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POLYCYCLIC AROMATIC HYDROCARBONS AND NUTRIENT COMPOSITION OF *Clarias gariepinus* AND *Oreochromis niloticus* PROCESSED WITH DIFFERENT FUEL TYPES

AHMED M.T.[1], A.B. DAUDA[1*], A.B. ABDULLAHI[2], A.K. SAIDU[1], A.A. AYELOJA[3], I.A. ABDULHAKEEM[4], A.R. ABDULAZEEZ[1] *AND* A.H. OLADELE[1]

[1]*Department of Fisheries and Aquaculture, Federal University Dutsin-Ma, PMB 5001 Dutsin-Ma, Katsina State, Nigeria*

[2]*Department of Animal Science, Federal University of Kashere, PMB 0182, Gombe State, Nigeria*

[3]*Department of Fisheries, Aquaculture and Wildlife, University of Abuja, PMB 6001, Abuja, Nigeria*

[4]*Department of Zoology, Histology and Cytology, Al-Farabi Kazakh National University, Kazakhstan*

*Corresponding author: ; +2348062085120

ABSTRACT

This study evaluated the quality and safety of two fish species, Oreochromis niloticus and Clarias gariepinus, processed using various fuel types: firewood (Anogeissus leiocarpus), maize cob, charcoal, and a solar tent dryer. A total of 80 samples, comprising 40 Oreochromis niloticus and 40 Clarias gariepinus, were purchased from fishermen at the Zobe reservoir in Dutsin-Ma, Katsina State, Nigeria. The fish samples were processed, smoke-dried, and analyzed for polycyclic aromatic hydrocarbons (PAHs), proximate composition, free fatty acids (FFA), and peroxide value (PV). Proximate composition was assessed at week 0 and after 12 weeks of storage. Twenty PAHs were identified; significant differences (P < 0.05) were observed in naphthalene, benzo(k)fluoranthene, and benzo(a)pyrene, with solar-dried fish showing unexpectedly elevated levels. Initial proximate composition (week 0) did not differ significantly among the treatments except for ash content, which was significantly higher in maize-cob-smoked fish compared to solar-tent-dried fish, with the highest individual value (15.05±1.65%) observed in charcoal-smoked O. niloticus. After 12 weeks of storage, significant differences emerged between the two fish species for all parameters except crude lipid. Moisture, crude lipid, and crude protein contents also varied significantly among the fuel types. Free fatty acids (FFA) ranged from 0.48 mg/g in solar-dried O. niloticus to 1.08 mg/g in maize-cob-smoked C. gariepinus, and were significantly higher in C. gariepinus. The peroxide value (PV) ranged between 2.16 meq/kg in charcoal-smoked O. niloticus and 3.61 meq/kg in solar-dried C. gariepinus, but was not influenced by fuel type. The results revealed that both fish species and fuel types significantly influenced the nutritional profile and contaminant accumulation of processed fish; while nutritional levels remain high, the elevated PAH contents pose critical food safety concerns.

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Keywords: Polycyclic aromatic hydrocarbons (PAHs), smoking fuels, proximate composition, free fatty acids, peroxide value, *Oreochromis niloticus*, *Clarias gariepinus*

INTRODUCTION

Fish is considered an excellent source of high-quality protein that can supplement or replace terrestrial meat, particularly given the rapid growth of the aquaculture industry in recent years (Abdul Kari *et al.,* 2020). It contains essential micro- and macro-nutrients, including high levels of polyunsaturated fatty acids (PUFAs) which help reduce cardiovascular diseases in humans (Mishra, 2020). However, fish is highly perishable and prone to rapid deterioration, rancidity, and microbial spoilage post-harvest. Therefore, processing and preservation are required to extend its shelf-life for human consumption (Wang & Zheng, 2025). Various preservation techniques are traditionally or industrially employed to curb spoilage and add value to products, including chilling, freezing, salting, canning, drying, and smoking (Rabiepour *et al.,* 2024; Ariyamuthu *et al.,* 2022). Smoking is the process of cooking, flavouring, and preserving food by exposing it to smoke from burning or smouldering material; most often wood. Smoking has been used for the preservation of food for centuries (Huong, 2014). The smoke is generated through incomplete combustion of wood and contains phenolic compounds, acids and carbonyls and the smoky flavor is primarily due to the volatile phenolic compounds. The relative concentration of phenolic compounds depends on the nature of the wood used in the smoking process (Nizio *et al*., 2023). The solar tent dryer, as an alternative drying system, does not produce smoke but works through evaporative drying using the greenhouse principle, when set up in the sun, solar energy passes through the transparent polythene but gets trapped within it thereby raising the internal temperature that reduce the fish moisture content. However, while smoking could improve the shelf-life of fish, it could also be of negative impacts as some of the chemicals in smoke including polycyclic aromatic compounds (PAHs) have been implicated as carcinogens and affect the fish consumers and processors that are exposed to smoke (Dauda *et al*., 2020). Fish processors use wood and other fuel types mostly based on convenience and availability and not necessarily on the quality it can confer on the fish and safety of both the processors and consumers (Dauda *et al*., 2020). It is therefore imperative to assess the level of PAHs and proximate compositions of smoked food products and comparing the experimental result obtained with the recommended limits so as to ensure consumer safety and healthy living since the compounds when present at lower or greater concentrations, pose hazard to human health. The current study therefore assessed the product quality and safety of traditional smoking method using different fuel types such as firewood, maize cob and charcoal in comparison with solar tent dryer for processing of African catfish (*Clarias gariepinus*) and Nile tilapia (*Oreochromis niloticus).*

MATERIALS AND METHODS

Study Area

The experiment was carried out at the fish processing and preservation unit of the Department of Fisheries and Aquaculture, Federal University Dutsin-Ma, Katsina State, Nigeria.

