides; health effects; cancer; protein foods; risk assessment

### 1. Introduction

Organochlorine pesticides (OCPs) are persistent organic pollutants (POPs) classified as organic disruptors, because of their ability to mimic thyroid hormones when they enter the human body [1]. OCPs consist three major classes such as dichlorodiphenylethanes (dichlorodiphenyltrichloroethane DDT, dichlorodiphenyldichloroethylene DDE and dichlorodiphenyldichloroethane DDD), cyclodienes (aldrin, dieldrin, endrin, chlordane, endosulfan and heptachlor) and cyclohexane (hexachlorocyclohexane HCH). OCPs have been applied to eradicate pests and vector of diseases [2]. For example, DDT has been used to control lice (vector of typhus) and mosquitoes (vector of malaria), while endrin has been utilized as insecticide, rodenticide and avicide [2,3]. This has invariably led to increased agricultural yields and protection of public health [2]. The application of DDT for malaria reduction program was reported to save more than five million lives [4]. However, the use of OCPs has resulted to environmental pollution of air, water and soil/sediments [2].

The environmental and health concerns of OCPs can be attributed to peculiar characteristics such as persistence (slow rate of degradation) high lipophilicity, bioaccumulation in biota and potential of long range transport [5–7]. DDT is moderately hazardous having high persistence and a half-life of between 2 and 15 years [8]. DDE, which is the metabolite of DDT has also been adopted as

a biomarker for DDT exposure in many epidemiological studies [9–11]. Many OCPs are carcinogenic, neurotoxic and immunotoxic [7,12]. They have also been implicated in a number of health problems such as low sperm count, birth defects, reproductive malfunctions and deformities, Parkinson's disease [13–15], endocrine disruption, and developmental disorders [16–18].

Although OCPs have been banned, but are still in use in many countries including Cote D'Ivoire, Ghana, India, Iran, Mexico, Nigeria and parts of China [7,19,20]. This might be linked to factors including effectiveness of the chemicals to control pests at relatively low cost, lack of appropriate regulatory control in management of production and use of OCPs, and laxity in environmental laws [7]. Consequently, considerable amounts of OCPs have been observed in sediments, soil, water, air, plants, animal tissues [21–24] and human samples (blood, adipose tissue and breast milk) [25]. It is, therefore, necessary to monitor and assess the risk of OCPs in environmental samples in order to protect the consumers' health. Human health risk evaluation involves the calculation of the upper limit in excess of lifetime cancer risk and non-carcinogenic hazard of a receptor [26]. This is based on the assumption that an adult might be exposed to chemical substances (e.g. metals, polycyclic aromatic hydrocarbons, OCPs) through ingestion of food. Protein

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foods (beef, cow skin, fish, crab, crayfish, goat meat, pork and snails) are important sources of vitamins and minerals recommended for good health [27,28]. Sea foods are dietary sources of  $\omega$ -3 polyunsaturated fatty acids (PUFA), for which there is increasing evidence of positive effects on fetal growth and length of gestation period of infants [29]. Fish is rich in lysine suitable for supplementing high carbohydrate food [28]. Crab contains eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA), which are important for preventing atherosclerosis, rheumatoid arthritis, dementia, Alzheimer and cardiovascular diseases [30]. There is a dearth of knowledge on the levels and associated risk of OCPs in protein foods [7,31,32]. The human health risk of OCPs in foodstuffs is yet to be reported in Abeokuta, the capital of Ogun state. It is the most populous city in the state in which the main occupation of the residents is farming. The possibility of past and present applications of OCPs for agricultural activities therefore may not be ruled out. This study seeks to bridge this research gap by evaluating the concentrations and health risk of OCPs in different protein foods from Abeokuta, southwestern Nigeria.

## 2. Materials and methods

### 2.1 Sample collection and preservation

A total of 66 protein food samples were purchased from Abeokuta in southwestern Nigeria between October and November 2019. The protein food samples were crabs, (6 samples), crayfish (12 samples), *Clarias gariepinus*, catfish (6 samples), *Tilapia zillii*, tilapia fish (12 samples), beef (6 samples), cow skin (6 samples), goat meat (6 samples), pork (6 samples) and snail (6 samples). A 0.25 kg of each foodstuff (muscle) was purchased from popular markets in Abeokuta, southwestern Nigeria. The samples were wrapped in aluminium foils (to avoid contamination) and placed inside a chest packed with ice cubes [33]. The samples were transported to the CTX-ION Analytics Ltd. Laboratory, Lagos, Nigeria for extraction and determination of OCPs.

### 2.2 Determination of organochlorine pesticides by GC-microECD

About 15 g tissue (muscle) samples of protein foodstuffs were macerated thoroughly with Teflon pestle homogenizer alongside 10 g anhydrous sodium sulphate [34]. A 2.0 g of each sample was weighed and transferred to a cellulose extraction

thimble and placed into a Soxhlet apparatus. Each sample was then extracted with 100 mL hexane for approximately 10 hours.

Sample clean up was done using a glass column packed with 4.0 g silica gel (previously activated for 6 hours at 130 °C in a petri dish, and loosely covered with foil). Exactly 2.0 g of anhydrous sodium sulphate was added to the column after which 10 mL hexane was introduced to the top of the column to wet and rinse the sodium sulphate and silica gel. The sample extract was transferred onto the column and eluted with 20 mL hexane. The extract was collected in a beaker and concentrated down to 2 mL using a rotary evaporator. The extract was analysed for 14 OCPs (aldrin, dieldrin, endosulfan I, endosulfan II, endosulfan sulphate, endrin, endrin aldehyde, endrin ketone, heptachlor, heptachlor epoxide, methoxychlor, p,p'-DDD, p,p'-DDE and p,p'-DDT) using Gas Chromatograph equipped with microElectron Capture Detector ( $\mu$ ECD) (Agilent 7890B, Santa Clara, CA, USA). The GC- $\mu$ ECD was fitted with a HP-5 capillary column coated with 5% Phenyl Methyl Siloxane (30 m length x 0.32 mm diameter x 0.25  $\mu$ m film thickness) (Agilent Technologies). A 1.0  $\mu$ L of the samples were injected in a splitless mode at an injection temperature of 300°C, a pressure of 15 PSI and a total flow of 21 mL min<sup>-1</sup>. Helium was used as the carrier gas. The purge flow to split vent was set at 15 mL min<sup>-1</sup> at 0.75 min. Oven was initially programmed at 100°C (1 min), ramped at 15°C/min to 120°C (0 min) and then to 230°C at 15°C min<sup>-1</sup>. The  $\mu$ ECD temperature was set at 300°C with nitrogen used as makeup gas (flow of 60 mL min<sup>-1</sup>).

Quantification of OCPs involved external calibration curves prepared from the standard solution of each of the OCPs. The OCP standard, 1000 mg L<sup>-1</sup> containing 14 congeners was obtained from AccuStandard (New Haven, CT, USA).

During the laboratory analysis, the analytical procedures observed include analysis of blank samples to cancel the matrix effects of extracting reagents and to calculate the limit of detection of the instrument as 3 x standard deviation [33]. All solvents used for extraction were of GC grade (Merck KGaA, Darmstadt, Germany).

### 2.3 Data analysis

Organochlorine pesticides data were subjected to simple descriptive (mean and standard deviation) and inferential (Duncan Multiple Range Test) statistics using SPSS for Windows (version 22.0).

