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Evaluation of Polycyclic Aromatic Hydrocarbon and Heavy Metals Contamination in Stockfish Species Available in Onitsha Market: Implication for Human Health Risk

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Abstract — This study evaluated the concentrations of polycyclic aromatic hydrocarbons (PAHs) and selected heavy metals in two commercially important stockfish species, Apama (Sepia species) and Haddock (Melanogrammus aeglefinus), obtained from Onitsha Market, Nigeria, to determine their potential human health implications. Six representative samples collected from different market locations were analyzed using Gas Chromatography–Mass Spectrometry (GC–MS) for PAHs and Atomic Absorption Spectrophotometry (AAS) for heavy metals. Iron was the predominant metal, ranging from 40.28–83.88 mg/kg, followed by zinc (13.00–21.00 mg/kg), copper (0.80–1.90 mg/kg), manganese (3.00–5.99 mg/kg), chromium (≤0.006 mg/kg), lead (ND–0.002 mg/kg), and cadmium (0.00001–0.00020 mg/kg), while nickel, arsenic, and mercury were not detected. The detected concentrations of lead, cadmium, mercury, and arsenic were below the maximum limits recommended by WHO/FAO for fish and fishery products, indicating minimal heavy metal contamination. Sixteen priority PAHs were identified, with phenanthrene, fluoranthene, and pyrene being the most frequently detected compounds. Total PAH concentrations ranged from 0.01034 to 0.23780 µg/kg, whereas benzo(a)pyrene concentrations (ND–0.00020 µg/kg) were far below the European Union maximum limit of 2 µg/kg for smoked fish products. Overall, the analyzed stockfish samples were considered safe for human consumption; however, routine monitoring remains essential to detect future contamination and ensure continued consumer protection.

Keywords— PAH, Heavy Metals, Stockfish, Onitsha Market, Health Risk.