Experimental Design and Preparation

The study employed a 2 by 4 randomized complete block design (RCBD), comprising of two fish species, *Clarias gariepinus* and *Oreochromis niloticus* as experimental units and four fuel types; charcoal, wood, maize cob and sunlight as treatments

Fuel Types

African birch (*Anogeissus leiocarpus*) commonly called "Marke" in Hausa language was used as firewood while wood log of the same tree was used for the production of charcoal which were both purchased from Dutsin-Ma Wednesday market. Maize cob was obtained from in and around Dutsin-Ma town at no cost, materials used for the construction of solar tent dryer were also purchased from the Wednesday market in Dutsin-Ma.

Fish Sample Collection

Eighty (80) pieces of fresh fish sample comprises of forty (40) pieces of *Clarias gariepinus* (length ranges between 30-33cm each and an average weight of 600g) and forty (40) pieces of *Oreochromis niloticus* (length range of 23-26cm, with an average weight of 500g) were purchased from the artisanal fishermen at the Zobe reservoir in Garhi landing site in Dutsin-Ma Local Government Area of Katsina State. Sensitive weighing balance (Sarut Pro SPU 401 OHAUS CORPORATION, USA) was used in weighing the fish at the landing site. The fish were then chilled on ice and immediately transported to the fish processing unit of the Department of Fisheries and Aquaculture, Federal University Dutsin-Ma.

Sample Preparation

Oreochromis niloticus was descaled and both fish species were eviscerated. After evisceration, the fish samples were washed thoroughly with potable water before immersed in 5% brine solution for thirty (30) minutes, drained and air dried for one hour before smoking and drying. *Clarias gariepinus* was coiled into a horse shoes shape by pushing the tail inside the mouth with clean skewering sticks for easy and uniform smoking.

Smoking and Drying Process

The smoking took place at the fish processing unit. Three modified metal drum kilns and a solar tent dryer were used for fish processing, the drum kilns were built using a 400L metal drum measuring 90 cm in length and 58 cm in diameter following the methods described by Abdullahi *et al.* (2024). The solar tent dryer was constructed following the design of Olokor *et al*. (2007), with a slight modification. Five pieces of straight wooden poles each measuring 180cm; two of these poles were nailed at one end and the two other ends were dug (10cm) into the ground at 160cm apart. The same was done for the third and fourth wooden poles which were dug in an opposite direction at a distance of 194cm apart. The fifth wooden frame was placed across the two pairs of wooden poles and fastened at both ends to form a tent-like structure. About 10 yards of transparent polythene was sown into shape of the wooden frame work that was placed over the wooden framework, on one of the longer side of the polythene, a mosquito net was attached to it; this opening served as an air inlet into the solar tent, at the extreme narrow tops of the triangular part of the tent, opening of 15cm x 15cm was made and also screened with mosquito net to serve as the outlet of the hot air from the dryer on the opposite side of the net. A ½ meter zip length was sewn into the middle of the polythene sheet to serve as an access opening into the tent for the processor to handle and inspect the fish on the rack inside the tent. A fibre rope was passed around the base of the tent for tying the polythene tent firmly on the wooden structure, to prevent wind from blowing the sheet away and also to prevent insects from escaping into the tent. A considerable number of rocks, about 30 pieces with an average weight of 20kg and these rocks were painted black and they were stacked within the base of the wooden frame work. These painted rocks served as capacitor by absorbing, retaining and releasing radiant energy needed for the drying fish. Drying rack is a wire mesh framed with wood (70x150cm) which was suspended by two ropes of 150cm each from the wooden frame. Before smoking, 40kg of each fuel (firewood, maize cob and charcoal) were weighed out. Each sample (*Clarias gariepinus* and *Oreochromis niloticus*) were divided into four equal parts of 10 pieces each. The fire was lit in the combustion chamber and solar tent dryer was also set 30 minutes before the fish were laid on the smoking and drying racks. The fish were first subjected to direct hot smoking in order to ensure that they are thoroughly smoked and evenly dried for the first 1hour and then to a lower temperature for 8 to 10 hours by constantly adjusting the burning fuel to regulate the heat and ensure even smoking. The solar-dried fish were carefully laid on the rack inside the dryer and left till they are full dried. After smoking and drying, the fish were allowed to cool and then were packaged in perforated cartons and stored at room temperature (Abolagba *et al*., 2011).

Polycyclic Aromatic Hydrocarbons (PAHs)

The extraction of PAHs from the samples was based on the solid-liquid extraction procedure as described by Ubwa *et al*. (2015). While the analysis was carried out using the GC/MS method as described by Nnaji & Ekwe (2018) using an Agilent 7890 Gas Chromatograph outfitted with an auto sampler and connected to an Agilent 5975 Mass Spectrometric Detector (MSD).

Proximate Compositions

The proximate compositions of the smoked and dried fishes were carried out according to the Association of Official Analytical Chemist method (AOAC, 2016) to determine the percentage moisture, Ash, crude protein, carbohydrate and crude fat. The analysis was carried out immediately after smoking and after twelve weeks of the storage period.

Peroxide Value

Two grams (2.0g) of the oil was weighed into a clean dry flask and 22 ml of a mixture of 12 ml of chloroform and 10 ml of acetic acid was added. This was followed by the addition of 0.5 ml of potassium iodide. The flask was covered and allowed to stay with constant shaking for 1 minute. After which 30 ml of distilled water was then added and titrated against 0.1 M of sodium thiosulphate ($Na_2S_2O_3$) solution until an initial yellow color disappeared and a faint blue color appeared. 0.5 ml of starch indicator was added quickly with continuous titration until there was a sudden disappearance of the blue color which signifies the end point. The peroxide value was obtained by the use of the following expression;

$$Peroxide\ Value\ (mERqH2O2/100g) = \frac{(S - B) \times M \times 1000}{W \times 100} \qquad (1)$$

Where: S = sample titre value (cm), B = Blank titre value (cm), M = [illegible] cm), 1000 = Conversi[illegible] nple

Free fatty acid Analysis

Free fatty acid methyl esters (FAME) were prepared by transesterification using trimethylsulfonium hydroxide (SRPS EN ISO 5509:2000 procedure). The GC instrument Shimadzu 2010 (Kyoto, Japan) used for FAME determination was equipped with a split/splitless injector, fused silica cyanopropyl HP-88 column (length 100 m, i.d. 0.25 mm, film thickness 0.20 µm, J&W Scientific, USA), and flame ionization detector and workstation (Shimadzu GC Solution ver. 2.3). The carrier gas was nitrogen at a flow rate of 1.33 ml/min and injector split ratio of 1:50. Injected volume was 1 µl and analysis lasted 50.5 min. The chromatographic peaks in the samples were identified by comparing relative retention times of FAME peaks with peaks in a Supelco 37 Component FAME mix standard (Supelco, Bellefonte, USA). The contents of fatty acids in the fish muscles are expressed as the percentage of total fatty acids.