**Table 1.** OCP levels in aquatic and terrestrial protein foods.

	Protein foods	N	mg kg <sup>-1</sup> (wet weight)			
			Mean	Std. Deviation	Minimum	Maximum
Aldrin	<i>Clarias gariepinus</i>	6	<0.01a		<0.01	<0.01
	<i>Tilapia zillii</i>	12	<0.01a		<0.01	<0.01
	Crayfish	12	0.21a	0.72	<0.01	2.5
	Crab	6	<0.01a		<0.01	<0.01
Dieldrin	<i>Clarias gariepinus</i>	6	0.46a	0.10	0.35	0.6
	<i>Tilapia zillii</i>	12	1.48a	2.53	<0.01	7.61
	Crayfish	12	0.92a	1.92	<0.01	6.59
	Crab	6	1.15a	1.88	0.13	4.96
Endosulfan I	<i>Clarias gariepinus</i>	6	0.05a	0.01	0.05	0.07
	<i>Tilapia zillii</i>	12	0.08a	0.03	<0.01	0.13
	Crayfish	12	0.12a	0.09	<0.01	0.22
	Crab	6	0.07a	<0.01	0.05	0.09
Endosulfan II	<i>Clarias gariepinus</i>	6	0.17a	0.06	0.07	0.22
	<i>Tilapia zillii</i>	12	0.10a	0.13	<0.01	0.4
	Crayfish	12	0.28a	0.79	<0.01	2.77
	Crab	6	0.46a	0.77	0.06	2.03
Endosulfan Sulphate	<i>Clarias gariepinus</i>	6	1.80 c	0.30	1.45	2.26
	<i>Tilapia zillii</i>	12	0.21a	0.16	<0.01	0.47
	Crayfish	12	0.15a	0.14	<0.01	0.36
	Crab	6	1.01b	0.98	0.08	2.37
Endrin	<i>Clarias gariepinus</i>	6	0.02a	0.03	<0.01	0.08
	<i>Tilapia zillii</i>	12	0.17ab	0.28	<0.01	0.99
	Crayfish	12	0.14ab	0.17	<0.01	0.52
	Crab	6	0.06ab	0.06	<0.01	0.12
Endrin Aldehyde	<i>Clarias gariepinus</i>	6	0.11a	0.04	0.07	0.19
	<i>Tilapia zillii</i>	12	0.18a	0.14	0.04	0.56
	Crayfish	12	0.28a	0.57	<0.01	2.04
	Crab	6	1.16b	2.10	0.05	5.42
Endrin Ketone	<i>Clarias gariepinus</i>	6	0.80ab	1.31	0.06	3.42
	<i>Tilapia zillii</i>	12	0.32a	0.18	0.09	0.78
	Crayfish	12	0.39a	0.69	<0.01	2.47
	Crab	6	0.21a	0.21	<0.01	0.52
Heptachlor	<i>Clarias gariepinus</i>	6	0.06a	0.12	<0.01	0.31
	<i>Tilapia zillii</i>	12	0.38a	0.79	<0.01	2.31
	Crayfish	12	0.39a	0.82	<0.01	2.83
	Crab	6	0.06a	0.14	<0.01	0.34
Heptachlor Epoxide	<i>Clarias gariepinus</i>	6	<0.01a		<0.01	<0.01
	<i>Tilapia zillii</i>	12	0.06a	0.09	<0.01	0.29
	Crayfish	12	0.33a	0.87	<0.01	3.06
	Crab	6	0.17a	0.15	<0.01	0.41
Methoxychlor	<i>Clarias gariepinus</i>	6	<0.01a		<0.01	<0.01
	<i>Tilapia zillii</i>	12	0.57a	1.78	<0.01	6.2
	Crayfish	12	2.19ab	3.57	<0.01	9.35
	Crab	6	4.57ab	9.76	<0.01	24.36
p,p'-DDD	<i>Clarias gariepinus</i>	6	<0.01a	0.01	<0.01	0.02
	<i>Tilapia zillii</i>	12	<0.01a		<0.01	<0.01
	Crayfish	12	0.20a	0.69	<0.01	2.38
	Crab	6	0.02a	0.01	<0.01	0.03
p,p'-DDE	<i>Clarias gariepinus</i>	6	0.29a	0.21	0.14	0.70
	<i>Tilapia zillii</i>	12	0.06a	0.06	<0.01	0.16
	Crayfish	12	0.67a	1.29	<0.01	3.85
	Crab	6	0.48a	0.57	0.07	1.57
p,p'-DDT	<i>Clarias gariepinus</i>	6	<0.01a		<0.01	<0.01
	<i>Tilapia zillii</i>	12	0.06a	0.1	<0.01	0.25
	Crayfish	12	0.31a	0.7	<0.01	2.5
	Crab	6	0.14a	0.15	<0.01	0.29
Aldrin	Beef	6	<0.01a		<0.01	<0.01
	Cowskin	6	<0.01a		<0.01	<0.01
	Goat meat	6	<0.01a		<0.01	<0.01
	Pork	6	<0.01a		<0.01	<0.01
	Snail	6	<0.01a		<0.01	<0.01
Dieldrin	Beef	6	1.92b	2.4	0.22	5.12
	Cowskin	6	0.03a	0.05	<0.01	0.09
	Goat meat	6	1.10b	1.88	0.19	4.93
	Pork	6	11.69 c	14.74	<0.01	40.39
	Snail	6	0.08a	0.09	<0.01	0.18
Endosulfan I	Beef	6	0.12a	0.07	0.05	0.23
	Cowskin	6	0.08a	0.03	0.04	0.12
	Goat meat	6	0.08a	0.02	0.05	0.11
	Pork	6	0.10a	0.05	<0.01	0.13
	Snail	6	0.08a	0.08	<0.01	0.21
Endosulfan II	Beef	6	0.14a	0.08	0.05	0.25
	Cowskin	6	0.02a	0.03	<0.01	0.08
	Goat meat	6	0.19a	0.08	0.11	0.32
	Pork	6	1.19b	0.88	<0.01	2.71
	Snail	6	0.13a	0.29	<0.01	0.73

(Continued)

Table 1. (Continued).

	Protein foods	N	mg kg <sup>-1</sup> (wet weight)			
			Mean	Std. Deviation	Minimum	Maximum
Endosulfan Sulphate	Beef	6	0.31a	0.19	0.13	0.65
	Cowskin	6	1.40 c	0.62	0.56	2.12
	Goat meat	6	0.56ab	0.57	0.09	1.46
	Pork	6	1.14bc	1.03	<0.01	2.34
	Snail	6	0.05a	0.05	<0.01	0.12
Endrin	Beef	6	0.25 c	0.18	0.06	0.51
	Cowskin	6	0.03a	0.05	0.01	0.13
	Goat meat	6	<0.01a		<0.01	<0.01
	Pork	6	0.18b	0.26	<0.01	0.68
	Snail	6	<0.01a		<0.01	<0.01
Endrin Aldehyde	Beef	6	0.38b	0.61	0.08	1.63
	Cowskin	6	0.79b	1.11	0.07	2.96
	Goat meat	6	0.03a	0.04	<0.01	0.11
	Pork	6	0.15a	0.11	<0.01	0.33
	Snail	6	0.04a	0.05	<0.01	0.14
Endrin Ketone	Beef	6	0.58ab	1.05	0.09	2.71
	Cowskin	6	1.37b	1.89	0.12	5.01
	Goat meat	6	0.09a	0.01	0.07	0.1
	Pork	6	0.19a	0.12	<0.01	0.35
	Snail	6	0.06a	0.07	<0.01	0.18
Heptachlor	Beef	6	0.37a	0.45	<0.01	0.94
	Cowskin	6	<0.01a		<0.01	0.01
	Goat meat	6	<0.01a		<0.01	0.01
	Pork	6	0.37a	0.19	<0.01	0.49
	Snail	6	<0.01a		<0.01	0.01
Heptachlor Epoxide	Beef	6	0.23a	0.14	<0.01	0.4
	Cowskin	6	<0.01a		<0.01	0.01
	Goat meat	6	<0.01a		<0.01	0.01
	Pork	6	0.18a	0.11	<0.01	0.31
	Snail	6	0.07a	0.1	<0.01	0.2
Methoxychlor	Beef	6	5.46a	6.92	0.09	17.62
	Cowskin	6	0.03a	0.03	<0.01	0.06
	Goat meat	6	0.52a	0.9	<0.01	2.19
	Pork	6	26.38b	18.63	<0.01	56.75
	Snail	6	0.02a	0.03	<0.01	0.07
p,p'-DDD	Beef	6	0.03a	0.06	<0.01	0.15
	Cowskin	6	<0.01a		<0.01	<0.01
	Goat meat	6	0.04a	0.03	<0.01	0.09
	Pork	6	0.03a	0.03	<0.01	0.08
	Snail	6	<0.01a		<0.01	<0.01
p,p'-DDE	Beef	6	0.24a	0.13	0.05	0.41
	Cowskin	6	0.21a	0.34	<0.01	0.87
	Goat meat	6	0.08a	0.05	<0.01	0.14
	Pork	6	0.61a	0.3	<0.01	0.81
	Snail	6	0.13a	0.07	0.06	0.26
p,p'-DDT	Beef	6	0.31a	0.14	0.15	0.53
	Cowskin	6	<0.01a		<0.01	0.01
	Goat meat	6	0.09a	0.2	<0.01	0.49
	Pork	6	<0.01a		<0.01	<0.01
	Snail	6	0.09a	0.21	<0.01	0.51