I. INTRODUCTION

Fish and fishery products constitute an essential component of the human diet because they provide high-quality protein, essential amino acids, omega-3 fatty acids, vitamins, and minerals required for growth, development, and the maintenance of good health (Obruche et al., 2025). In many developing countries, including Nigeria, fish serves as a major source of animal protein due to its affordability, nutritional value, and wide availability. Among the various fish products consumed in Nigeria, stockfish (locally known as okporoko) occupies a unique position because of its long shelf life, ease of storage, and widespread use in both rural and urban households (Ikechukwu et al., 2026). The increasing demand for stockfish has resulted in a substantial volume of imported products being distributed through major commercial markets such as Onitsha Main Market, one of the largest trading centers in West Africa. Consequently, ensuring the safety and quality of stockfish available to consumers has become an important public health concern (Etus and Obruche, 2026). Despite its nutritional benefits, fish is recognized as an efficient bioaccumulator of environmental contaminants due to its direct contact with aquatic ecosystems (Ekpo et al., 2023). Pollutants introduced into rivers, lakes, and oceans through industrial discharge, petroleum exploration, agricultural runoff, municipal waste, and atmospheric deposition may accumulate in aquatic organisms and eventually enter the human food chain. Among these contaminants, heavy metals and polycyclic aromatic hydrocarbons (PAHs) are of particular concern because of their persistence, toxicity, bioaccumulative characteristics, and potential to induce adverse health effects following prolonged exposure (Festus-Amadi et al., 2021). Human exposure to these contaminants occurs primarily through dietary intake, making food safety monitoring an essential component of environmental and public health protection. Heavy metals such as lead (Pb), cadmium (Cd), mercury (Hg), arsenic (As), chromium (Cr), nickel (Ni), copper (Cu), zinc (Zn), iron (Fe), and manganese (Mn) occur naturally in the environment but may reach elevated concentrations through anthropogenic activities (Sarah et al., 2026). While essential metals such as iron, zinc, copper, and manganese play important physiological roles, excessive intake may cause toxic effects (Itodo et al., 2021; Jacintha et al., 2025). Conversely, non-essential metals including lead, cadmium, mercury, and arsenic possess no known biological function and are toxic even at relatively low concentrations (Umudi et al., 2025). Chronic exposure to these metals has been associated with neurological disorders, kidney dysfunction, cardiovascular diseases, developmental abnormalities, immune suppression, and various forms of cancer. Therefore, monitoring heavy metal concentrations in commonly consumed food products remains an important strategy for minimizing dietary exposure and protecting public health (Erienu et al., 2022). Polycyclic aromatic hydrocarbons comprise a large group of organic compounds containing two or more fused aromatic rings. They are generated mainly through incomplete combustion of organic materials, petroleum processing, fossil fuel combustion, industrial emissions, biomass burning, and environmental oil pollution. Some PAHs may also be introduced into fish during processing, transportation, storage, and handling through contamination from smoke, packaging materials, or polluted environments. Several PAHs, particularly benzo(a)pyrene, benzo(a)anthracene, chrysene, and dibenzo(a,h)anthracene, have been identified as mutagenic and carcinogenic compounds (Okpanachi et al., 2025). Long-term dietary exposure to these substances has been linked with increased risks of cancer, reproductive disorders, endocrine disruption, and genetic damage. Consequently, international regulatory agencies such as the World Health Organization (WHO), the Food and Agriculture Organization (FAO), the Codex Alimentarius Commission, and the European Food Safety Authority have established guidelines and maximum permissible limits for selected contaminants in foods to safeguard public health. Nigeria is one of the largest consumers of imported stockfish in Africa, with products distributed through major wholesale markets before reaching consumers across the country (Umudi et al., 2025). Onitsha Main Market serves as a principal distribution hub because of its enormous commercial activities and extensive food trading network (Ogwuche and Obruche, 2020). The market experiences intense human activities, heavy vehicular traffic, and prolonged storage of food commodities, conditions that may increase the possibility of contamination during transportation, handling, storage, and marketing (Nathan et al., 2025). Although numerous studies have evaluated heavy metal contamination in fresh and smoked fish harvested from rivers and coastal waters in Nigeria, comparatively fewer investigations have simultaneously assessed both heavy metals and the sixteen United States Environmental Protection Agency (USEPA) priority PAHs in imported stockfish sold in major commercial markets. Furthermore, available studies are often limited to a few contaminants or specific geographical locations, providing insufficient information for comprehensive dietary risk assessment. The scarcity of current data on contaminant levels in imported stockfish marketed in Onitsha represents an important knowledge gap (Ugochukwu et al., 2025). Continuous monitoring is necessary because contamination patterns may change over time due to differences in source waters, processing methods, transportation conditions, environmental pollution, and market handling practices. Moreover, consumers generally assume that dried imported fish is inherently safe because of its preservation process, despite limited scientific evidence supporting this assumption (Obruche et al., 2019). Reliable contaminant data are therefore required to evaluate compliance with international food safety standards and to provide evidence-based recommendations for regulatory authorities, food safety agencies, traders, and consumers. This study was therefore undertaken to determine the concentrations of selected heavy metals and the sixteen priority PAHs in Apama (Sepia species) and Haddock (Melanogrammus aeglefinus) stockfish sold in Onitsha Main Market, Nigeria.

II. MATERIALS AND METHOD

1. Pre-treatment

To minimize the possibility of sample contamination and ensure analytical accuracy, sample pre-treatment was carried out according to the procedure described by (Obruche et al., 2018). All sample containers were soaked overnight in nitric acid (HNO_3), thoroughly rinsed with distilled water, and sun-dried for approximately 2–3 hours before use. The cleaned containers were clearly labeled as A1–A6 to facilitate proper sample identification and record keeping. Samples A1, A2, and A3 represented Apama (Sepia species) obtained from different locations within Onitsha Market, while samples A4, A5, and A6 represented Haddock (Melanogrammus aeglefinus) collected from separate sections of the same market.

2. Sample Collection

Sample collection was performed following the procedure reported by Umudi et al. (2025). A random sampling approach was adopted to obtain two commonly consumed stockfish species, Apama (Sepia species) and Haddock (Melanogrammus

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aeglefinus), locally known as okporoko, from different sales points within Onitsha Main Market. Onitsha Market, recognized as one of the largest commercial markets in West Africa, experiences intense commercial activities, heavy vehicular movement, and a high human population, making it an appropriate location for evaluating food contamination. The purchased fish samples were thawed where necessary, descaled, cut into smaller portions, and thoroughly rinsed with clean water before laboratory processing.

