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Occurrence and Potential Risk of Organophosphorus Pesticide Residues in Some Edible Vegetable Oils Sold in Markets in Warri, Delta State

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ABSTRACT: The study was to determine the concentrations and risks of organophosphorus pesticides (OPPs) residues in edible vegetable oils (EVOs) sold in markets in Warri, Delta State. OPPs in six edible vegetable oils purchased from local vendors in markets in Warri, Delta State were quantified with a gas chromatograph coupled with a mass spectrometry (GC-MS) after solvent extraction. The results showed concentration of the ∑14 OPPs varying from 4.10 to 22.82 ng/L. The unbranded edible vegetable oils had higher concentrations than their respective branded vegetable oils. On average, azinphos ethyl was the dominant OPP compound in the edible vegetable oils. The concentrations of the individual OPPs compound found in the edible vegetable oil were lower than their respective maximum residue limit (MRLs) stipulated by the Food and Agriculture Organization/World Health Organization (FAO/WHO). The computed possible hazard index (HI) values were generally < 1 and indicated that there is no adverse or carcinogenic risk from the intake of these edible vegetable oils. The input of each OPP congener to the HI was in the order of EPN (O-ethyl O-4-nitrophenyl phenylphosphonothioate) > pirimiphos methyl > quinalphos > chloropyrifos methyl > azinphos ethyl > diazinone > chloropyrifos.

Keywords: OPPs, Oils, GC-MS, Hazard index, Maximum residue limit

Introduction

Organophosphorus pesticides (OPPs) are a class of compounds that includes derivatives of phosphoric, phosphorous, thiono-phosphoric, and thion-thiolo phosporic acids esterified with methyl or ethyl and different alcohol groups which were first made available for purchase in the 1930s (Vale and Lotti, 2015). Depending on the alcoholic moiety, the polarity of OPPs ranges from water-soluble compounds such as dimethoate to lipophilic compounds such as bromophos-ethyl (Di Muccio *et al*., 2006). The majority of OPPs are insecticides with anticholinesterase activity. OPPs are the most commonly used pesticides, making up for about 40% of the global pesticide market, despite the fact that demand for them is still rising due to their effectiveness, reliability, lack of pest resistance, broad range of applications, capacity for multi-pest control, low cost, and other recent economic advantages (Sapbamrer and Hongsihong, 2014). Many OPPs are endocrine disrupting chemicals, carcinogenic, cytotoxic, mutagenic, immunotoxic and teratogenic (Boulanouar *et al*., 2018). Although banned for many years, these compounds still exist in the environment at present (Barakat *et al*., 2013; Li *et al*., 2013). More seriously, because of the volatility and long-range transport through the atmosphere, they can be found in areas where they have never been used or produced (Cabrerizo *et al*., 2012). In other words, they may be detected in any place in the environment. OPPs can be taken up by crops from the contaminated soil or the polluted air (Tesi *et al*., 2022), and transferred into different tissues of plants. For oil crops, they may accumulate in the leaves and the oil seeds easily and consequently exist in the oils because of their lipophilicity (Li *et al*., 2014). The OPPs in the oils can be transferred along the food chain and dietary exposure to these compounds may cause risks to human health.

Edible vegetable oils are ranked among the top widely consumed food items globally. Edible vegetable oils are foodstuffs which are composed primarily of glycerides of fatty acids being obtained only from vegetable or plant sources (Ahmed *et al*., 2021). Vegetable oils constitute the main cooking oils consumed by humans. They possess a rich content of essential fatty acids, phytosterols, tocopherol, as well as high levels of antioxidative nutrients like monosaturated fatty acids, which unlike animal fats, are predominantly saturated (Amadi *et al*., 2022). After carbohydrates, they are the second most important source of energy and essential fatty acids in the human diet. Edible vegetable oils also serve as a heating medium and are important in the generation of aroma, some of which arise from direct interaction of lipids and/or their degradation products with food constituents (Shahidi, 2004). Ingestion of contaminants such as OPPs through consumption of edible oils represents one third of the total intake from food (Iwegbue *et al*., 2020). Therefore, oil consumption is one of the major sources of human exposure to contaminants such as OPPs. As a response, the European Union, the World Health Organization (WHO) and the Codex Alimentarius Commission of the Food and Agriculture Organization (FAO) of the United Nations have established maximum pesticide residue limits (MRL) for edible oils (Liu *et al*., 2013). Maximum residue limits (MRLs) set by the FAO for phorate and pirimiphos-methyl is 50 ng/g, chlorpyrifos is 10 ng/g, diazinon is 20 ng/g and chlorpyrifos-methyl are 100 ng/g in soybean oil while Fenamiphos and phorate in peanut oil, were set at 50 ng/g. The European Union standards for MRLs of oils for phorate and methidathion were 20 ng/g, parathion 50 ng/g, fenamiphos 5 ng/g, chlorpyrifos and diazinon were 10 ng/g.

