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A Comparative Analysis on the Use of DDT, Dichlorvos, Endosulfan, Heptachlor and Chlorpyrifos in the Preservation of Smoked-Dried Fish in Ogwashi-Uku, Delta State

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Abstract

Naturally, fish is highly perishable, thus its careful handling, prompt preservation and processing are vital factors to consider in order to avoid economic and nutritional losses in fish trade. This study was carried out to ascertain the presence and the quantity of three organochlorine pesticides (Endosulfan, DDT and Heptachlor) and two organophosphate pesticides (Dichorvos and Chlorpyrifos) used in the preservation of two different smoke-dried fish samples namely; Catfish (*Clarias gariepinus*) and Bonga shad fish (*Ethmalosa fimbriata*) sold in Ogwashi-Uku market, Aniocha South, Delta State, Nigeria. Gas chromatography (GC – MS) technique was used to determine the pesticide residues in both samples and the results showed positive identification of these pesticides in the samples ranging from 0.4212ppm-0.1760ppm (Endosulfan), 1.2317ppm-0.7270ppm (DDT), 1.1265ppm-0.7019ppm (Heptachlor), 0.0536ppm-0.4274ppm (Chlorpyrifos) with the exception of Dichlorvos which was below determination level (BDL) in the catfish sample but present at 0.0415ppm value in the Bonga shad sample. This study also found that there are possible health implications such as; immune suppression, hormone disruption, diminished intelligence, reproductive abnormalities and cancer in humans.

Keywords: *Pesticides, catfish, Clarias gariepinus, Ethmalosa fimbriata, health effect, chromatography*

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Introduction

According to FAO/WHO (1996) [1] on Pesticides and Management Act No. 528, pesticides can be defined as a substance or mixture of substances intended for preventing, destroying, repelling or reducing the destructive effect of pest and/or substance or mixture of substance intended for use as a plant regulator, defoliant, desiccant or wood preservative. Pesticides actually cover a range of chemical compound including insecticides, fungicides, herbicides, rodenticides, molluscicides, nematicides, plant growth regulators, etc. and consist of a wide variety of chemicals and different chemical structures. Consequently, they tend to have large differences in their mode of action, uptake, biotransformation and elimination. The chemical classes of pesticides include organochlorine compounds, carbonates, organophosphates and chlorophenoxy compounds.

Fish is of great importance on the diet of Nigerians [2]. It is made up of nutrient needed for complementing infants, adults and even animals' diet [3]. Fish is very perishable because it provides support for the multiplication and growth of microorganisms after death [4, 5]. Once spoilage starts in fish, its originally nutritional value, odour, flavor, texture, colour and composition change. Spoilage starts the moment fish is caught.

It has been observed that about 60-70% of fresh fish harvested in Nigeria waters is further processed into cure and smoked-dried products. Preservation, packaging, transportation and storage methods place the practitioners in the cure fish trade in desperate position as a result of its deterioration changes caused by insects' attacks and spoilage. Some fish mongers and processors resort to the use of unapproved chemicals such as Lindane (Gammalin 20R), Gardona, Malathion, and "a locally mixed pesticdes" to prevent insects and mould infestation [6]. The insects associated with spoilage of fish includes; *Chrysomyia marginalis* (blowfly), and *Dermestes spp* (beetle) etc. *Dermestes spp* are the most important insect pests of dried fish. They invade fish from the earliest stages of drying but unlike flies, will continue to infest and breed in the dried fish product. Losses of about 50% in dried fish have been estimated due to the destruction by *Dermestes spp* on unsalted fish in Nigeria.

Control measures against infestation on dried and smoked fish include the injudicious use of harmful chemical or pesticide such as Dichlorvos, DDT, Endosulfan, Heptachlor and Chlorpyrifos to keep away insects and other pests [7,8], leading to the application of higher pesticide doses [9].

Numerous studies on both human and laboratory animals provide strong evidence of the toxic potential of exposure to pesticides residues [10-12]. Continuous ingestion of pesticides by man lead to symptoms such as headache, nausea, vomiting, irritation of the skin and eyes. Therefore, risk characterization of pesticides in

environmental samples, foods and dietary products is an important step and a vital tool in the assessment of food safety risk [13]. The toxic effects of pesticides to man and the environment is a major issue that gives rise to concerns at local, national and global scales and is the basis for the control, monitoring, and prohibition of pesticides in food [14]. The Nigerian government has prohibited the utilization of chemicals in the production and preservation of fish. However, some fishermen continue to use these chemicals. These include DDT, a locally mixed pesticide dubbed "Otapiapia", which has active components of aluminum phosphide and dichlorvos [15]. Thus, it has become expedient to regulate the use of pesticides in order to ensure the minimum residue on food which can be considered safe for human consumption and for the environment.