Similar letters along the rows are not statistically different at  $p > 0.05$  according to Duncan Multiple Range Test

## 2.4 Health risk assessment

The health risk assessment of OCPs was calculated for Average Daily Dose (ADD), Hazard Quotient (HQ), Hazard Index (HI) and Cancer Risk (CR) using the formula highlighted by USEPA [35–37].

$$ADD = \frac{C \times IR \times EF \times ED}{BW \times AT} \quad (1)$$

where ADD is an average daily dose ( $\text{mg kg}^{-1} \text{ day}^{-1}$ ), C is the concentration of OCPs determined in protein foods ( $\text{mg kg}^{-1}$ ), IR is the ingestion rate of protein foods ( $24.7 \text{ g day}^{-1}$ ) [38], ED is exposure duration (years) = 30 years for carcinogenic effects for adults, EF is exposure frequency ( $\text{day/year}$ ) = 350  $\text{days year}^{-1}$  [33], AT is averaging time or life expectancy = 54.5 years [28,39], AT is equal to ED for non-

carcinogenic effects, BW is body weight ( $\text{kg}$ ) = 60kg for an adult [7,28,33].

$$HQ = \frac{ADD}{RfD} \quad HI = \sum_{i=1}^n HQ_i = 1 \dots n \quad (2)$$

where ADD is an average daily dose ( $\text{mg kg}^{-1} \text{ day}^{-1}$ ), RfD is the reference dose ( $\text{mg kg}^{-1} \text{ day}^{-1}$ ) [35], and n is the number of observed elements.

HQ > 1 denotes adverse health effects; HQ < 1 indicates no adverse effects,

$$\text{Cancer Risk} = ADD \times SF \quad (3)$$

where ADD is average daily dose ( $\text{mg kg}^{-1} \text{ day}^{-1}$ ), and SF is slope factor ( $\text{mg}^{-1} \text{ kg day}$ )

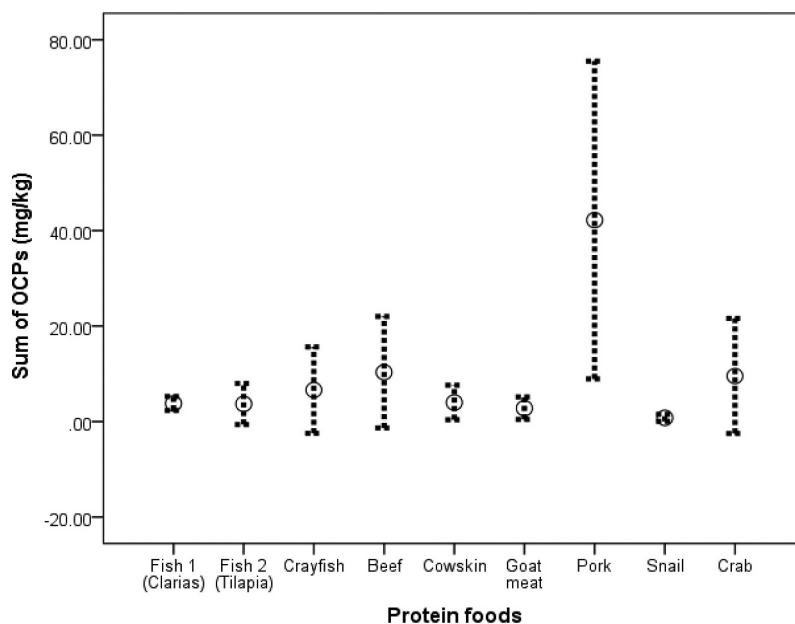


Figure 1. The sum of OCPs in protein foods.

### 3. Results and discussion

#### 3.1 Concentrations of OCPs in protein foods

The fractions of individual OCP measured in protein food samples are presented in Figure S1 (in the supplementary information). Aldrin was detected only in crayfish, representing only 2% of the total number of food samples, while in terrestrial food samples, aldrin was observed below the detection limit ( $<0.01 \text{ mg kg}^{-1}$ ). The prominent OCPs determined in food samples were endrin ketone (89%), endosulfan I (88%), endosulfan sulphate/endrin aldehyde (82%), p,p'-DDE (79%), dieldrin (77%) and endosulfan II (70%). The present data showed that endrin, endosulfan and dieldrin are still widely used by many farmers in Africa, similar to the reports from the past studies [31,40]. These pesticides could remain in the environment for a long period of time and also possessed the potential to bioaccumulate in plants and animals [41].

Tables 1 present the levels of OCPs in aquatic and terrestrial protein foods. Methoxychlor was the highest measured OCP in both aquatic and terrestrial protein food samples. In aquatic food samples (Table 1), the highest level of methoxychlor was measured in crab ( $4.57 \pm 9.76 \text{ mg kg}^{-1}$ , wet weight); while for terrestrial tissues (Table 1), methoxychlor was observed highest in pork ( $26.38 \pm 18.63 \text{ mg kg}^{-1}$ , wet weight). The high level of methoxychlor observed in pork might be linked to high-fat content [42] and recent application of the pesticide [43]. The use of methoxychlor is seasonal and expected to be higher during the period of insect

control on animals [42,43]. The concentrations of endosulfan sulphate, dieldrin and endrin aldehyde were generally greater than  $1.0 \text{ mg kg}^{-1}$  in crab. In the terrestrial protein foods, the OCPs having the average values greater than  $1.0 \text{ mg kg}^{-1}$  were dieldrin in beef, goat meat and pork, endosulfan II in pork, endosulfan sulphate and methoxychlor in cowskin and pork, and aldrin ketone in cowskin. Furthermore, dieldrin and endosulfan sulphate revealed levels higher than  $1.0 \text{ mg kg}^{-1}$  in *Tilapia zillii* and *Clarias gariepinus*. The dieldrin value in *Clarias gariepinus* ( $0.46 \pm 0.10 \text{ mg kg}^{-1}$ ) was similar to those previous reported by Ezemonye *et al.* [44] in catfish samples ( $0.38 \pm 1.10$ – $0.53 \pm 0.90 \text{ mg kg}^{-1}$ ) collected from three rivers in Edo State, Nigeria. The study, however, documented lower concentrations of dieldrin in *Tilapia zillii*.

There were no significant variations ( $p > 0.05$ ) in the concentrations of most OCPs measured in aquatic protein foods. The exceptions were endosulfan sulphate whose level was statistically ( $p < 0.05$ ) observed highest in *Clarias gariepinus* ( $1.8 \pm 0.30 \text{ mg kg}^{-1}$ ) and endrin aldehyde ( $1.16 \pm 2.10 \text{ mg kg}^{-1}$ ) in crab. The United States Federal Drug Agency permissible limit for heptachlor, aldrin, dieldrin, endrin, and methoxychlor in fish is  $0.3 \text{ mg kg}^{-1}$ , while for DDT, DDE, DDD and endosulfan, the value is  $5 \text{ mg kg}^{-1}$  [44]. This therefore established that the amounts of dieldrin measured in all the aquatic protein foods, endrin aldehyde in crab, endrin ketone in *Clarias gariepinus*, *Tilapia zillii* and crayfish, and heptachlor in *Tilapia zillii* and crayfish were higher than the



FDA [45] permissible limit of  $0.3 \text{ mg kg}^{-1}$ . The past study of Akan *et al.* [46] reported high dieldrin value of  $3.1 \text{ mg kg}^{-1}$  in fish samples from Lake Chad. Other OCP congeners in protein foods showed values lower than the FDA permissible limits indicating safe consumption. The occurrence of dieldrin in terrestrial protein foods followed similar pattern like those of aquatic protein foods, where the highest significant ( $p < 0.05$ ) amount was observed in pork. Furthermore, the concentrations of dieldrin in beef, goat meat and pork, endosulfan II in pork, endosulfan sulphate in beef, cow skin, goat meat and pork, endrin aldehyde in beef and cow skin, and heptachlor in beef and pork were also higher than the permissible standard of  $0.3 \text{ mg kg}^{-1}$  [45].