In contemporary daily cooking practice, animal oils have been gradually replaced by vegetable oils due to health problems related to fat. As a necessity in daily life, edible vegetable oil is a large part in daily diet, particularly in Africa and Nigeria in particular. Edible vegetable oils are consumed by all the tribes in Nigeria, and constitute a major ingredient of most Nigerian soups, stews and other foods (Iwegbue *et al*., 2020). The per capita consumption of edible oil in Nigeria as at 2021 was 11.1 kg per capita per year (Iwegbue *et al*., 2019; Iwegbue *et al*., 2020) and Nigeria's edible oils consumption is expected to reach around 2.2 million metric tons by 2026 (Nigeria Edible Oil Industry Outlook, 2022-2026). So, the determination and monitoring of OPPs in edible vegetable oils are significant for the assessment of dietary safety. Many studies have reported pesticides (OPPs and OCPs) contamination in edible oils in different parts of the World (Di Muccio *et al*., 2006; Li *et al*., 2007; Bajpal *et al*., 2007; Skrbic and Predojevic, 2008; Li *et al*., 2013; Liu *et al*., 2015; Wu *et al*., 2020). However, based on literature search, there are no information on the occurrence of OPPs in edible oils in Nigeria. The only available studies on edible oils in Nigeria focused on metals and PAHs (e.g. Iwegbue *et al*., 2019; Iwegbue *et al*., 2020; Ojezele *et al*., 2021; Amadi *et al*., 2022). Thus, the objectives of this study are to determine the concentrations and risks of organophosphate pesticide residues in edible vegetable oils sold in the Nigerian market.

Materials and methods

Collection of Samples: A total of 6 edible vegetable oils comprising branded and unbranded palm oil, groundnut oil and soybean oil (i.e. three branded oil and three unbranded oil) were used. The edible vegetable oils were purchased from retail outlets in Warri, Delta State, Nigeria. The samples were stored in the refrigerator prior to analysis.

Reagents: All reagents used were HPLC grade. Acetonitrile, ethyl acetate, methanol, petroleum ether, and n-hexane were acquired from BDH (Poole, UK). The standard of fourteen (14) OPPs, including Pyrachlofos, azinphos ethyl, pyrazophos, phosalone, EPN, triphenyl phosphate, chlorpyrifos, quinaphos, pirimiphos ethyl, fenitrothion, pirimiphos methyl, chlorpyrifos methyl, isazophos and diazinone were from Accu Standard, USA. Anhydrous sodium sulphate was from Merck (Darmstadt, Germany).

OPPs extraction and quantification: The extraction method of Li *et al*., (2007) was adopted for the extraction of OPPs from the vegetable oils in this study. 5 grams of the edible vegetable oils was weighed and transferred into 50-mL centrifuge tubes. 10 mL of acetonitrile was added, and the cap was screwed on. The sample was shaken vigorously by the mixer for 5 min. The tubes were stored horizontally in the freezer overnight at -20 °C for oil precipitation. Then a 1.0-mL aliquot of extract was transferred into a 2-mL centrifuge tube, evaporated under gentle nitrogen stream until nearly dry. Thereafter, the extract volume was increased up to 0.5 mL with ethyl acetate, and finally transferred into an autosampler vial for chromatographic analysis. The determination of OPPs in the sample extracts was done using a gas chromatograph (Agilent 6890 N) coupled with a mass spectrometry (GC–MS). The working conditions of the gas chromatograph was documented during the analysis.

Quality control and assurance: The study employed the European Commission's (2019) requirements for technique validation. Blanks and recovery study were used. OPP values in blank samples were below the limit of detection. Already examined samples were spiked with 20 ng/g of OPPs standard solutions and then evaluated. The percentage recovered were subsequently computed. The percentage of OPPs recovered varied from 91.4% to 98.7%.