This research aim at determining the presence of three organochlorine pesticides (Endosulfan, DDT and Heptachlor) and two organophosphate pesticides (Dichorvos and Chlorpyrifos) used in the preservation of two different smoke-dried fish samples namely; Catfish (*Clarias gariepinus*) and Bonga shad fish (*Ethmalosa fimbriata*) sold in Ogwashi-Uku market, Aniocha South, Delta State, Nigeria and as its possible health implications.

Materials and Methods

2.1 Sampling

Two different species of fish was collected for this analysis namely; Bonga shad (*E. fimbriata*) and catfish (*C. gariepinus*). Different sample size for each species were randomly purchased from the market. The fish samples were collected into plain polyethylene bags and labelled accordingly. The samples were taken to spring board laboratory for proper analysis. The samples were analyzed according to AOAC 1990.

2.2 Sample Preparation and Analysis

Extraction of PCB from samples

A ten gram grounded sample was weighed and quantitatively transferred into a 500 mL beaker. Six gram (6g) sodium sulphate was added and extracted using 300ml n hexane. The filtrate was concentrated. A 1ml of filtered residue was dissolved in 50ml of chloroform and transferred to a 100ml volumetric flask and diluted to the mark.

Most of the chloroform was evaporated at room temperature. Following this, 1 ml of the reagent {20 vol% benzene and 55 vol% methanol} was added to the solution. It was then sealed and subjected to a 40ºC water bath for a duration of 10 minutes. After the heating step, the organic sample was extracted using hexane and water, resulting in a final mixture of the reagent, hexane, and water in a 1:1:1 proportion (i.e., 1 ml of hexane and 1 ml of water were each added to the reaction mixture). This mixture was vigorously shaken by hand for a period of 2 minutes. In cases where a stable emulsion formed, it was subsequently broken through centrifugation. To prepare for injection, approximately half of the top hexane phase was carefully transferred to a small test tube, ensuring the extraction of only the organic layer. Injection was carried out with great caution, avoiding direct injection from the reaction vial to mitigate the risk of injecting water, which could potentially harm the GC column.

Gas Chromatographic conditions for PCB determination

The final extracts were analyzed by Gas Chromatograph-Buck M910 scientific gas chromatography equipped with Electron capture detector that allowed the detection of contaminants even at trace level concentrations (in the lower µg/g and µg/kg range) from the matrix to which other detectors do not respond. The GC conditions used for the analysis were capillary column HP 88 capillary column (100m x 0.25µm film thickness,) CA, USA.

The injector and detector temperature were set at 250 ºC and 290ºC respectively. The oven temperature was programmed as follows: 110 ºC held for 10 min, ramp at 10 ºC/ min to 200 ºC, held for 5 min, and finally ramp at 10 ºC/ min to 320 ºC. Helium was used as carrier gas at a flow rate of 1.0 mL/ min and detector make-up gas of 29 ml min-1. The injection volume of the GC was 8.0 µL. The total run time for a sample was 48 min.

Quantification of PCB residues

The residue levels of PCB were quantitatively determined by the external standard method using peak area. Measurement was carried out within the linear range of the detector. The peak areas whose retention times coincided with the standards were extrapolated on their corresponding calibration curves to obtain the concentration

Preparation of standard

Ten micromilliliter (10µl) of a standard was injected in the chromatography and the retention time compared with retention time of standard.

2.5. Statistical Analysis

The results of the analysis were statistically analyzed using mean values.

Results and Discussion

Table 1 shows the mean values for both fish samples; Catfish (*C. gariepinus)* and Bonga shad fish (*E. fimbriata*) while figures 1 and 2 shows percentage (%) of Catfish (*C. gariepinus)* and Bonga shad fish (*E. fimbriata*) respectively.

Table 1: Mean values of Pesticide residues present in both fish samples

Parameters	Catfish (*C. gariepinus)*, ppm	Bonga shad fish (*E. fimbriata)*, ppm
Endosulfan	0.4212	0.1760
DDT	1.2317	0.7270
Heptachlor	1.1265	0.7019
Chlorpyrifos	0.0536	0.4274
Dichlorvos	Below determination level (BDL)	0.0415

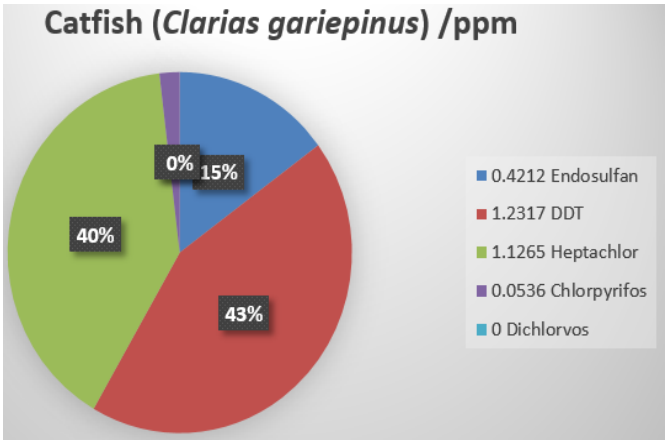

Figure 1: Bar chart showing the percentage of Catfish (*Clarias gariepinus*)