Methoxychlor value in goat meat was slightly higher than the permissible limit of  $0.3 \text{ mg kg}^{-1}$ , while in meat and pork, the concentrations were more than 18 and 88 times higher than the acceptable limit, respectively. The OCP of major concern in the terrestrial protein foods is methoxychlor having values higher than those reported for fish in past studies [46–48]. In the review work of Taiwo [7], DDT, aldrin and dieldrin were the OCPs of health concerns in fish samples collected from different locations in Africa.

The individual contribution to  $\Sigma$ OCPs in protein foods is shown in Figure S2. Methoxychlor constituted 46% of the total measured OCPs followed by dieldrin (23%), and endosulfan sulphate (7%). The distribution of OCPs in food samples followed the sequence of methoxychlor > dieldrin > endosulfan sulphate > endosulfan ketone > endrin aldehyde/DDE > endosulfan II > heptachlor/heptachlor epoxide > endosulfan I/DDD/DDT/endrin > aldrin.

Figure 1 shows the  $\Sigma$ OCP levels in different protein food. The sequence of distribution of OCPs in protein foods follows the decreasing order of pork > beef > crab > crayfish > cow skin > *Clarias gariepinus* > *Tilapia* > goat meat > snail. This study established pork as the most contaminated protein food, probably due to its high fat content, which has accumulated OCPs over a period of time. Snail is the least contaminated protein food having  $\Sigma$ OCP concentration less than  $1.0 \text{ mg kg}^{-1}$ , and thus signifying safest consumption.

### 3.2 Health risk assessment of OCPs in protein foods

Tables S1a and S1b show the estimated Average Daily Dose (ADD) values of OCPs in protein foods. Methoxychlor was the most dosed OCPs showing the highest concentrations in pork, beef and crab.

The ADDs of methoxychlor in these protein foods were generally higher than  $1.0 \text{ } \mu\text{g kg}^{-1} \text{ day}^{-1}$ . In contrast, the past study of Taiwo [7] revealed ADD value of  $\Sigma$ DDT greater than  $1.0 \text{ } \mu\text{g kg}^{-1} \text{ day}^{-1}$  in fish samples.

The non-carcinogenic adverse health effects of OCPs for Hazard Quotient (HQ) and Hazard Index (HI) in protein foods are presented in Tables 2. In aquatic foods (Table 2), the HQs of dieldrin, heptachlor, heptachlor epoxide (except in *Clarias gariepinus*), aldrin in crayfish, endrin ketone in *Clarias gariepinus*, and endrin aldehyde in crab were greater than the permissible limit of 1.0 indicating non-carcinogenic deleterious health effects through consumption of the protein foods. This was similar to the study of Barnhoorn *et al.* [31], which demonstrated the  $\text{HQ} > 1.0$  for dieldrin in fish samples collected from a polluted river in South Africa. A related study by Ezemonye *et al.* [44] reported  $\text{HQs} > 1.0$  for heptachlor epoxide and aldrin in *Clarias gariepinus* and *Tilapia zillii* taken from three rivers in Edo State. The study also revealed non-carcinogenic adverse effects for dieldrin and endrin in the *Clarias gariepinus* samples.

In terrestrial protein foods (Table 2), the HQs of dieldrin in beef, goat meat and pork, methoxychlor in pork, heptachlor and heptachlor epoxide in beef and pork, endrin ketone and endrin aldehyde in cow skin, and heptachlor epoxide in snail samples were also greater than 1.0. This suggests an adverse health risk on consumption of these protein foodstuffs. A previous study has shown the HQ value greater than 1.0 for heptachlor epoxide in beef samples from Benin City, Nigeria [32].

The HI, which is the sum of HQ for each constituent of OCP in the food samples revealed values greater than 1.0 thereby indicating unsafe consumption. The highest HI value was estimated for pork, while snail and cow skin had the lowest HIs. The pattern of HI of OCPs in protein foods followed the decreasing order of pork > beef > crayfish > tilapia fish > crab > goat meat > catfish > cow skin > snail.

Figure 2 presents the contributions of OCPs to non-carcinogenic adverse health effects. Dieldrin and heptachlor contributed mostly to the total HQ. It formed about 88% to the non-carcinogenic HI in goat meat and 42% in beef. The fractions constituted by dieldrin in *Clarias gariepinus*, *Tilapia zillii*, crayfish and crab were 48, 44, 21 and 48%, respectively. Similarly, the study of Barnhoorn *et al.* [31] has shown dieldrin as the major contributor to non-carcinogenic effects in fish samples. Also, heptachlor and heptachlor epoxide formed a considerable fraction of adverse health effects in beef with estimated HI values of 31 and 20%,

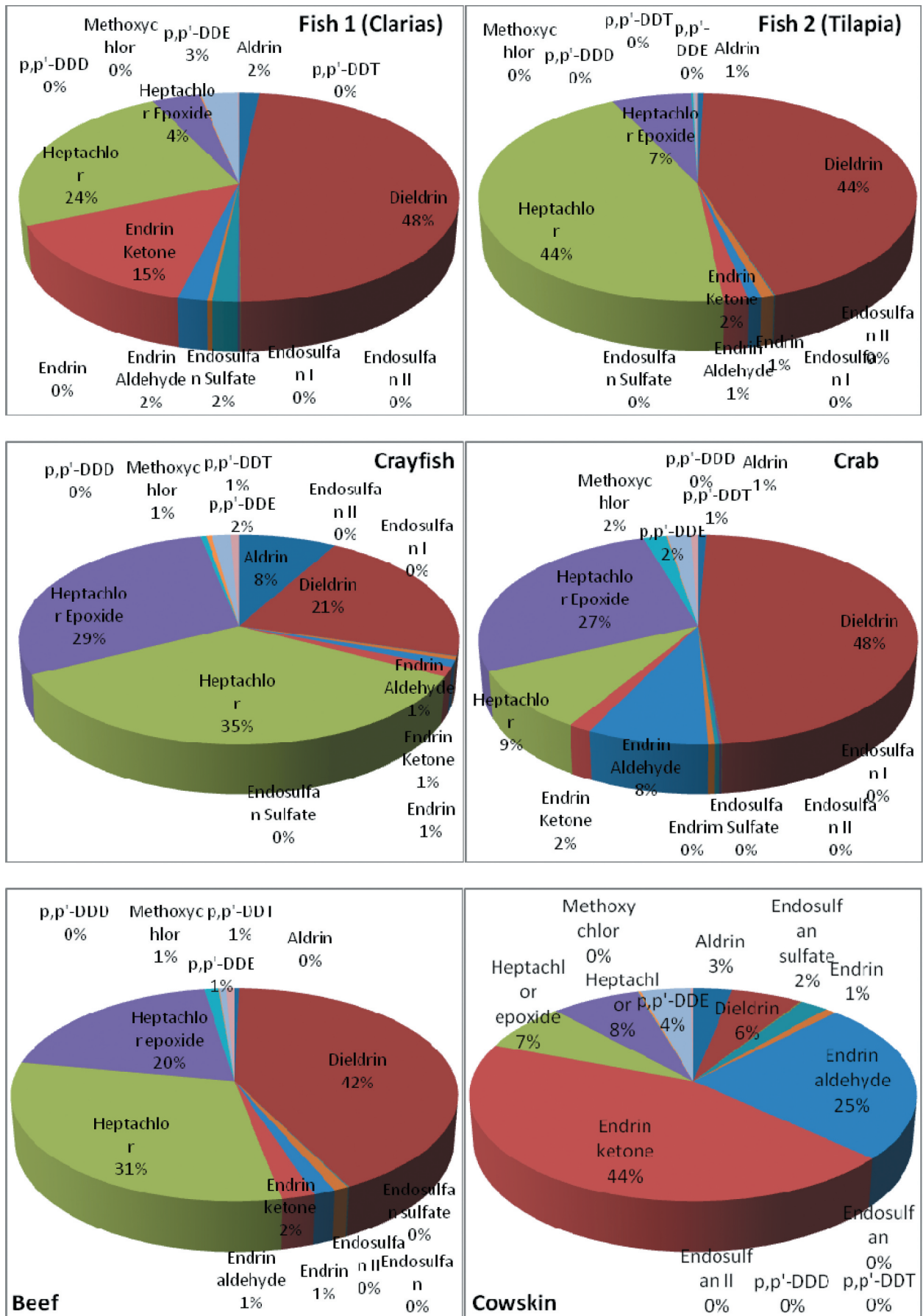


Figure 2. % Contribution to HI by OCPs in protein foods.