Statistical analysis: The analysis of data was done with the IBM-SPSS software (version 23). T-test was used to determine if the concentrations of OPPs in the branded and unbranded vegetable oil varied significantly. Also, analysis of variance was used to determine if the OPPs concentration varied significantly among the different types of vegetable oils for a given category.

Estimation of daily intake (EDI): The EDI of OPPs through consumption of these vegetable oils was obtained using the expression (1);

$$\text{Daily intake (ng/kg bw/day)} = \frac{\text{Concentration of OPPs in oil} \times \text{Ingestion Rate (IR)}}{\text{Body Weight (BW)}} \quad (1)$$

The IR was gotten from the per capita vegetable oil consumption of 11.1 kg per annum per person in Nigeria which amount to 30.4 g/day and a BW of 60 kg for adult and 15 kg for children (Iwegbue *et al*., 2019; 2020).

Estimation of non-cancer: The non-cancer risk associated with OPPs via consumption of the vegetable oils were assessed as hazard index (HI) and were evaluated using equations (2) and (3) (Iwegbue *et al*., 2019; 2020; USEPA 2022; Tesi *et al*., 2022). The hazard quotient (HQ) for the individual OPPs congeners was computed and the HI was obtained by adding up the HQs of the OPPs on the basis of dose additivity (Bommuraj *et al*., 2019; Mukiibi *et al.*, 2021) as expressed in equations 2 and 3.

$$HI = HQ1 + HQ2 + HQ3 + \ldots + HQn \quad (2)$$

$$HQ = \left[\frac{\text{Concentration} \times IR \times EF \times ED}{BW \times AT_{nc}} \times 10^{-6} \right] / RfD \quad (3)$$

where, RfD = oral reference dose. The RFD values that were used are chlorpyrifos (1×10^{-3}), chlorpyrifos methyl (1×10^{-2}), diazinone (7×10^{-4}), pirimyphos methyl (1×10^{-2}), quilnaphos (5×10^{-4}) and EPN (1×10^{-5}) (USEPA, 2022). The EF = exposure frequency (day/yr) = 350; ED = exposure duration = 6 and 30 years for children and adults respectively (USEPA, 2022); ATnc = averaging time for non-cancer = ED x 365 while IR = ingestion rate = 30.4 g/day. The HI value > 1 indicates the possibility of deleterious non-carcinogenic risk (USEPA, 2022).

Results and Discussion

Concentrations of OPPs in the edible vegetable oils: The concentrations of OPPs in the edible vegetable oils (EVOs) are shown in Table 1.

Table 1: OPPs concentrations (ng g^{-1}) in edible vegetable oils (EVOs)

OPPs	% DF	Branded Groundnut Oil	Unbranded Groundnut Oil	Branded Palm Oil	Unbranded Palm Oil	Branded Soyabean Oil	Unbranded Soyabean Oil
Diazinone	50	<0.01	0.08	<0.01	<0.01	0.68	0.88
Isazophos	67	0.35	0.65	0.62	0.62	<0.01	<0.01
Chlorpyriphos Methyl	100	0.67	1.37	0.41	1.81	0.65	0.95
Pirimiphos Methyl	100	0.79	1.09	0.12	1.92	0.50	0.87
Fenitrthion	67	0.43	1.43	<0.01	<0.01	0.40	0.90
Pirimiphos Ethyl	100	0.61	1.11	0.04	0.74	0.43	0.95
Quinalphos	83	0.81	1.09	0.11	0.91	0.61	<0.01
Chlorpyrifos	17	<0.01	<0.01	<0.01	<0.01	<0.01	1.01
Triphenyl Phosphate	100	0.85	1.05	0.53	1.12	0.63	1.03
EPN	100	1.05	2.05	0.14	0.14	0.83	1.03
Phosalone	100	0.93	3.33	0.84	0.86	0.61	1.11
Pyrazophos	100	1.19	2.19	0.02	0.72	1.96	3.06
Azinphos Ethyl	100	0.95	4.45	0.53	0.73	1.75	4.57
Pyraclofos	100	0.43	2.93	0.74	0.94	1.91	4.91
∑14 OPPs	**100**	**9.06**	**22.82**	**4.10**	**10.51**	**10.96**	**21.27**