Statistical analysis

Data obtained for PAHs concentration and proximate composition were presented using (means and standard deviation). After testing of homogeneity and normality, they were subjected to two-way analysis of variance. Where the differences are significant at 95% confidence interval, Duncan Multiple Range Test (DMRT) was used as follow-up test to separate the means. All analysis was done using IBM SPSS version 22.

RESULTS AND DISCUSSIONS

Polycyclic Aromatic Hydrocarbons (PAHs)

The results of Polycyclic Aromatic Hydrocarbons (PAHs) content in fish processed with different fuel types are shown in Table 1. Twenty PAHs were detected but they were not significantly different among the fuel types and between the fish species except Naphthalene, Benzo(k)Fluoranthene and Benzo(a)pyrene. For Naphthalene, 0.88±0.25mg/Kg was found to be the highest value detected in *O. niloticus* processed with firewood while the least was found to be below detection level in *C. gariepinus* processed with maize cob. There was significant difference ($P<0.05$) due to fuel types, with fish smoked with firewood and solar-dried having higher naphthalene content. Highest amount of Benzo(k)fluoranthene (0.41±0.13mg/kg) was detected in *C. gariepinus* processed with Solar tent dryer but it was not detected in *O. niloticus* processed with maize cob, solar dried and *C. gariepinus* processed with firewood and maize cob. Benzo(k)fluoranthene was significantly higher ($P<0.05$) in *C. gariepinus* compared to *O. niloticus*. Among the fuel types, it was significantly higher in solar-tent dried fish compared to others. Benzo(a)pyrene was found to be highest in *C. gariepinus* solar-dried (0.49±0.13mg/kg) while the least were below detection level (ND) in *O. niloticus* processed with maize cob, charcoal, solar-dried and *C. gariepinus* processed with firewood and maize cob. *C. gariepinus* had significantly higher ($P<0.05$) Benzo(a)pyrene compared to *O. niloticus,* while among the fuel types solar-dried fish had significantly higher Benzo(a)pyrene compared to maize cob smoked fish.

Proximate Composition

The results of the initial and final proximate composition of the two fish species processed with different fuel types are shown in Tables 2 and 3. In the initial proximate composition, moisture, crude lipid and crude protein were neither different among the fuel types nor between the fish species ($P>0.05$). While Ash content was also similar between the fish species, it was different significantly ($P<0.05$) among the fuel types. The overall highest content of 15.05±1.65% was observed *O. niloticus* smoked with charcoal. Comparatively, the fish smoked with maize cob showed significantly ($P<0.05$) higher ash than solar-dried fish. After 12 weeks of storage, the final proximate showed a different pattern with all the proximate parameters different between the two fish species except crude lipid. Moisture and carbohydrate were higher significantly in *O. niloticus* ($P<0.05$). While crude protein and ash were higher significantly ($P<0.05$) in *C. gariepinus*. Among the fuel types, ash and carbohydrate were not different significantly. Moisture was significantly lower ($P>0.05$) in charcoal smoked and solar-dried fish compared to other treatments. Crude lipid was higher significantly ($P<0.05$) in maize cob smoked fish compared to firewood and charcoal smoked. Overall highest crude protein of 47.72±0.77% was observed in *C. gariepinus* smoked with firewood, while comparatively, firewood smoked fish was significantly higher in crude protein than solar-dried.

Table 1: Polycyclic Aromatic Hydrocarbons (PAHs) content in fish processed with different fuel types