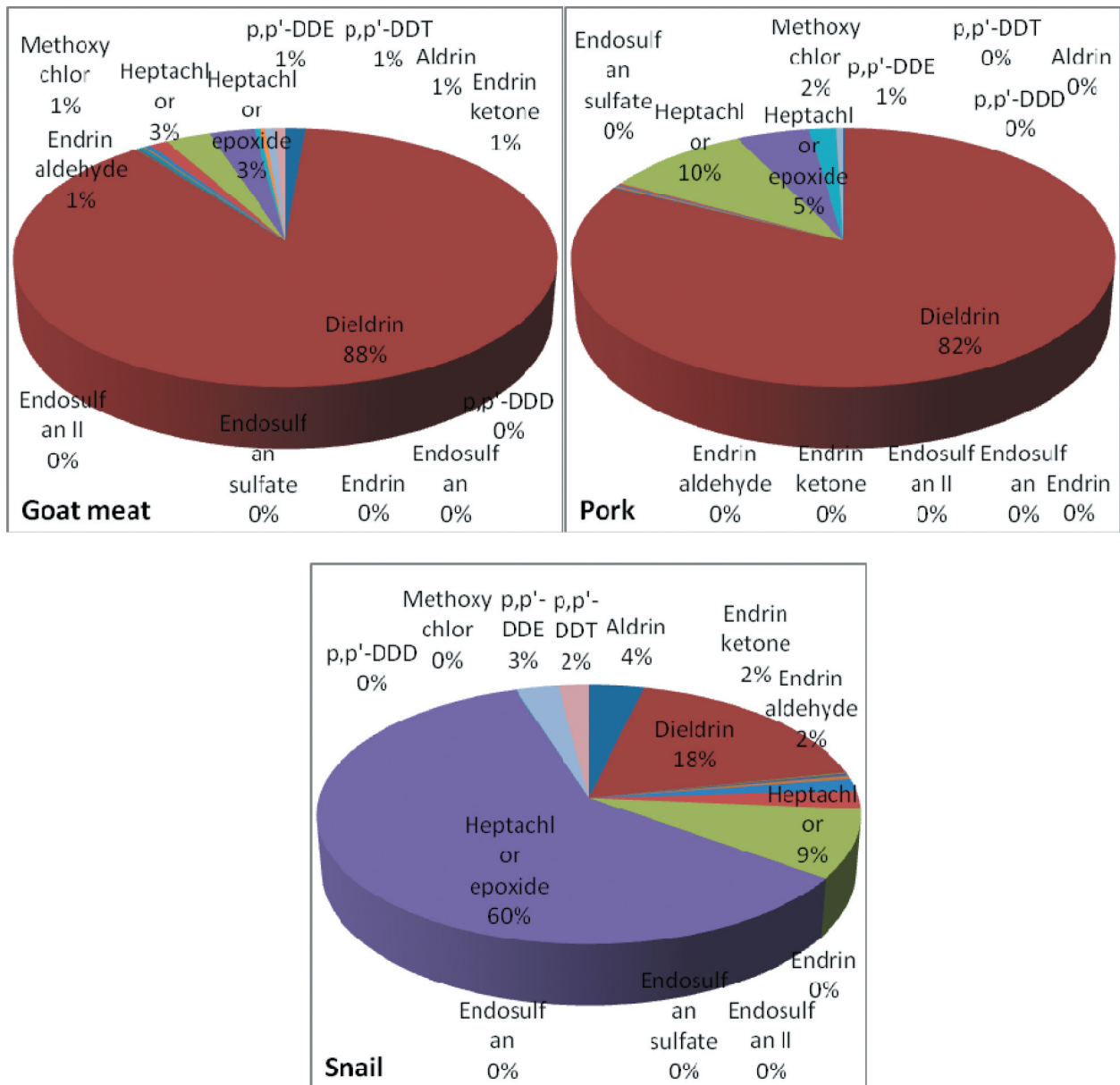


Figure 2. (Continued).

respectively. Heptachlor constituted 24, 44 and 35% of the total HQ in *Clarias gariepinus*, *Tilapia zillii* and crayfish, respectively.

The cancer risk data of OCPs in protein foodstuffs are shown in Tables 3. The CR values of dieldrin, heptachlor and heptachlor epoxide (except *Clarias gariepinus*) in aquatic protein foods were greater than the USEPA permissible limit of  $1.0 \times 10^{-4}$  [37]. The high CR of dieldrin obtained in this study was similar to the reported value by Barnhoorn *et al.* [31] in *Clarias gariepinus* collected from a polluted river in South Africa. For terrestrial protein samples, the CRs of dieldrin in pork, p,p'-DDE in beef, cowskin and pork, and p,p'-DDT in beef were also greater than the acceptable limit of  $1.0 \times 10^{-4}$  indicating possible development of cancer on exposure to protein foods through

ingestion. The  $\Sigma$ CRs in protein foods ranged from  $1.8 \times 10^{-3}$  (*Clarias gariepinus*) to  $6.1 \times 10^{-3}$  (*Tilapia zillii*) for aquatic samples; and  $2.0 \times 10^{-4}$  (snail) to  $4.2 \times 10^{-2}$  (pork) for terrestrial samples. These figures suggested high risk in terms of cancer development by consumers; similar to the recent study of Taiwo [7] on health risk of OCPs in fish samples from different sampling sites in Africa. Furthermore, the CR value greater than  $1.0 \times 10^{-4}$  was documented by Pardío *et al.* [49] in pork samples from Veracruz, Mexico. The high CR value observed for OCPs in pork may be related to the high fat content, and slow metabolism [42].

The contributory fraction of individual OCP to total cancer burden is presented in Figure 3. Like the non-carcinogenic risk, dieldrin was the main contributor to carcinogenic effects in both aquatic