DF = Detection frequency

The detection frequency of the individual OPPs congeners in the EVOs varied from 17 to 100 %. The presence of OPPs in the studied EVOs could be because vegetables and plants generally are extremely vulnerable to pests during cultivation and constant usage of pesticides is necessary to get rid of pests, which ultimately lead to their elevated levels (Tesi *et al*., 2025; Qin *et al*., 2015). The concentration of the ∑14 OPPs vary from 4.10 to 22.82 ng g^{-1}. T-test analysis showed that there was significant variation ($p < 0.05$) in the concentrations of OPPs between the branded and unbranded edible oils. The unbranded edible vegetable oils had higher concentrations than their respective branded vegetable oils. This might be due to inadequate quality control and non-uniform application of active ingredients, in addition to uneven contamination during the vegetable or plant contamination (Tesi *et al*., 2024). There was also significant variation ($p < 0.05$) in the concentrations of OPPs among the different types of edible oils. The significant variation observed might be due to production process. The concentrations of the ∑14 OPPs in the EVOs were in the order of unbranded soyabean oil > unbranded groundnut oil > unbranded palm oil > branded soyabean oil > branded groundnut oil > branded palm oil. On average, the concentrations of the OPPs in the EVOs were in the order of Azinphos ethyl (2.16 ng g^{-1}) > pyraclofos (1.98 ng g^{-1}) > pyrazophos (1.52 ng g^{-1}) > phosalone (1.28 ng g^{-1}) > chlorpyriphos methyl (0.98 ng g^{-1}) > pirimiphos methyl (0.88 ng g^{-1}) > triphenyl phosphate (0.87 ng g^{-1}) > EPN (0.87 ng g^{-1}) > pirimiphos ethyl (0.65 ng g^{-1}) > quinalphos (0.59 ng g^{-1}) > fenitrothion (0.53 ng g^{-1}) > isazophos (0.37 ng g^{-1}) > diazinone (0.27 ng g^{-1}) > chlorpyrifos (0.17 ng g^{-1}) (Figure 1). Unbranded groundnut oil recorded the highest level of isazophos, fenithrothion, pirimiphos ethyl, quinalphos, EPN and phosalone. Unbranded palm oil recorded the highest level of chlorpyriphos methyl, pirimiphos methyl and triphenyl phosphate while unbranded soyabean oil recorded the levels of diazinone, chlorpyrifos, pyrazophos, azinphos ethyl and pyraclofos.

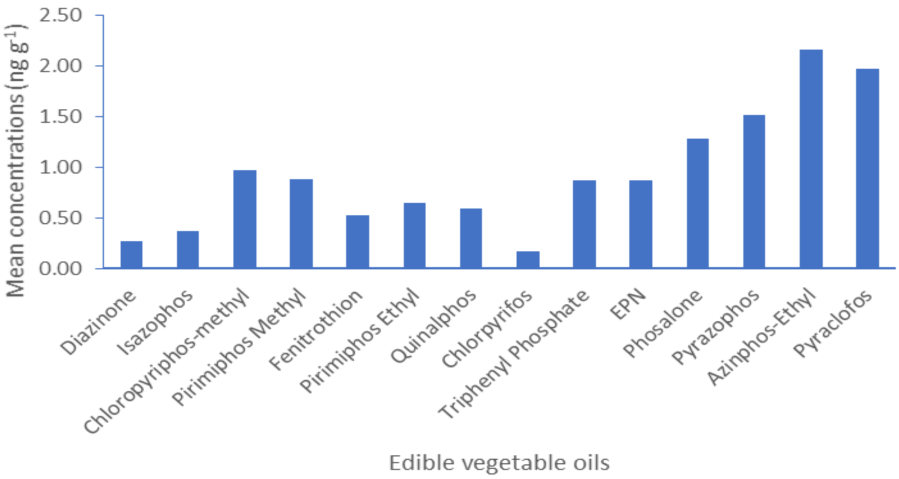

Figure 1: Mean distribution of OPPs congeners in the edible vegetable oils

The concentrations of the individual OPPs found in the edible vegetable oil were lower than the maximum residue limit (MRL) of 10 ng g^{-1} stipulated by the Food and Agriculture Organization/World Health Organization (FAO/WHO). The concentrations of ∑14 OPPs obtained in this study were comparable to the 25 ng g^{-1} reported for a peanut oil (Li *et al*., 2007). Whereas, the concentrations of the individual OPPs obtained in this study were comparable to the limit of detection and limit of quantification of 0.2 to 0.6 and 0.7 to 2.0 ng g^{-1} respectively reported by Wu *et al*. (2019); 1.1 to 6.7 ng g^{-1} and 4.8 to 18.3 ng g^{-1} respectively reported by Liu *et al*. (2013) and 2.0 to 5.0 ng g^{-1} and 6.0 to 18.0 ng g^{-1} respectively reported by Li *et al*. (2017).