Parameters	Fish	Fuel type				Statistics		Interaction
		Firewood (mg/Kg)	Maize cob (mg/Kg)	Charcoal (mg/Kg)	Solar dryer (mg/Kg)	Fish	Fuel Types	Fish*Fuel Types
Benzene, 1,2,3-trimethyl-	*O. niloticus*	0.25±0.21	0.17±0.24	ND	0.50±0.16	0.305	0.054	0.057
	C. gariepinus	0.82±0.06	ND	0.32±0.45	0.23±0.04			
Naphthalene	*O. niloticus*	0.88±0.25^{a}	0.11±0.16^{b}	0.11±0.16^{b}	0.55±0.05^{a}	0.293	0.038	0.396
	C. gariepinus	0.33±0.44	ND	0.17±0.06	0.56±0.49			
2-Methylnaphthalene	*O. niloticus*	0.55±0.76	0.10±0.13	ND	0.54±0.76	0.424	0.385	0.855
	C. gariepinus	0.07±0.04	ND	ND	0.41±0.45			
Acenaphthylene	*O. niloticus*	1.25±1.07	0.17±0.06	0.17±0.23	0.94±1.32	0.248	0.484	0.628
	C. gariepinus	0.26±0.28	0.16±0.22	0.20±0.28	0.31±0.33			
Acenaphthene	*O. niloticus*	0.07±0.04	0.22±0.15	0.16±0.23	ND	0.431	0.969	0.593
	C. gariepinus	0.17±0.20	0.11±0.16	0.21±0.29	0.30±0.32			
Fluorene	*O. niloticus*	0.12±0.11	0.20±0.18	0.01±0.01	ND	0.564	0.621	0.612
	C. gariepinus	0.12±0.01	0.12±0.16	0.16±0.22	0.08±0.08			
Anthracene	*O. niloticus*	0.70±0.48	1.54±1.79	0.05±0.07	0.99±1.40	0.101	0.617	0.683
	C. gariepinus	ND	0.06±0.08	0.01±0.02	0.15±0.07			
Phenanthrene	*O. niloticus*	0.60±0.85	ND	0.08±0.11	ND	0.604	0.609	0.388
	C. gariepinus	ND	ND	0.21±0.29	0.13±0.15			
Fluoranthene	*O. niloticus*	0.51±0.72	ND	0.06±0.08	ND	0.402	0.531	0.419
	C. gariepinus	ND	ND	0.03±0.04	0.08±0.11			
Pyrene	*O. niloticus*	0.20±0.28	ND	0.01±0.01	ND	0.664	0.430	0.166
	C. gariepinus	ND	0.03±0.04	ND	0.30±0.27			
Benz(a)anthracene	*O. niloticus*	0.18±0.25	0.17±0.23	0.29±0.08	ND	0.953	0.826	0.164
	C. gariepinus	ND	0.10±0.01	0.06±0.08	0.45±0.46			
Chrysene	*O. niloticus*	0.01±0.01	0.16±0.23	0.12±0.01	ND	0.890	0.376	0.155
	C. gariepinus	ND	0.07±0.06	0.02±0.02	0.02±0.07			
Benzo(b)fluoranthene	*O. niloticus*	0.03±0.04	ND	0.11±0.15	ND	0.254	0.089	0.031
	C. gariepinus	ND	ND	0.02±0.02	0.33±0.18			
Benzo(k)Fluoranthene	*O. niloticus*	0.04±0.06^{B,a}	ND^{a}	0.05±0.07^{a}	ND^{b}	0.016	0.007	0.003
	C. gariepinus	ND^{A},	ND	0.06±0.08	0.41±0.13			
Benzo(a)pyrene	*O. niloticus*	0.05±0.07^{B,ab}	ND^{a}	ND^{ab}	ND^{b}	0.016	0.032	0.019
	C. gariepinus	ND^{A}	ND	0.17±0.23	0.49±0.13			
Dibenz(g,h.i)anthracene	*O. niloticus*	0.96±1.34	0.11±0.15	ND	0.03±0.04	0.414	0.531	0.385
	C. gariepinus	0.01±0.01	ND	0.04±0.05	0.23±0.03			
Indeno(1,2,3-c,d)perylene	*O. niloticus*	1.13±1.10	0.07±0.08	0.14±0.12	0.01±0.01	0.337	0.289	0.160
	C. gariepinus	0.06±0.07	0.15±0.10	0.06±0.08	0.26±0.01			
Benzo(g,h,i)perylene	*O. niloticus*	1.02±1.31	0.08±0.07	0.29±0.09	0.04±0.06	0.451	0.650	0.365
	C. gariepinus	0.02±0.01	0.16±0.07	0.26±0.37	0.21±0.26			
O-Terphenyl	*O. niloticus*	ND	0.19±0.27	0.25±0.11	ND	0.205	0.208	0.461
	C. gariepinus	ND	0.11±0.16	0.0±10.01	ND			
Diben(a,h)anthracene	*O. niloticus*	ND	0.06±0.08	0.16±0.08	ND	0.754	0.081	0.873
	C. gariepinus	ND	0.08±0.06	0.10±0.14	ND			

Different letters as superscripts across the rows for each parameter indicate significant difference among the fuel types (P<0.05)
Different letters (upper case) as superscripts within the column for each parameter indicate significant difference between the fish species (P<0.05)

Table 2: Initial Proximate composition (week 0) of *Clarias gariepinus* and *Oreochromis niloticus* processed with different fuel types

Parameters (%)	Fish	Fuel types				Two-way ANOVA (Pvalue)		
		Firewood	Maize cob	Charcoal	Solar	Fish	Fuel Types	Fish*Fuel Types
Moisture	*O. niloticus*	10.63±1.12	11.20±2.71	8.74±0.55	11.30±1.58	0.516	0.448	0.930
	C. gariepinus	9.78±1.17	9.70±3.27	8.96±1.59	10.86±1.65			
Ash	*O. niloticus*	14.34±1.29^{ab}	14.59±2.35^{b}	15.05±1.65^{ab}	10.99±2.30^{a}	0.113	0.039	0.663
	C. gariepinus	13.16±1.03	14.21±1.09	11.83±1.84	9.90±0.97			
Crude Lipid	*O. niloticus*	17.89±2.55	17.74±1.87	17.47±3.15	17.24±1.00	0.165	0.986	0.984
	C. gariepinus	19.49±2.74	19.10±2.14	18.77±2.00	19.61±0.85			
Crude Protein	*O. niloticus*	38.36±1.80	35.79±0.65	33.48±5.42	33.22±8.73	0.473	0.531	0.889
	C. gariepinus	35.65±3.05	32.7±2.21	31.76±0.95	34.37±3.83			
Carbohydrate	*O. niloticus*	18.78±1.65	20.69±3.83	25.26±9.67	27.27±8.45	0.523	0.411	0.896
	C. gariepinus	21.93±7.99	24.21±4.29	28.69±0.48	25.27±5.37			

Different letters as superscripts across the rows for each parameter indicate significant difference among the fuel types (P<0.05)
Different letters (upper case) as superscripts within the column for each parameter indicate significant difference between the fish species (P<0.05)

Table 3: Final Proximate composition of *Clarias gariepinus* and *Oreochromis niloticus* processed with different fuel types

Parameters (%)	Fish	Fuel types				P-value		Interaction
		Firewood	Maize cob	Charcoal	Solar	Fish	Fuel Types	Fish*Fuel Types
Moisture	*O. niloticus*	8.76±0.10^{bA}	9.29±0.50^{b}	7.90±0.48^{a}	8.28±0.17^{b}	0.000	0.005	0.013
	C.gariepinus	7.63±0.42^{B}	6.46±0.16	5.90±0.62	7.71±0.12			
Ash	*O. niloticus*	3.22±0.57^{B}	3.33±0.21	3.07±0.18	3.23±0.08	0.003	0.758	0.761
	C.gariepinus	4.87±0.48^{A}	4.76±0.92	4.65±0.19	4.02±1.29			
Crude Lipid	*O. niloticus*	6.15±0.62^{a}	6.82±0.05^{b}	5.51±0.49^{a}	5.87±0.60^{ab}	0.368	0.035	0.234
	C.gariepinus	4.98±0.59	6.63±0.13	5.71±0.59	6.09±0.50			
Crude Protein	*O. niloticus*	37.65±1.17^{bB}	36.91±0.45^{ab}	36.91±0.30^{ab}	35.17±2.33^{a}	0.000	0.032	0.844
	C.gariepinus	47.72±0.77^{A}	46.87±0.08	47.02±1.28	43.94±1.66			
Carbohydrate	*O. niloticus*	44.23±1.12^{A}	43.67±0.79	46.62±0.85	47.46±3.18	0.000	0.088	0.952
	C.gariepinus	34.82±1.29^{B}	35.29±0.54	36.74±1.06	38.26±3.58			