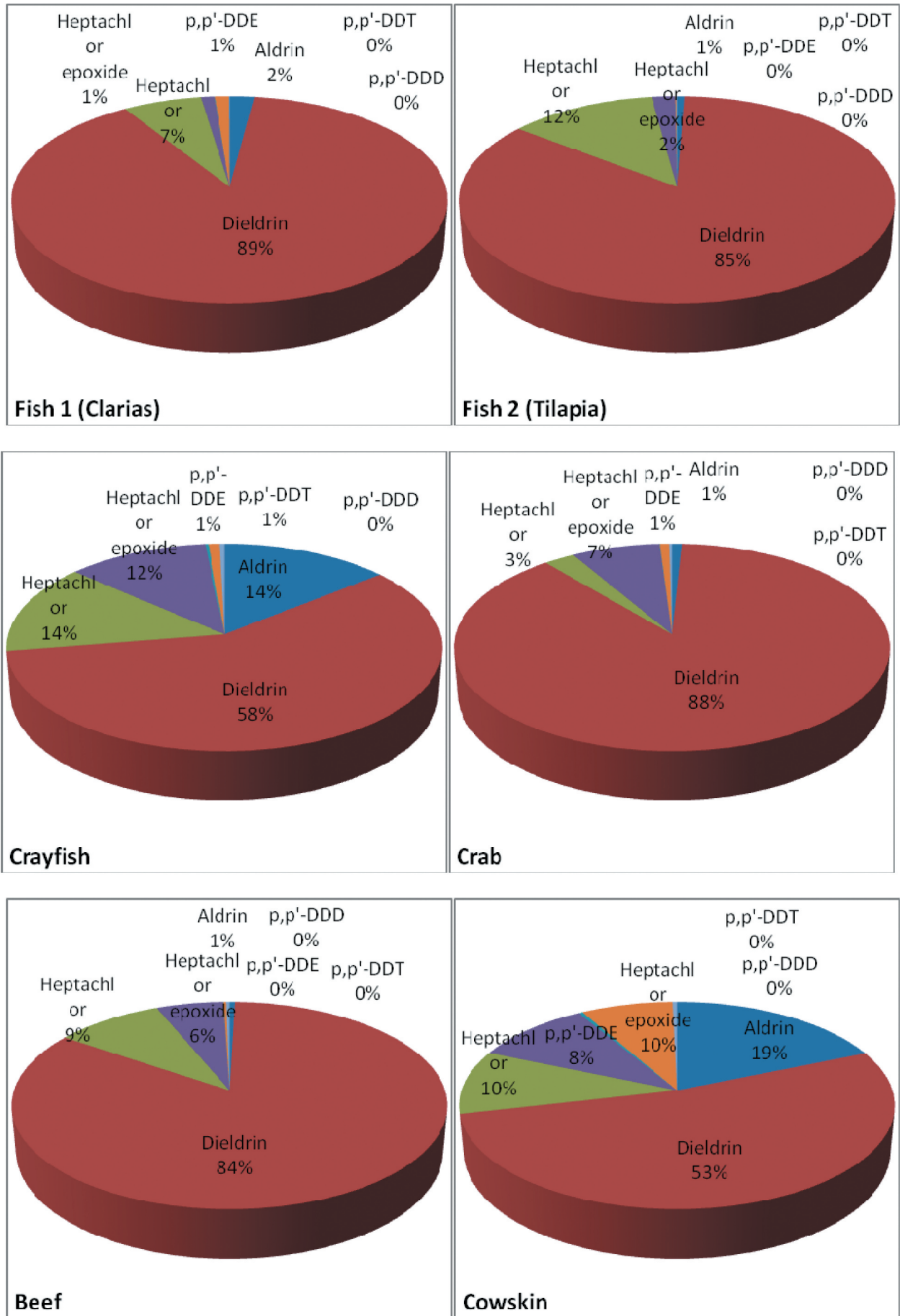


Figure 3. % Contribution to cancer risk by OCPs in protein foods.

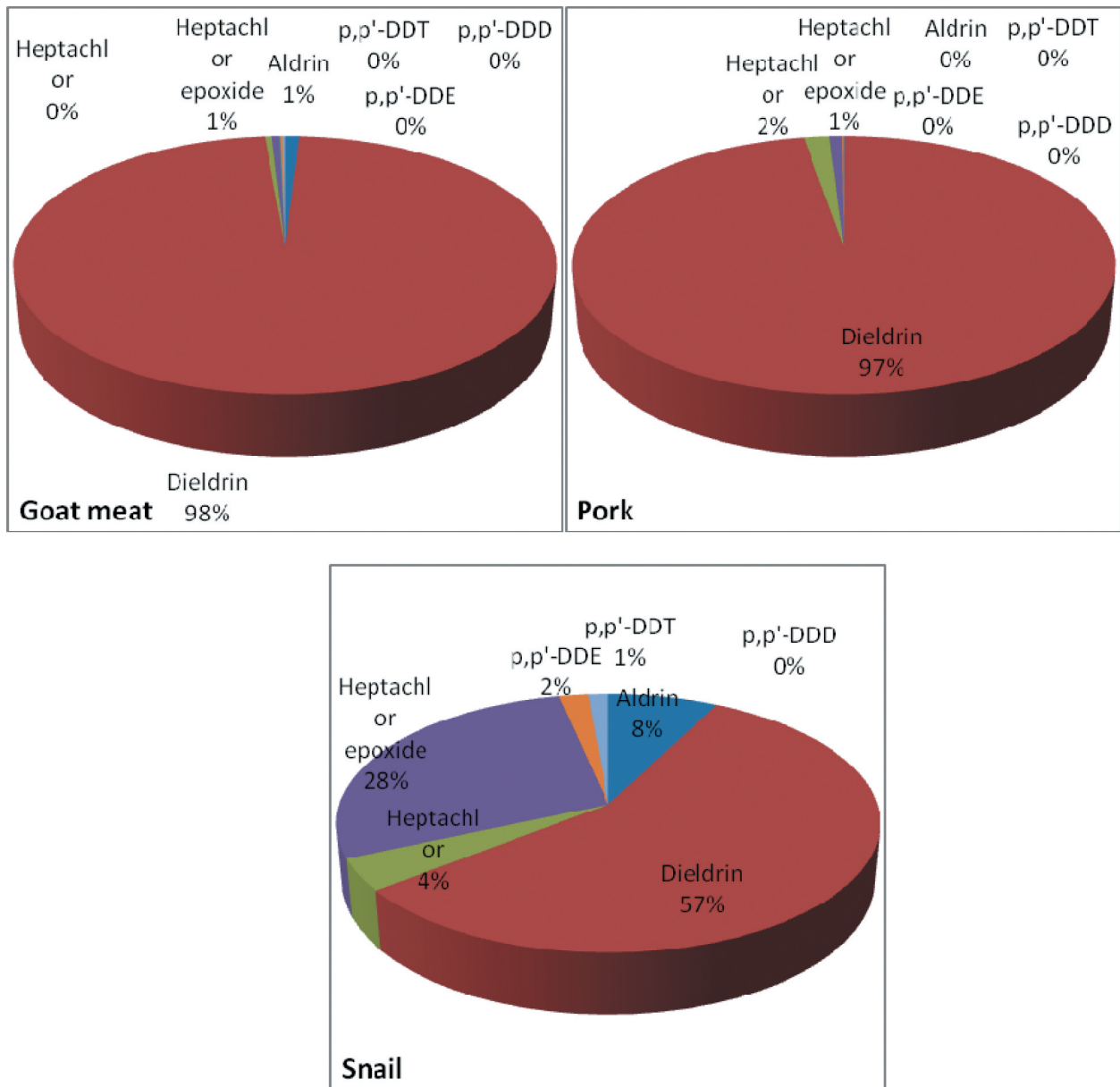


Figure 3. (Continued).

(58–89%) and the terrestrial protein foods (57–98%). Recent studies have shown annual increment in cancer incidence in Nigeria [50–52]. This, therefore, suggests a probable contribution from OCPs contaminated protein foods.