3. Sample Preparation

Immediately after collection, the fish samples were transported to the laboratory to minimize contamination and deterioration (Ekpo et al., 2025). Each sample was homogenized separately using a ceramic mortar and pestle to obtain a uniform matrix and improve extraction efficiency. The homogenized samples were air-dried under controlled laboratory conditions for three days to eliminate residual moisture, enhance sample stability, and prevent microbial growth. After drying, the samples were further pulverized into fine powder to ensure homogeneity and facilitate efficient extraction of polycyclic aromatic hydrocarbons. The powdered samples were transferred into clean, airtight containers and stored until analysis.

4. Standards and Reagents

A certified standard mixture containing the sixteen United States Environmental Protection Agency (USEPA) priority polycyclic aromatic hydrocarbons (2000 µg/mL in dichloromethane:benzene) comprising naphthalene, acenaphthene, fluorene, phenanthrene, anthracene, anthracene-d10, pyrene, benzo[a]anthracene, chrysene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, dibenz[a,h]anthracene, and benzo[ghi]perylene, each with purity greater than 98%, was obtained from Supelco (Bellefonte, USA) through its Nigerian representative. Calibration standards were prepared by serial dilution using HPLC-grade acetonitrile supplied by Chem-Lab Belgium. All analytical solvents used throughout the study were of HPLC grade, while ultrapure water was produced using a Millipore Milli-Q purification system (Millipore, Molsheim, France).

5. Preparation of Calibration Standards

Individual PAH stock solutions (1 mg/mL) were prepared in toluene according to established analytical procedures (Obruche et al., 2019). The prepared solutions were transferred into amber glass bottles and stored at 4°C until required for analysis. Before use, the standards were equilibrated to room temperature for approximately 30 minutes. A mixed intermediate standard solution (100 ng/mL) was subsequently prepared by diluting the stock solutions with methanol. Calibration standards containing 0.35, 0.70, 1.40, 2.80, and 5.60 ng/mL were prepared by spiking 10 mL aliquots of blank water with appropriate volumes (35–560 µL) of the mixed standard solution. Anthracene-d10 (1 mg/mL in toluene) served as the internal standard, and 70 µL of a 100 ng/mL methanolic solution was added to each calibration sample before proceeding with the extraction procedure.

6. Extraction of Polycyclic Aromatic Hydrocarbons

Extraction of PAHs from the fish samples was carried out following the method described by Agerstach and Skog (2005). Initially, the samples were digested using alcoholic potassium hydroxide to break down the organic matrix and liberate PAHs. Liquid–liquid extraction was then performed using 1,1,2-trichlorotrifluoroethane (TCTFE) to isolate the target compounds from the sample matrix. The crude extracts were purified by column chromatography using neutral activity I alumina as the adsorbent. The alumina was prepared as a slurry with hexane and packed into glass chromatographic columns (1 cm internal diameter) to a height of approximately 10 cm. The concentrated extracts, previously reconstituted in hexane, were loaded onto the columns. Hexane was first used to elute aliphatic hydrocarbons and other non-polar impurities, after which benzene was employed to recover the aromatic fraction containing the PAHs. The purified extracts were subsequently concentrated and prepared for gas chromatographic analysis (Umudi et al., 2025).

7. GC–MS Analysis of PAHs

Quantitative determination of PAHs was performed using a Varian 3800 gas chromatograph coupled with a Varian Saturn 2200 mass spectrometer equipped with a 30 m × 0.25 mm internal diameter WCOT CP-Sil 8 CB capillary column. The oven temperature program commenced at 70°C and was maintained for one minute before increasing to 300°C at a rate of 10°C/min, followed by a 30-minute hold. The injector and detector temperatures were maintained at 250°C and 300°C, respectively, while helium was used as the carrier gas at a constant flow rate of 2 mL/min. Compound identification was achieved by comparing retention times and mass spectra with those of reference standards, whereas quantification was based on calibration curves generated from seven concentration levels ranging from 2 to 2000 ng/L. Instrument detection limits ranged from 0.8 to 2.0 ng/L at a signal-to-noise ratio of 3.