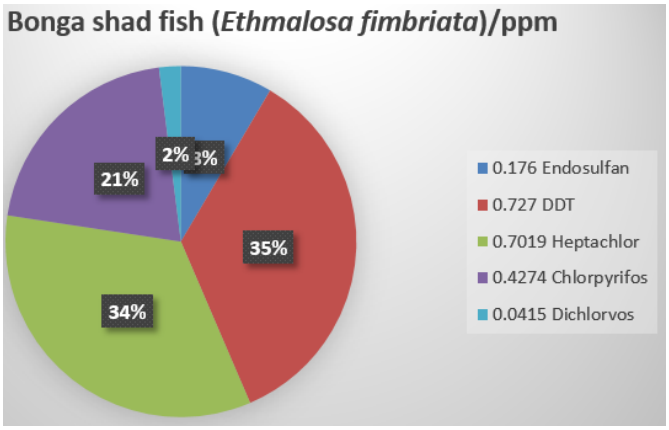

Figure 2: Bar chart showing the percentage of Bonga shad fish (*Ethmalosa fimbriata*)

Endosulfan residue concentration in catfish sample is $0.4212ppm$ while that of Bonga shad fish is $0.1760ppm$. The results from this study are in agreement with those of Adeyemi et al. (2008) [16]. They reported lower levels of endosulfan residues in Lagos lagoon. The concentrations of endosulfan in all the cured fish were far higher than the 0.0001 µg/g maximum residue limit (MRL) set by WHO and 0.6×10^{-5} µg/g Acceptable Daily Intake value (ADI) established by FAO (2009). Endosulfan is a xenoestrogen and endocrine disruptor, causing developmental damage in humans and animals. It is neurotoxic in mammals and insects and is extremely deadly to aquatic organisms and it has bioaccumulating consequences on fish [19].

The concentrations of *DDT* residues in the smoked-dried-fish samples were $1.2317ppm$ for catfish and $0.7270ppm$ for that of Bonga shad fish. The results from this study is not in line with the results reported by Musa et al. (2010) who detected 4.220±0.599 µg/g for *Gymnarchus niloticus*, 3.323±0.395 µg/g for Catfish and 2.844±0.68 µg/g for Tilapia from South-East Nigeria. The high concentration and even distribution of DDT are suggestive of the fact that DDT is used for preservation fish by fish processors during and after curing.

Dichlorvos also known as DDVP (2,2-dichlorovinyl dimethyl phosphate) is an organophosphate pesticide [21]. It is traded under name such as DDVP, Sniper etc. [22]. Dichlorvos has been used in fish farming to eradicate crustacean ectoparasites [23]. Its toxic effects when accumulated in the body system include muscle cramps, muscle weakness, drowsiness, fatigue, mental confusion, headache, diarrhea, convulsion excess bronchial secretion and even death [24]. The concentrations of Dichlorvos residues in the smoked-dried-fish samples shows that it was *below detectable limit* in catfish and $0.0415ppm$ for that of Bonga shad fish. The concentration reported in Bonga shad fish in this study can be harmful to human if it accumulates over time.

Chlorpyrifos residues in smoked-dried fish samples were *0.0536ppm* for catfish and *0.4274ppm* for Bonga shad fish. Chlorpyrifos has been used as a pesticide since 1995 in both agricultural and non-agricultural areas, however, in 2000 all outdoor residential and outdoor non-residential uses were eliminated [21]. Therefore, Chlorpyrifos application to food commodities results in food being considered adulterated. Chlorpyrifos can cause cholinesterase inhibition in human at high enough doses that leads to over stimulation of the nervous system causing nausea, dizziness, confusion, and at very high exposure (e.g accident or major spills), respiratory par alysis and death.

CONCLUSION

This study provides important information on pesticides residue levels in smoked-dried fish samples (Catfish and Bonga shad fish) collected from local markets in Ogwashi-Uku and it was revealed that pesticide residue levels were above maximum residue limit (MRL) and dietary intake and could be an important process of transferring residues to humans. Traditionally, fish is vigorously washed with hot water before cooking but such practice does not completely remove the pesticides from the tissues and dietary intake could still be significant; organochlorine pesticides are lip soluble and can only be drained out with lots fats and oils. In addition to this, the most pertinent concern however, is that these smoked-dried fishes are sometimes eaten without having to cook them, because they have been smoked and are edible. Since organochlorine (Endosulfan, DDT, Heptachlor) synthetic chemicals and organophosphates (Chlorpyrifos, Dichlorvos) could cause danger to human health, these pesticides should not be used in the preservation of smoked-dried fishes in Nigeria. Furthermore, government should ensure that mechanisms are put in place to effectively ensure that all banned chemicals are not brought into the country and those already in the country, and on display with complete impunity in our markets, should be removed and disposed off correctly.

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