Different letters as superscripts across the rows for each parameter indicate significant difference among the fuel types (P<0.05)
Different letters (upper case) as superscripts within the column for each parameter indicate significant difference between the fish species (P<0.05)

Free Fatty Acids (FFA)

The results of free fatty acid of *Clarias gariepinus* and *Oreochromis niloticus* processed with different fuel types were shown in Figure 1. The highest free fatty acids content, 1.08mg/g was observed in *C. gariepinus* smoked with maize cob while the least free fatty acids 0.48mg/g was recorded in *O. niloticus* in solar-dried, Free Fatty Acids was higher significantly (P<0.05) in *C. gariepinus* compared to *O. niloticus* and there was no significant difference (P>0.05) among the fuel types. Free fatty acids (FFAs) are very important lipid components, as the energy providers, and constituents of the membranes. High level of FFA signifies hydrolysis and rancidity.

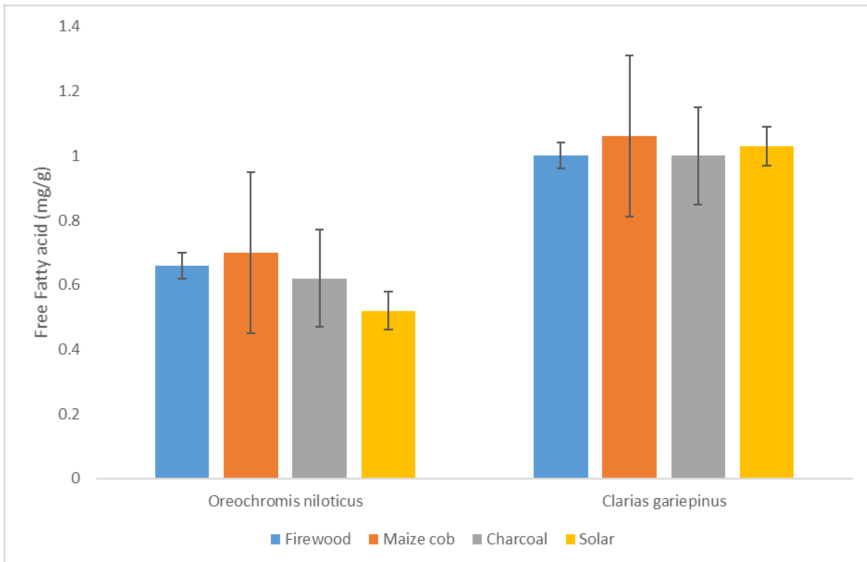

Fig. 1. Free Fatty Acid *of Clarias gariepinus* and *Oreochromis niloticus* processed with different fuel types
*Free fatty acid was higher significantly (P<0.05) in *C. gariepinus* compared to *O. niloticus*
* Free fatty acid was not different significantly (P>0.05) among the fuel types for any of the species

Peroxide Value (PV)

The results of Peroxide Value (PV) of *Oreochromis niloticus* and *Clarias gariepinus* smoked with different fuel types are shown in Figure 2. The highest peroxide value 3.61meq/kg was observed in *C. gariepinus* solar-dried, while the least peroxide value 2.16meq/kg was recorded in *O. niloticus* smoke-dried with charcoal. Peroxide value was higher significantly (P<0.05) in *C. gariepinus* compared to *O. niloticus* and there was no significant difference (P>0.05) among the fuel types.

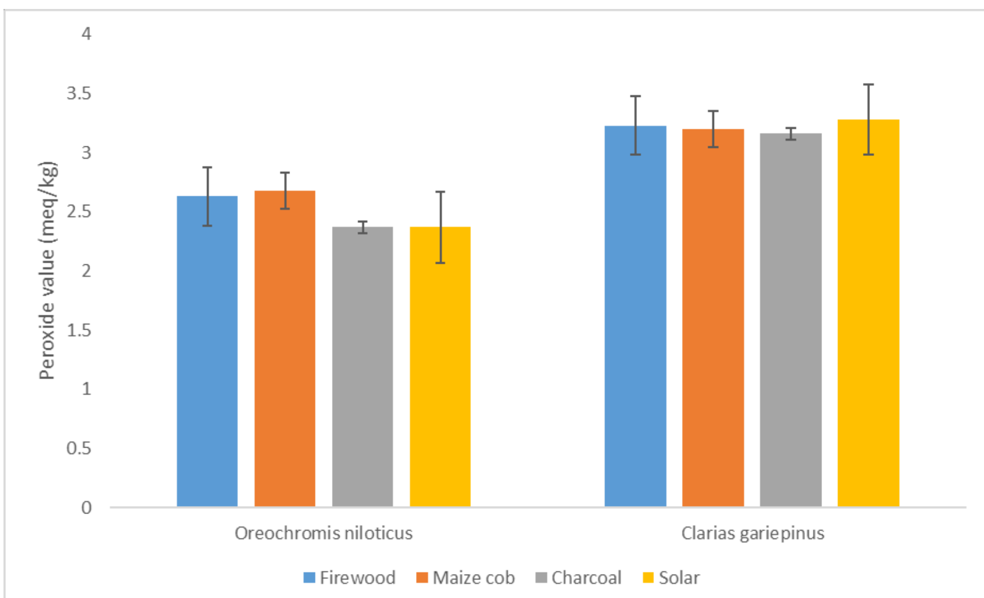

Fig. 2. Peroxide value of *Clarias gariepinus* and *Oreochromis niloticus* processed with different fuel types
*Peroxide value was higher significantly (P<0.05) in *C. gariepinus* compared to *O. niloticus*
*Peroxide value was not different significantly (P>0.05) among the fuel types.