Compositional pattern of OPPs in the edible vegetable oils: The compositional patterns of the OPPs in the vegetables are presented in Figure 2.

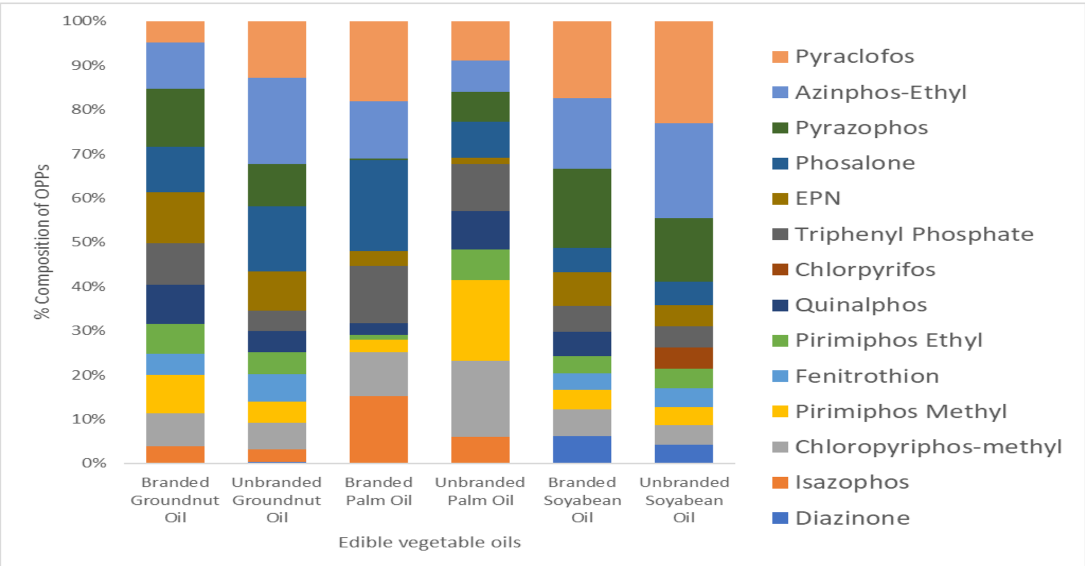

Figure 2: Compositional pattern of OPPs in the edible vegetable oils

The diazinone levels in 50 % of the EVOs varied between 0.08 and 0.88 ng g^{-1} constituting 0.4 to 6.2 % of the ∑14 OPPs. Diazinone was not detected in branded groundnut oil, branded palm oil and unbranded groundnut oil. The isazophos concentrations in 67 % of the samples ranged from 0.35 to 0.65 ng g^{-1} and accounted for 2.8 to 15.1 % of the ∑14 OPPs. Isazophos was not detected in branded and unbranded soyabean oils. The concentrations of chloropyrifos-methyl in all the EVO samples ranged from 0.41 to 1.81 ng g^{-1}. The Chloropyrifos-methyl constituted 4.5 to 17.2 % of the ∑14 OPPs. The pirimiphos-methyl concentrations in the EVO samples ranged from 0.12 to 1.92 ng g^{-1}. Pirimiphos-methyl constituted 2.9 to 18.3 % of the ∑14 OPPs. The fenitrothion levels 67 % of the EVOs varied from 0.4 to 1.43 ng g^{-1} and made up 3.6 to 6.3 % of the ∑14 OPPs. The pirimiphos-ethyl content in all the EVOs ranged from 0.04 to 1.11 ng g^{-1} and constituted 1.0 to 7.0 % of the ∑14 OPPs. The quinalphos levels in 83 % of the EVOs ranged from 0.11 to 1.09 ng g^{-1} and constituted 2.7 to 8.9 % of the ∑14 OPPs. Chlorpyrifos was detected only in unbranded soyabean oil with a concentration of 1.01 and constituted 4.7 % of the ∑14 OPPs. The triphenyl phosphate concentrations in the EVOs ranged from 0.53 to 1.12 ng g^{-1} and constituted 4.6 to 12.9 % of the ∑14 OPPs. The EPN contents of the EVOs ranged between 0.14 and 2.05 ng g^{-1} constituting 1.3 to 11.6 % of the ∑14 OPPs. The phosalone content in the EVOs ranged from 0.61 to 3.33 ng g^{-1} constituting 5.2 to 20.5 % of the ∑14 OPPs. The pyrazophos content in the EVOs ranged between 0.02 and 3.06 ng g^{-1} constituting 0.5 to 17.9 % of the ∑14 OPPs. The azinphos-ethyl concentrations in the EVOs ranged from 0.53 to 4.57 ng g^{-1} and constituted 6.9 to 21.5 % of the ∑14 OPPs. The pyraclofos concentrations in the EVOs ranged from 0.43 to 4.91 ng g^{-1} and constituted 4.7 to 23.1 % of the ∑14 OPPs. The amount of OPPs residues in plants depends on many factors including the biological properties of the plants and the physicochemical properties of OPPs (Tesi *et al*., 2025). These factors may have synergistic or additive effects (negative or positive) on the half-lives of OPPs. Therefore, it can be concluded that under some conditions, some of the OPPs may degrade or be eliminated faster than the others from environment and be undetectable (Qin *et al.*, 2015). Furthermore, plant exudates which are comprised of various phytochemicals and lipids, play a crucial role in OPPs accumulation. These small molecules, released by plants, possess a strong affinity for soil organic content. As a result, they affect the mobility of pollutants such as OPPs, reducing their hydrophobic properties and aiding in their sequestration by plant tissues (Liu *et al*., 2015).