8. Sample Digestion for Heavy Metal Determination

For heavy metal analysis, each sample was digested with 10 cm^3 of an acid mixture consisting of perchloric acid ($HClO_4$), nitric acid (HNO_3), and sulfuric acid (H_2SO_4) mixed in a ratio of 1:2:2. Digestion was carried out on a hot plate inside a fume cupboard until dense white fumes appeared, indicating complete mineralization of the organic matter.

Determination of Heavy Metals by Atomic Absorption Spectrophotometry

Following digestion, the samples were cooled and diluted with 20 cm³ of distilled water to dissolve the target metals, including zinc, iron, chromium, cadmium, nickel, lead, mercury, copper, manganese, and arsenic. The digests were allowed to reach room temperature, filtered through ashless Whatman filter paper into 100 cm³ volumetric flasks, and diluted to volume with distilled water. Metal concentrations were determined using an AAS Phoenix 986 Atomic Absorption Spectrophotometer (Biotech, UK) in accordance with standard APHA, AWWA, and WEF analytical procedures. Mercury was analyzed using the cold vapour atomic absorption technique, while arsenic determination was performed by hydride generation atomic absorption spectrophotometry to achieve enhanced analytical sensitivity.

III. RESULTS AND DISCUSSION

This section presents the result of the concentrations of ten heavy metals and PAHs in Apama (Sepia species) and Haddock (Melaogrammus aeglefinus), both with local name called okporoko stockfish species collected from onitsha market are shown in table 1 and 2.

Table 1: Concentrations of heavy metals in the Apama and Haddock

Parameter	A1	A2	A3	A4	A5	A6
Nickel	ND	ND	ND	ND	ND	ND
Iron	40.28	66.01	67.55	80.07	82.25	83.88
Lead	0.001	0.0001	0.00001	0.002	0.001	ND
Chromium	0.004	BDL	0.003	0.005	0.006	0.003
Arsenic	ND	ND	ND	ND	ND	ND
Mercury	ND	ND	ND	ND	ND	ND
Copper	0.80	1.84	1.90	1.61	1.60	1.43
Zinc	13.00	19.00	19.01	17.37	17.99	21.00
Manganese	3.76	5.00	3.77	5.99	3.00	3.79
Cadmium	0.0001	0.0001	0.0002	0.0001	0.00001	0.0001

A1, A2 and A3 = Apama fish from different locations in Onitsha market, A4, A5, A6 = Haddock stockfish from different location in Onitsha market. ND =not detected

The summary of the concentrations of various PAHs present in Apama (Sepia species) and Haddock (Melaogrammus aeglefinus), stockfish samples collected from Onitsha market in Anambra state, Nigeria is shown in Table 2.

Table 2: Polyaromatic Hydrocarbon (PAHs) concentrationtockfishs of Apama (Sepia species) and Haddock (Melaogrammus aeglefinus) of samples in (µg/kg)

Parameter	A1	A2	A3	A4	A5	A6
Naphthalene	ND	ND	ND	0.00001	0.0001	0.0001
2 Methylnaphthalene	0.0001	ND	0.00001	ND	0.0001	ND
Acenapthylene	0.0001	0.001	ND	0.0001	ND	0.0001
Acenaphthene	ND	0.001	ND	0.00001	ND	0.00001
Fluorene	0.0001	0.001	0.00001	ND	0.003	0.00001
Phenanthrene	0.002	0.004	0.003	0.002	0.003	0.005
Anthracene	0.0004	0.0003	0.0002	0.0003	0.001	0.001
Fluoranthene	0.005	0.004	0.007	0.0010	0.022	0.010
Pyrene	0.001	0.003	0.002	0.002	0.0001	0.001
Benzo(a)anthracene	0.0001	0.00001	0.002	0.00001	0.002	0.0001
Chrysene	0.0002	0.00016	0.00001	0.005	0.0001	0.0001
Benzo(b)fluoranthene	0.003	ND	0.0002	0.00001	0.0001	0.0002
Benzo(k)fluoranthene	0.00013	0.00016	0.00012	0.0006	0.0008	0.0001
Benzo(a)pyrene	0.00001	ND	0.0001	ND	0.0001	0.0002
Dibenzo(a,h)anthracene	0.00012	0.00001	0.002	0.0001	0.007	0.0001
Benzo(g,h,i)perylene	0.00013	0.00011	0.0004	0.0001	0.0003	0.0002
Indeno(1,2,3-d)pyrene	0.0001	0.00012	0.00012	0.0001	0.0001	0.0003
Total	0.01226	0.01487	0.01917	0.01034	0.2378	0.01852