DISCUSSIONS

Polycyclic Aromatic Hydrocarbons (PAHs)

The results also show that a total of twenty PAHs were found in varying concentrations in the two fish species that were processed using different fuel types, with naphthalene, benzo(k)fluoranthene and benzo(a)pyrene showing significant differences between them. The most prevalent PAHs identified include benzo(a)pyrene which is considered a human carcinogen by the International Agency on Research in Cancer (IARC), and was used to measure the carcinogenicity of carcinogenic PAHs in food. The concentrations of PAHs in this study can be attributed to the deposition of smoke particles onto the fish surface during processing, with partial migration into the muscle depending on the fuel type and processing method. This aligns with the findings in smoked Nile tilapia (*O. niloticus*) where the PAH levels were higher in firewood-smoked samples than oven-dried ones due to incomplete combustion and exposure to smoke (Ekwere *et al*., 2025). Although elevated level of some PAHs in solar-dried came as a surprise and this is likely attributed to the materials used in painting the stones and some other components of the solar tent dryer (Abdel-Shafy & Mansour 2016; Oriji *et al*., 2012).

For naphthalene, the highest concentration was in firewood-processed *O. niloticus* and absent in the maize cob-processed *C. gariepinus*, which was also consistent with the positions on fuels-specific PAHs. This is in agreement with the results obtained from smoked *Mormyrus caschive* and O. *niloticus*, where traditional smoking with wood fuels resulted in higher light PAHs such as naphthalene (0.5 mg/kg) as a result of the variability in combustion temperatures. In this study maize cob and charcoal give cleaner smoke probably due to lower temperatures, which reduces the formation of light PAHs (Borodi *et al*., 2022). However, these values are lower than those reported in smoked *Arius heudelotii* in from Senegal (1.2 mg/kg) where uncontrolled traditional smoking resulted to an increased Naphtalene deposition as reported by Diop *et al.* (2023), unlike this study where alternative fuels reduced PAHs concentration, the naphthalene in solar-dried *O. niloticus* likely came from materials used in the solar dryer.

Benzo(k)fluoranthene and benzo(a)pyrene showed highest concentrations in solar-dried *C. gariepinus*, which is unexpected for non-smoking but possibly from environmental contamination and material used in the dryer such as carbon black and paint used for some of the components (Savin *et al*., 2024; Abdel-Shafy & Mansour 2016). Despite higher values in one species, they were below detection level in *O. niloticus*, suggesting species-specific absorption of PAHs. These findings partially align with elevated heavy PAHs in traditionally smoked fish exceeding EU limits, as the higher levels in solar-dried *C. gariepinus* may reflect species-specific lipid content facilitating PAHs retention or migration, though the unexpected pattern in solar drying suggests influences beyond traditional smoking, such as pre-processing contamination (Diop *et al.*, 2023). However, the absence in some treatments (e.g., maize cob), is also consistent with the fact that controlled smoking reduces heavy PAHs in agreement with the differences between fuel-type observed here where charcoal and maize cob inhibit high-temperature pyrolysis (Borodi *et al*., 2022).

The PAH4 index (benz(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(a)pyrene) components were detected but not different among the treatments except benzo(a)pyrene and related compounds detailed for significant differences. Compared to literature, the summed ∑PAH4 here (0.49 mg/kg from available components) is higher than in oven-dried tilapia (0.05 mg/kg) but similar to firewood-smoked samples (0.37 mg/kg), due to traditional processes, agreeing with the higher levels of firewood because uncontrolled smoking elevates PAH4 (Ekwere *et al*., 2025). Variability depends on factors such as fuel type, combustion temperature, and fish lipid content, which were observed in the smoked fish (Borodi *et al*., 2022). EU Regulation (EC) No 1881/2006 as amended by (EU) 2020/1255, the maximum limit for ∑PAH4 in smoked fish is 0.012 mg/kg (12 µg/kg), with benzo(a)pyrene at 0.002 mg/kg (2 µg/kg); all detected levels here exceeded the limit, implying health risks unless under derogations, which are special exemptions or allowances granted to certain EU member states for traditional smoked products to permit higher levels while preserving cultural and sensory attributes.

Proximate Composition

Nutritional value of fish is mostly predetermined by the protein content of fish which contains essential amino acids. Processing technique, storage, species variations and environmental effects may cause variations in proximate composition (Lawal *et al*., 2026, Shadyeva *et al.*, 2019). The proximate composition was similar among the treatment at the initial check (week 0) except for the ash content among the fuel types, the least ash content in the solar-dried reflect increased mineral concentration in the smoked fish which might be caused by dehydration due to smokes and elevated heat supply. After the weeks of storage (week 12), moisture content decreased while protein and carbohydrate contents increased, with *C. gariepinus* showing higher protein (47.72 ± 0.77%) and ash contents, and *O. niloticus* exhibiting higher carbohydrate content. This agrees with effects of storage on smoked *O. niloticus* where protein value was increased over time as a result of moisture loss that concentrated nutrient (Ayeloja *et al*., 2020).

However, the higher protein in *C. gariepinus* contrasts with reports of lower protein in catfish in comparison to tilapia in dried forms, possibly due to species-specific responses to smoking, which is contrary to the findings in this study, where catfish's denser muscle retained more protein after dehydration (Fitri *et al*., 2022). Moisture was lowest in charcoal-smoked fish, as expected with better preserved fish, whereas firewood-smoked contained more protein, consistent with heat-induced concentration of proteins, which agrees with the fuel effects observed here, as charcoal's even heat effectively reduces moisture (Tenyang *et al*., 2022). The overall decrease of moisture and lipid during post-storage is in line with dried fish reviews by Fitri *et al*. (2022), where lipid oxidation and volatilization occur, which is in agreement with the storage decreases observed in this study due to the influence of ambient storage that supports evaporation and breakdown.