Estimated daily intake of OPPs in the edible vegetable oils: The EDI of OPPs from the ingestion of the studied edible vegetable oils by children and adults are shown in Figure 3. The daily intake values based on the ∑14 OPPs concentrations varied from 8.31 to 46.2 ng kg^{-1} bw day^{-1} for children and 2.08 to 11.6 ng kg^{-1} bw day^{-1} for adults. The maximum daily intake values of OPPs were found in unbranded groundnut oil while the minimum daily intake was found in branded palm oil.

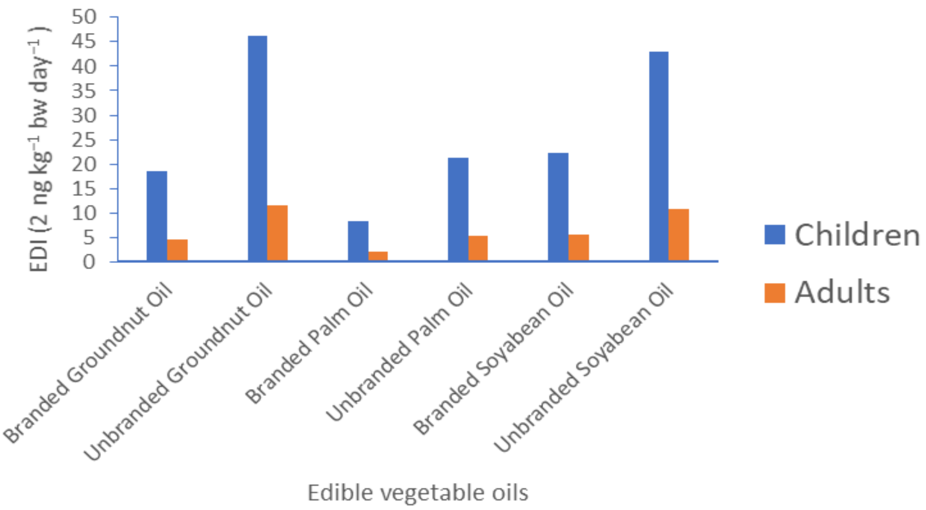

Figure 3: Estimated daily intake of OPPs in the edible vegetable oils

Health risk of OPPs in the edible vegetable oils: The results of the non-carcinogenic risk obtained as HI associated with the OPPs exposure to children and adults through intake of the edible vegetable oils is shown in Table 2. The HI values varied from 2.91×10^{-2} to 4.11×10^{-1} for children and 1.41×10^{-2} to 2.02×10^{-1} for adults. The HI values were generally < 1 and signified that there is no adverse non-carcinogenic risk from ingestion of these edible vegetable oils. The input of each OPP congener to the HI was in the order of EPN (O-ethyl O-4-nitrophenyl phenylphosphonothioate) > pirimiphos methyl > quinalphos > chloropyrifos methyl > azinphos ethyl > diazinone > chloropyrifos.