### Conclusion

This study evaluated the health risk of OCPs in different protein foods collected from Abeokuta in southwestern Nigeria. Endrin ketone was found in 89% of the food samples, while aldrin was only detected in crayfish, which represented 2% of the total protein foods. Methoxychlor was the highest observed OCP in food samples. The  $\Sigma$ OCP levels in

protein foods samples followed the decreasing sequence of pork > beef > crab > crayfish > cow-skin > *Clarias gariepinus* > *Tilapia zillii* > goat meat > snail. The Hazard Index (HI) values of OCPs in all the protein foods were found greater than 1.0 indicating adverse health effects. Dieldrin and heptachlor epoxide were the major contributors to the HI in food samples. The total cancer risk data showed carcinogenic effects in all the protein foods with values greater than the acceptable limit of  $1.0 \times 10^{-4}$ . The individual CR values of dieldrin, heptachlor and heptachlor epoxide (except in *Clarias gariepinus*, beef, cowskin, goat meat and snail), p,p'-DDE in beef, cowskin and pork, and p,p'-DDT in beef were also greater than

**Table 2.** HQ and HI values of OCP in aquatic and terrestrial protein foods.

	Protein foods	RfD mg kg <sup>-1</sup> day <sup>-1</sup>	Mean	Std. Deviation	Minimum	Maximum
Aldrin	<i>Clarias gariepinus</i>	0.00003	0.13	0.000	0.13	0.13
	<i>Tilapia zillii</i>		0.13	0.000	0.13	0.13
	Crayfish		2.76	9.47	0.13	32.90
	Crab		0.13	0.000	0.13	0.13
Dieldrin	<i>Clarias gariepinus</i>	0.00005	3.63	0.79	2.76	4.74
	<i>Tilapia zillii</i>		11.68	19.97	0.08	60.08
	Crayfish		7.26	15.16	0.08	52.03
	Crab		9.08	14.84	1.03	39.16
Endosulfan I	<i>Clarias gariepinus</i>	0.006	0.003	0.001	0.003	0.005
	<i>Tilapia zillii</i>		0.01	0.002	0.001	0.01
	Crayfish		0.01	0.01	0.001	0.01
	Crab		0.005	0.001	0.003	0.01
Endosulfan II	<i>Clarias gariepinus</i>	0.006	0.01	0.004	0.005	0.01
	<i>Tilapia zillii</i>		0.01	0.01	0.001	0.03
	Crayfish		0.02	0.05	0.001	0.18
	Crab		0.03	0.05	0.004	0.13
Endosulfan Sulphate	<i>Clarias gariepinus</i>	0.006	0.12	0.02	0.10	0.15
	<i>Tilapia zillii</i>		0.01	0.01	0.001	0.03
	Crayfish		0.01	0.01	0.001	0.02
	Crab		0.07	0.06	0.01	0.16
Endrin	<i>Clarias gariepinus</i>	0.0003	0.03	0.04	0.01	0.11
	<i>Tilapia zillii</i>		0.22	0.37	0.01	1.30
	Crayfish		0.18	0.22	0.01	0.68
	Crab		0.08	0.08	0.01	0.16
Endrin Aldehyde	<i>Clarias gariepinus</i>	0.0003	0.14	0.05	0.09	0.25
	<i>Tilapia zillii</i>		0.24	0.18	0.05	0.74
	Crayfish		0.37	0.75	0.01	2.68
	Crab		1.53	2.76	0.07	7.13
Endrin Ketone	<i>Clarias gariepinus</i>	0.0003	1.05	1.72	0.08	4.50
	<i>Tilapia zillii</i>		0.42	0.24	0.12	1.03
	Crayfish		0.51	0.91	0.01	3.25
	Crab		0.28	0.28	0.01	0.68
Heptachlor	<i>Clarias gariepinus</i>	0.000013	1.82	3.64	0.30	9.41
	<i>Tilapia zillii</i>		11.54	23.99	0.30	70.14
	Crayfish		11.84	24.90	0.30	85.93
	Crab		1.82	4.25	0.30	10.32
Heptachlor Epoxide	<i>Clarias gariepinus</i>	0.000013	0.30	0.000	0.30	0.30
	<i>Tilapia zillii</i>		1.82	2.73	0.30	8.81
	Crayfish		10.02	26.42	0.30	92.92
	Crab		5.16	4.55	0.30	12.45
Methoxychlor	<i>Clarias gariepinus</i>	0.005	0.001	0.000	0.001	0.001
	<i>Tilapia zillii</i>		0.05	0.14	0.001	0.49
	Crayfish		0.17	0.28	0.001	0.74
	Crab		0.36	0.77	0.001	1.92
p,p'-DDD	<i>Clarias gariepinus</i>	0.0005	0.01	0.01	0.01	0.02
	<i>Tilapia zillii</i>		0.01	0.000	0.01	0.01
	Crayfish		0.16	0.54	0.01	1.88
	Crab		0.02	0.01	0.01	0.02
p,p'-DDE	<i>Clarias gariepinus</i>	0.0005	0.23	0.17	0.11	0.55
	<i>Tilapia zillii</i>		0.05	0.05	0.01	0.13
	Crayfish		0.53	1.02	0.01	3.04
	Crab		0.38	0.45	0.06	1.24
p,p'-DDT	<i>Clarias gariepinus</i>	0.0005	0.01	0.000	0.01	0.01
	<i>Tilapia zillii</i>		0.05	0.08	0.01	0.20
	Crayfish		0.24	0.55	0.01	1.97
	Crab		0.11	0.12	0.01	0.23
HI	<i>Clarias gariepinus</i>		7.49			
	<i>Tilapia zillii</i>		26.23			
	Crayfish		34.10			
	Crab		19.04			
Aldrin	Beef	0.00003	0.13	0.000	0.13	0.13
	Cowskin		0.13	0.000	0.13	0.13
	Goat meat		0.13	0.000	0.13	0.13
	Pork		0.13	0.000	0.13	0.13
	Snail		0.13	0.000	0.13	0.13
Dieldrin	Beef	0.00005	15.16	18.95	1.74	40.42
	Cowskin		0.24	0.39	0.08	0.71
	Goat meat		8.68	14.84	1.50	38.92
	Pork		92.29	116.37	0.08	318.88
	Snail		0.63	0.71	0.08	1.42
Endosulfan	Beef	0.006	0.01	0.005	0.003	0.02
	Cowskin		0.01	0.002	0.003	0.01
	Goat meat		0.01	0.001	0.003	0.01
	Pork		0.01	0.003	0.001	0.01
	Snail		0.01	0.01	0.001	0.01
Endosulfan II	Beef	0.006	0.01	0.01	0.003	0.02

(Continued)

**Table 2.** (Continued).

	Protein foods	RfD mg kg <sup>-1</sup> day <sup>-1</sup>	Mean	Std. Deviation	Minimum	Maximum
	Cowskin		0.001	0.002	0.000	0.01
	Goat meat		0.01	0.01	0.01	0.02
	Pork		0.08	0.06	0.001	0.18
	Snail		0.01	0.02	0.001	0.05
	Beef	0.006	0.02	0.01	0.01	0.04
Endosulfan Sulphate	Cowskin		0.09	0.04	0.04	0.14
	Goat meat		0.04	0.04	0.01	0.10
	Pork		0.08	0.07	0.001	0.15
	Snail		0.003	0.003	0.001	0.01
	Beef	0.0003	0.33	0.24	0.08	0.67
Endrin	Cowskin		0.04	0.07	0.01	0.17
	Goat meat		0.01	0.00	0.01	0.01
	Pork		0.24	0.34	0.01	0.89
	Snail		0.01	0.000	0.01	0.01
	Beef	0.0003	0.50	0.80	0.11	2.14
Endrin Aldehyde	Cowskin		1.04	1.46	0.09	3.89
	Goat meat		0.04	0.05	0.01	0.14
	Pork		0.20	0.14	0.01	0.43
	Snail		0.05	0.07	0.01	0.18
	Beef	0.0003	0.76	1.38	0.12	3.57
Endrin Ketone	Cowskin		1.80	2.49	0.16	6.59
	Goat meat		0.12	0.01	0.09	0.13
	Pork		0.25	0.16	0.01	0.46
	Snail		0.08	0.09	0.01	0.24
	Beef	0.000013	11.24	13.66	0.30	28.54
Heptachlor	Cowskin		0.30	0.00	0.30	0.30
	Goat meat		0.30	0.00	0.30	0.30
	Pork		11.24	5.77	0.30	14.88
	Snail		0.30	0.00	0.30	0.30
	Beef	0.000013	6.98	4.25	0.30	12.15
Heptachlor Epoxide	Cowskin		0.30	0.000	0.30	0.30
	Goat meat		0.30	0.000	0.30	0.30
	Pork		5.47	3.34	0.30	9.41
	Snail		2.13	3.04	0.30	6.07
	Beef	0.005	0.43	0.55	0.01	1.39
Methoxychlor	Cowskin		0.002	0.002	0.001	0.005
	Goat meat		0.04	0.07	0.001	0.17
	Pork		2.08	1.47	0.001	4.48
	Snail		0.002	0.002	0.001	0.01
	Beef	0.0005	0.02	0.05	0.01	0.12
p,p'-DDD	Cowskin		0.01	0.000	0.01	0.01
	Goat meat		0.03	0.02	0.01	0.07
	Pork		0.02	0.02	0.01	0.06
	Snail		0.000	0.000	0.000	0.01
	Beef	0.0005	0.19	0.10	0.04	0.32
p,p'-DDE	Cowskin		0.17	0.27	0.01	0.69
	Goat meat		0.06	0.04	0.01	0.11
	Pork		0.48	0.24	0.01	0.64
	Snail		0.10	0.06	0.05	0.21
	Beef	0.0005	0.24	0.11	0.12	0.42
p,p'-DDT	Cowskin		0.01	0.000	0.01	0.01
	Goat meat		0.07	0.16	0.01	0.39
	Pork		0.01	0.000	0.01	0.01
	Snail		0.07	0.17	0.01	0.40
	Beef	0.0005	36.028			
HI	Cowskin		4.140			
	Goat meat		9.856			
	Pork		112.565			
	Snail		3.530			
	Beef	0.0005	36.028			