Free Fatty Acids (FFA)
The FFAs in this study varied in the samples, it was higher in *C. gariepinus* than *O. niloticus*, and there was no significant difference for any of the species among the fuel types. This species effect aligns with higher FFA in smoked *Polypterus bichir bichir* due to lipid-rich tissues, agreeing with the species differences here (despite no fuel-type differences) because catfish's higher fat content promotes hydrolysis, leading to elevated FFA levels in *C. gariepinus* (Tenyang *et al*., 2022). The FFA values (0.48-1.08 mg/g) imply potential rancidity onset, as they approach or exceed the 0.5-1.5% threshold for noticeable off-flavors; this may result from heat-accelerated lipolysis during processing, agreeing with increased FFA in smoked herring post-smoking (Tenyang *et al*., 2018). However, they were within the FAO limit of 2.5% for safe consumption, contrasting higher post-storage FFA in some fish exceeding limits.

Peroxide Value (PV)
Primary lipid oxidation is measured by peroxide value; elevated levels are an indicator of rancidity risk. In this experiment, PV was higher in *C. gariepinus* compared to *O. niloticus*, with no significant differences observed among the fuel types. Values of 2.16-3.61 meq/kg are below the <20 meq/kg initial rancidity threshold, which indicate good quality. This is consistent with smoked Polypterus where PV remained low post-smoking, with similarly low levels observed across all fuels including firewood and maize cob (Tenyang *et al*., 2022). This concurs with herring studies showing PV <10 meq/kg as safe for consumption, agreeing with the safe limits here because the 12-week storage period did not cause PV to exceed oxidation thresholds, indicating effective preservation against rancidity (Tenyang *et al.*, 2018).

CONCLUSION
The study revealed a total of twenty polycyclic aromatic hydrocarbons (PAHs) that were similar among the treatments, except naphthalene which was different among the fuel types, and benzo (k)fluoranthene and benzo(a)pyrene that were either different among the species and fuel types. In contrast, solar-dried *C. gariepinus*. showed elevated level of the three PAHs. The lowest levels were found in maize cob smoked. Many of the PAHs including the carcinogenic PAH4 exceeded the EU limit (0.002 mg/kg for benzo(a)pyrene, 0.012 mg/kg for ∑PAH4). The proximate profile showed reduced moisture, ash and lipid after 12 weeks of storage while crude protein increased, with the least crude protein in solar dried and higher values in *C. gariepinus*. The FFAs and PV showed species specific results with higher amount in *C. gariepinus* while there was no difference among the fuel types and all the fish were within the range for safe consumption. Therefore, all the processed fish can be said to be wholesome for consumption due to their proximate composition, free fatty acids and peroxide value. However, it is recommended that attention should be paid to materials to be used in constructing solar dryer in order not to deny the dried fish the benefits associated with not using smoke-based fuel types.

CRediT authorship contribution statement
Conception and design of the study - A.B. Dauda, M.T Ahmed, A.K, Saidu and A.H. Oladele
Acquisition of data – M.T. Ahmed, A.K. Saidu, A.B. Abdullahi, I.A. Abdulhakeem and A.R. Abdulazeez
Analysis and interpretation of data – A.B. Dauda, A.A. Ayeloja, A.B. Abdullahi and I.A. Abdulhakeem
Drafting of manuscript – A.B. Dauda, A.B, A.H. Oladele, Abdullahi, and I.A. Abdulhakeem.
Revision of the manuscript – All the authors
Final approval of the manuscript – All the authors

Ethical consideration
The research was carried out in compliance with local ethical standards as approved by the ethical committee of Federal University Dutsin-Ma, Katsina State, Nigeria.

Declaration of competing interest
Authors declare no known area of competing interest.

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REFERENCES
Abdel-Shafy, H.I. & Mansour, M.S.M. (2016). A review on polycyclic aromatic hydrocarbons: Source, environmental impact, effect on human health and remediation. *Egyptian Journal of Petroleum*, 25, (1), 107-123. .

Abdul Kari, Z., Kabir, M. A., Abdul Razab, M. K. A., Munir, M. B., Lim, P. T. & Wei, L. S. (2020). A replacement of plant protein sources as an alternative to fish meal ingredient for African catfish, *Clarias gariepinus*: A review. *Journal of Tropical Resources and Sustainable Science, 8*(1), 47–59.

Abdullahi, B., Dasuki, A. & Dauda, A. (2024). Comparison of fuelwoods efficiency and keeping quality of *Bagrus bajad* (Fabricius, 1775) smoked with different wood types. *Nigerian Journal of Fisheries, 21*(1), 2803–2809.

Abolagba, O. J, Adekunle, A. T, Dede, A. P. O. & Omoigui, G. O. (2011) Microbial assessment of smoked fish (*Clarias spp*) in Benin Metropolis, Edo State, *Nigeria. Nigerian Journal of Agriculture, Food and Environment.*7: (3) 55-58.

Abubakar R. (2016). Physical, chemical and Quality indices of MSc. Dissertation Department of Biological sciences, Faculty of Science, Ahmadu Bello University, Zaria, Nigeria.1-51.

Association of Official Analytical Chemists [AOAC]. (2016). *Official Methods of Analysis* (20th ed.). Washington, DC: AOAC International. .

Ariyamuthu, R., Albert, V. R. & Je, S. (2022). An overview of food preservation using conventional and modern methods. *Journal of Food and*

Nutrition Sciences, 10(3), 70–79.

Ayeloja, A. A., Jimoh, W. A., Adetayo, M. B. & Abdullahi, A. (2020). Effect of storage time on the quality of smoked *Oreochromis niloticus*. *Heliyon*, *6*(1), e03284.

Borodi, C. M. S., Peter, A., Margaret, M. & Stephen, M. (2022). Effect of smoking technologies on nutritional values and concentration levels of polycyclic aromatic hydrocarbons in smoked fish (*Mormyrus caschive*and *Oreochromis niloticus*) of Terekeka, South Sudan. *African Journal of Food Science*, 16(1), 1-10.