Discussion

The concentrations of heavy metals detected in the Apama (Sepia species) and Haddock (Melanogrammus aeglefinus) stockfish samples indicate generally low levels of contamination. Iron was the most abundant metal, with concentrations ranging from 40.28 to 83.88 mg/kg, followed by zinc (13.00–21.00 mg/kg), manganese (3.00–5.99 mg/kg), and copper (0.80–1.90 mg/kg). These metals are essential micronutrients required for normal physiological functions, and their concentrations were within acceptable levels reported for edible fish. Conversely, toxic metals including lead (ND–0.002 mg/kg), cadmium (0.00001–0.00020 mg/kg), and chromium (below detection limit–0.006 mg/kg) were detected only in trace amounts, while nickel, arsenic, and mercury were not detected in any sample. The concentrations of lead and cadmium were substantially below the maximum permissible limits of 0.30 mg/kg and 0.05 mg/kg, respectively, recommended by the FAO/WHO Codex Alimentarius Commission for fish and fishery products. Similarly, the absence of detectable mercury and arsenic indicates compliance with the recommended maximum limits of 0.50 mg/kg for mercury and 0.10 mg/kg for inorganic arsenic, suggesting that consumption of these stockfish species poses minimal risk of heavy metal toxicity. This result is in agreement with the work of (Etus and Obruche, 2026)

The PAH profile also demonstrated very low contamination across all samples. Total PAH concentrations ranged from 0.01034 to 0.23780 µg/kg, with sample A5 exhibiting the highest concentration. Phenanthrene and fluoranthene were the dominant compounds, whereas benzo(a)pyrene, one of the most toxic and carcinogenic PAHs, occurred at extremely low concentrations (ND–0.00020 µg/kg). These values are far below the European Union maximum limit of 2.0 µg/kg for benzo(a)pyrene in smoked fish products and the 12 µg/kg limit established for the sum of four marker PAHs (PAH4), which are widely adopted in international food safety assessments because WHO/FAO has not established specific limits for PAHs in fish. This research work is in agreement with the work of (Micheal et al., 2026). The generally low PAH concentrations suggest minimal contamination from environmental pollution, processing, transportation, or storage. Although sample A5 recorded comparatively higher total PAHs, its concentration remained well below internationally accepted safety thresholds. Overall, the results indicate that both Apama and Haddock stockfish sold in Onitsha Market are safe for human consumption with respect to heavy metals and PAHs. Nevertheless, continuous surveillance is recommended to detect emerging contamination trends, maintain compliance with international food safety standards, and minimize long-term dietary exposure to potentially hazardous contaminants.

IV. CONCLUSION

This study evaluated the concentrations of selected heavy metals and polycyclic aromatic hydrocarbons (PAHs) in Apama (Sepia species) and Haddock (Melanogrammus aeglefinus) stockfish marketed in Onitsha, Nigeria, to assess their potential implications for human health. The results revealed that iron, zinc, copper, and manganese were the predominant metals detected, whereas toxic metals such as lead, cadmium, and chromium occurred only at trace concentrations. Nickel, arsenic, and mercury were not detected in any of the analyzed samples. Similarly, sixteen priority PAHs were identified at very low concentrations, with phenanthrene and fluoranthene being the most abundant compounds. The concentrations of toxic heavy metals were below the maximum permissible limits established by the FAO/WHO Codex Alimentarius Commission, while benzo(a)pyrene and total PAHs were considerably lower than the maximum limits specified by the European Union for fishery products. These findings indicate that the analyzed stockfish samples were not significantly contaminated and are unlikely to pose immediate health risks to consumers through dietary exposure. Nevertheless, the detection of trace quantities of potentially carcinogenic PAHs highlights the importance of continuous monitoring of imported stockfish during transportation, storage, and marketing. Regular surveillance, strict enforcement of food safety regulations, and periodic contaminant assessments are recommended to ensure consumer protection, maintain food quality, and support public health in Nigeria.

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