RfD-Reference dose [35]



**Table 3.** Cancer risk values of OCP in aquatic and terrestrial protein foods.

	Protein foods	Slope Factor mg <sup>-1</sup> mg day	Mean	Std. Deviation	Minimum	Maximum
Aldrin	<i>Clarias gariepinus</i>	17	3.7E-05	0.0E+00	3.7E-05	3.7E-05
	<i>Tilapia zillii</i>		3.7E-05	0.0E+00	3.7E-05	3.7E-05
	Crayfish		7.8E-04	2.7E-03	3.7E-05	9.2E-03
	Crab		3.7E-05	0.0E+00	3.7E-05	3.7E-05
Dieldrin	<i>Clarias gariepinus</i>	16	1.6E-03	3.5E-04	1.2E-03	2.1E-03
	<i>Tilapia zillii</i>		5.1E-03	8.8E-03	3.5E-05	2.6E-02
	Crayfish		3.2E-03	6.7E-03	3.5E-05	2.3E-02
	Crab		4.0E-03	6.5E-03	4.5E-04	1.7E-02
Heptachlor	<i>Clarias gariepinus</i>	9.1	1.2E-04	2.4E-04	2.0E-05	6.1E-04
	<i>Tilapia zillii</i>		7.5E-04	1.6E-03	2.0E-05	4.6E-03
	Crayfish		7.7E-04	1.6E-03	2.0E-05	5.6E-03
	Crab		1.2E-04	2.8E-04	2.0E-05	6.7E-04
Heptachlor Epoxide	<i>Clarias gariepinus</i>	9.1	2.0E-05	0.0E+00	2.0E-05	2.0E-05
	<i>Tilapia zillii</i>		1.2E-04	1.8E-04	2.0E-05	5.7E-04
	Crayfish		6.5E-04	1.7E-03	2.0E-05	6.1E-03
	Crab		3.4E-04	3.0E-04	2.0E-05	8.1E-04
p,p'-DDD	<i>Clarias gariepinus</i>	0.34	7.4E-07	7.4E-07	7.4E-07	1.5E-06
	<i>Tilapia zillii</i>		7.4E-07	0.0E+00	7.4E-07	7.4E-07
	Crayfish		1.5E-05	5.1E-05	7.4E-07	1.8E-04
	Crab		1.5E-06	7.4E-07	7.4E-07	2.2E-06
p,p'-DDE	<i>Clarias gariepinus</i>	0.34	2.1E-05	1.6E-05	1.0E-05	5.2E-05
	<i>Tilapia zillii</i>		4.4E-06	4.4E-06	7.4E-07	1.2E-05
	Crayfish		4.9E-05	9.5E-05	7.4E-07	2.8E-04
	Crab		3.5E-05	4.2E-05	5.2E-06	1.2E-04
p,p'-DDT	<i>Clarias gariepinus</i>	0.34	7.4E-07	0.0E+00	7.4E-07	7.4E-07
	<i>Tilapia zillii</i>		4.4E-06	7.4E-06	7.4E-07	1.8E-05
	Crayfish		2.3E-05	5.2E-05	7.4E-07	1.8E-04
	Crab		1.0E-05	1.1E-05	7.4E-07	2.1E-05
ΣCR	<i>Clarias gariepinus</i>		1.8E-03			
	<i>Tilapia zillii</i>		6.1E-03			
	Crayfish		5.5E-03			
	Crab		4.5E-03			
Aldrin	Beef	17	3.7E-05	0.0E+00	1.3E-07	1.3E-07
	Cowskin		3.7E-05	0.0E+00	1.3E-07	1.3E-07
	Goat meat		3.7E-05	0.0E+00	1.3E-07	1.3E-07
	Pork		3.7E-05	0.0E+00	1.3E-07	1.3E-07
	Snail		3.7E-05	0.0E+00	1.3E-07	1.3E-07
Dieldrin	Beef	16	6.7E-03	3.3E-05	3.0E-06	7.0E-05
	Cowskin		1.0E-04	6.8E-07	1.4E-07	1.2E-06
	Goat meat		3.8E-03	2.6E-05	2.6E-06	6.7E-05
	Pork		4.1E-02	2.0E-04	1.4E-07	5.5E-04
	Snail		2.8E-04	1.2E-06	1.4E-07	2.4E-06
Heptachlor	Beef	9.1	7.3E-04	1.1E-05	2.4E-07	2.2E-05
	Cowskin		2.0E-05	0.0E+00	2.4E-07	2.4E-07
	Goat meat		2.0E-05	0.0E+00	2.4E-07	2.4E-07
	Pork		7.3E-04	4.5E-06	2.4E-07	1.2E-05
	Snail		2.0E-05	0.0E+00	2.4E-07	2.4E-07
Heptachlor Epoxide	Beef	9.1	4.5E-04	3.3E-06	2.4E-07	9.6E-06
	Cowskin		2.0E-05	0.0E+00	2.4E-07	2.4E-07
	Goat meat		2.0E-05	0.0E+00	2.4E-07	2.4E-07
	Pork		3.6E-04	2.6E-06	2.4E-07	7.4E-06
	Snail		1.4E-04	2.4E-06	2.4E-07	4.8E-06
p,p'-DDD	Beef	0.34	2.2E-06	3.8E-05	6.4E-06	9.6E-05
	Cowskin		7.4E-07	0.0E+00	6.4E-06	6.4E-06
	Goat meat		3.0E-06	1.9E-05	6.4E-06	5.8E-05
	Pork		2.2E-06	1.9E-05	6.4E-06	5.1E-05
	Snail		0.0E+00	0.0E+00	0.0E+00	6.4E-06
p,p'-DDE	Beef	0.34	1.8E-05	8.3E-05	3.2E-05	2.6E-04
	Cowskin		1.6E-05	2.2E-04	6.4E-06	5.6E-04
	Goat meat		5.9E-06	3.2E-05	6.4E-06	8.9E-05
	Pork		4.5E-05	1.9E-04	6.4E-06	5.2E-04
	Snail		9.6E-06	4.5E-05	3.8E-05	1.7E-04
p,p'-DDT	Beef	0.34	2.3E-05	8.9E-05	9.6E-05	3.4E-04
	Cowskin		7.4E-07	0.0E+00	6.4E-06	6.4E-06
	Goat meat		6.6E-06	1.3E-04	6.4E-06	3.1E-04
	Pork		7.4E-07	0.0E+00	6.4E-06	6.4E-06
	Snail		6.6E-06	1.3E-04	6.4E-06	3.3E-04
ΣCR	Beef		7.9E-03			
	Cowskin		2.0E-04			
	Goat meat		3.9E-03			
	Pork		4.2E-02			
	Snail		4.9E-04			

the acceptable limit of  $1.0 \times 10^{-4}$ . The  $\Sigma$ CR was mostly contributed by p,p'-DDE and dieldrin. The health risk data indicated that despite the ban of OCPs in many developing countries; the present concentrations in protein foods are of serious health concerns.

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## Disclosure statement

No potential conflict of interest was reported by the authors.

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