Dasuki, A., Abdullahi, B.A. & Adikwu, I. (2022). Characterization of Polycyclic Aromatic Hydrocarbons Concentrations in Silver Catfish (*Bagrus bajad*) Smoked with Different Wood types. In: K.A. Abubakar, O.A. Sogbesan, and H.A. Adebowale (eds). *Proceedings of the 37th Annual Conference of Fisheries Society of Nigeria (FISON)* held between 30th October – 4th November, at Federal College of Education, Yola, Adamawa State. Pp 541-545

Dauda, A. B., Omosalewa, O. O. & Garuba, A. M. (2020). Preference and perception of fish processors on the influence of wood types on the quality of smoked-dried fish in Katsina State. *Proceedings of the 35th Annual National Conference of Fisheries Society of Nigeria (FISON),* 357-361.

Diop, E. H. M., Ndiaye, B., Sow, A., Sambe, F. M., Sall, M. & Thiam, M. S. (2023). Polycyclic aromatic hydrocarbon (PAH) contents of four species of smoked fish from different sites in Senegal. *International Journal of Analytical Chemistry*, Article 2931615.

Ekwere, I. O., Okpoji, A. U., Igwegbe, K. C., Okonkwo, C. O., Yekeen, A. A., Obunezi, O. C., Okpanachi, C. B., Garuba, M. H., Ogini, O. R. & Odibo, U. E. (2025). Nutritional–Toxicological Trade-Off: Comparative Study of Polycyclic Aromatic Hydrocarbons in Smoked and Oven-Dried Nile Tilapia (*Oreochromis niloticus*). *Journal of Environment, Climate, and Ecology*, 2(2), 90-97.

FAO. (2015). *Fisheries & Aquaculture Report*. No. 889. Food & Agriculture Organization of the United Nations (FAO), Cairo, 61.

Fitri, N., Chan, S. X. Y., Che Lah, N. H., Jam, F. A., Misnan, N. M., Kamal, N., Sarian, M. N., Mohd Lazaldin, M. A., Low, C. F., Hamezah, H. S., Rohani, E. R., Mediani, A. & Abas, F. (2022). A comprehensive review on the processing of dried fish and the associated chemical and nutritional changes. Foods, 11(19), 2938.

Huong, D. T. T. (2014). The effect of smoking methods on the quality of smoked Mackerel. United Nations University Fisheries Training Programme, Iceland [final project].

Lawal, K.I., Bichi, A.H., Moruf, R.O., Maradun, H.F. & Dauda, A.B. (2026). Nutritional and microbial evaluation of some frozen marine fish species sold in selected local government areas of Kano State, Nigeria *Acta Scientiarum*. *Animal Sciences*, v. 48, e74710.

Mishra, S.P. (2020). Significance of fish nutrients for human Health. *International Journal Fisheries Aquaculture Resources* 5(3):47-49.

Nizio, E., Czwartkowski, K. & Niedbała, G. (2023). Impact of smoking technology on the quality of food products: Absorption of polycyclic aromatic hydrocarbons (PAHs) by food products during smoking. *Sustainability, 15*(24), 16890.

Nnaji, J. & Ekwe, N. (2018). Effect of smoking on polycyclic aromatic hydrocarbons (PAHs) concentrations in catfish and tilapia muscles. *Journal of Applied Sciences and Environmental Management, 22*(2), 293–297.

Olokor, J.O., Ihuahi, J.A., Omojono, F.S. & Adelomo, E.O. (2007) Handbook of Practical Fisheries Technology Division, National Institute of Freshwater Fisheries Research (NIFFR). Remi-Thomas Press, New-Bussa, Niger State, Nigeria. Rem-Thomas press pp 13 – 42

Oriji, O. G., Nartey, V. K. & Brimah, A. K. (2012). Determination of the Polycyclic Aromatic sHydrocarbon and heavy metal contents of some Nigerian emulsion paints. *International Journal of Applied Chemistry*, *8*(3), 215-225.

Rabiepour, A., Zahmatkesh, F. & Babakhani, A. (2024). Preservation techniques to increase the shelf life of seafood products: An overview. *Journal of Food Engineering and Technology, 13*(1), 1–24.

Savin, R.-L., Ladoși, D., Ladoși, I., Păpuc, T., Becze, A., Cadar, O., Török, I., Simedru, D., Mariș, Ș. C. & Coroian, A. (2024). Influence of fish species and wood type on polycyclic aromatic hydrocarbons contamination in smoked fish meat. *Foods, 13*(12), 1790.

Suárez-Medina, M. D., Sáez-Casado, M. I., Martínez-Moya, T. & Rincón-Cervera, M. Á. (2024). The Effect of Low Temperature Storage on the Lipid Quality of Fish, Either Alone or Combined with Alternative Preservation Technologies. *Foods (Basel, Switzerland)*, *13*(7), 1097.

Tenyang, N., Tiencheu, B. & Womeni, H.M. (2018) Effect of smoking and refrigeration on lipid oxidation of *Clupea harengus*: A fish commonly consumed in Cameroon. *Food Sci Nutr*. 2018; 6: 464–473.

Tenyang, N., Ateufack Mawamba, L., Ponka, R., Mamat, A., Tiencheu, B. & Womeni, H. M. (2022). Effect of cooking and smoking methods on proximate composition, lipid oxidation and mineral contents of *Polypterus bichir bichir* fish from far-north region of Cameroon. Heliyon, 8(10), Article e10921.

Ubbor, S. C., Arukwe, D. C., Ejechi, M. E. & Ekeh, J. I. (2022). Physicochemical and sensory evaluation of cookies produced from composite flours of wheat, Bambara nut and orange-fleshed sweet potato. *Journal of Agriculture and Food Sciences, 20*(1), 60–77.

Ubwa, S. T., Abah, J., Tarzaa, L., Tyohemba, R. L. & Ahile, U. J. (2015). Effects of traditional smoking methods on the concentrations of polynuclear aromatic hydrocarbons (PAHs) in some species of smoked fish traded in Benue State, Nigeria. *Journal of Food Research, 4*(2), 119–127.

Wang, X. & Zheng, Z. (2025). Mechanistic insights into fish spoilage and integrated preservation technologies. *Applied Sciences, 15*(14), 7639.