Table 2: Hazard index of OPPs from consumption of the edible vegetable oils

Group	EVOs	Diazinone	Chloropyriphos methyl	Chlorpyrifos	Pirimiphos Methyl	Quinalphos	Azinphos Ethyl	EPN	HI
Children	Branded Groundnut Oil	0.0	$1.30×10^{-3}$	0.0	$2.10×10^{-3}$	$3.15×10^{-3}$	$6.15×10^{-4}$	$2.04×10^{-1}$	**$2.11×10^{-1}$**
	Unbranded Groundnut Oil	$2.22×10^{-4}$	$2.66×10^{-3}$	0.0	$2.90×10^{-3}$	$4.24×10^{-3}$	$2.88×10^{-3}$	$3.98×10^{-1}$	**$4.11×10^{-1}$**
	Branded Palm Oil	0.0	$7.97×10^{-4}$	0.0	$3.19×10^{-4}$	$4.28×10^{-4}$	$3.43×10^{-4}$	$2.72×10^{-2}$	**$2.91×10^{-2}$**
	Unbranded Palm Oil	0.0	$3.52×10^{-3}$	0.0	$5.11×10^{-3}$	$3.54×10^{-3}$	$4.73×10^{-4}$	$2.72×10^{-2}$	**$3.98×10^{-2}$**
	Branded Soyabean Oil	$1.89×10^{-3}$	$1.26×10^{-3}$	0.0	$1.33×10^{-3}$	$2.37×10^{-3}$	$1.13×10^{-3}$	$1.61×10^{-1}$	**$1.69×10^{-1}$**
	Unbranded Soyabean Oil	$2.44×10^{-3}$	$1.85×10^{-3}$	$1.96×10^{-3}$	$2.32×10^{-3}$	0.0	$2.96×10^{-3}$	$2.00×10^{-1}$	**$2.12×10^{-1}$**
Adults	Branded Groundnut Oil	0.0	$3.26×10^{-4}$	0.0	$5.26×10^{-4}$	$7.87×10^{-4}$	$1.54×10^{-4}$	$1.02×10^{-1}$	**$1.04×10^{-1}$**
	Unbranded Groundnut Oil	$5.55×10^{-5}$	$6.66×10^{-4}$	0.0	$7.25×10^{-4}$	$1.06×10^{-3}$	$7.21×10^{-4}$	$1.99×10^{-1}$	**$2.02×10^{-1}$**
	Branded Palm Oil	0.0	$1.99×10^{-4}$	0.0	$7.99×10^{-5}$	$1.07×10^{-4}$	$8.58×10^{-5}$	$1.36×10^{-2}$	**$1.41×10^{-2}$**
	Unbranded Palm Oil	0.0	$8.79×10^{-4}$	0.0	$1.28×10^{-3}$	$8.84×10^{-4}$	$1.18×10^{-4}$	$1.36×10^{-2}$	**$1.68×10^{-2}$**
	Branded Soyabean Oil	$4.72×10^{-4}$	$3.16×10^{-4}$	0.0	$3.33×10^{-4}$	$5.93×10^{-4}$	$2.83×10^{-4}$	$8.07×10^{-2}$	**$8.26×10^{-2}$**
	Unbranded Soyabean Oil	$6.11×10^{-4}$	$4.62×10^{-4}$	$4.91×10^{-4}$	$5.79×10^{-4}$	0.0	$7.40×10^{-4}$	$1.00×10^{-1}$	**$1.03×10^{-1}$**

Conclusion

The status of organophosphate pesticides (OPPs) in edible vegetable oils was investigated in this study. The result showed that all the edible vegetable oils sold in Warri market investigated were contaminated with OPPs but at concentrations lower than the maximum residue limit (MRL) of 10 ng g^{-1} stipulated by the Food and Agriculture Organization/World Health Organization (FAO/WHO). The unbranded edible vegetable oils have higher concentrations of OPPs than the branded ones. The possible risk assessed using hazard index signified that there is no adverse carcinogenic risk from consumption of these vegetable oils. We hereby recommend that the appropriate regulatory agency should establish MRLs for OPPs in foodstuffs in Nigeria and ensure strict compliance with the MRLs of contaminants in foodstuff.

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