Research Article

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Performances of the barrel kiln used in cottage industry for fish processing and effects on physicochemical characteristics and safety of smoked fish products

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Abstract

BACKGROUND: In the traditional food sector, the smoking process and smoking-drying process are widely used to increase the shelf-life of seafood products. The smoking process and smoking-drying process are mainly performed using barrel kiln and wood as fuel in many West African countries. The present study evaluated the performances of the barrel kiln and its effects on physicochemical characteristics and safety of smoked fish (SF) and smoked-dried fish (SDF). Twelve follow-ups were conducted with three experimental processors and 24 samples of fish collected at different steps of processing were analyzed in a laboratory using standard methods.

RESULTS: The extreme values of combustion temperature recorded during the smoking process (456.4 °C) and smoking-drying process (482.8 °C) were higher than 450 °C, the temperature at which wood pyrolysis generates polycyclic aromatic hydrocarbons (PAHs). Smoked fish were highly contaminated with PAHs, and showed maximal levels of benzo[a]pyrene (52.7 μg kg^{-1}) and PAH4 (i.e. sum of benzo[a]pyrene, chrysene, benzo[b]fluoranthene and benz[a]anthracene) (290.9 μg kg^{-1}) exceeding the European Union limits by about 25-fold. After smoking of *Scomber scombrus* and smoking-drying of *Cypselurus cyanopterus*, no significant differences were recorded for lipid, protein and biogenic amine contents between fresh and processed fish, even if the histamine content of both fish exceeded the limit fixed by the European Union regulation.

CONCLUSION: The results obtained in the present study showed that smoked fish and smoked-dried fish produced using barrel kiln and wood fuel are highly contaminated by PAHs. Therefore, there is a need to improve the preservation practices of raw fish and smoking conditions to limit the contamination of end-products by PAHs known to be carcinogenic components for humans and to ensure consumer safety.

Keywords: fish processing; barrel kiln; polycyclic aromatic hydrocarbons; biogenic amines; food safety

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INTRODUCTION

Fish and its by-products play a considerable role in the food supply for West Africa populations because 15–20% of protein consumed in this area is from aquatic source.[1] Furthermore, from a nutritionally point of view, fishing products are considered as important sources of polyunsaturated fatty acids, vitamins (A, B, D and E), mineral salts (calcium, phosphorus, magnesium, selenium and iron) and trace elements such as iodine.[2] However, fish preservation in a tropical region, particularly in West Africa, remains a problem because of its very perishable nature, lack of appropriate infrastructures for fresh fish preservation and the climatic conditions that cause fish degradation in a few hours.[3] Thus, to limit post capture losses, different traditional processing technologies such as drying, salting, fermentation and smoking, have been developed and used individually or in combination to preserve fresh fish in West Africa.[3,4] The smoking process, which is the object of the present study, is known as a fish processing method that has been practiced for many generations to promote food diversification, as well as to preserve foodstuffs such as cheese, meat and fish.[5] The smoking and smoking-drying processes belong to artisanal processing technologies that are used the most for fish preservation. Smoked and smoked-dried fish are mainly intended for local consumption in Benin and some of the smoked-dried fish is exported to bordering countries such as Togo, Nigeria, Burkina Faso and Niger.[6] The quality of these products is not stable because the processing technologies are still rudimental. In addition, different types of fuel from various origins, including charcoal, wood and sometimes plastic bag and tires from vehicles, are used.[7,8] Indeed, these kinds of fuels generate several types of chemical contaminants such as polycyclic aromatic hydrocarbons (PAHs), which are known to be carcinogenic and genotoxic, and could be harmful for the health of smoked fish consumers.[9-11]

Other studies carried out in Benin reported that smoked shrimp, smoked fish and smoked-dried fish obtained traditionally and collected on different processing sites were highly contaminated by PAHs with levels of benzo[a]pyrene (BaP) and the sum of four PAHs [BaP, chrysene (CHR), benzo[b]fluoranthene (BbF) and benzo[a]anthracene (BaA)] that were not compliant with the maximum levels of European Union (EU) regulation (No. 1881/2006 of 19 December 2006).[11,12] As indicated by Codex Alimentarius Commission (2009),[13] the process as well as the functioning mode of the smoking kiln also influenced the quality of the end-products. It has also been reported that smoking process impaired the occurrence of biogenic amines such as histamine, tyramine, cadaverine, putrescine, spermine and spermidine in smoked fish and smoked-dried ham compared to those of non-smoked products.[14,15] Other studies have reported high levels of biogenic amines in foods, particularly in fish, as well as food poisoning cases caused by histamine and tyramine present in fish.[7,16-18] Before achieving any improvement of smoking technology, it is important to carry out follow-up trials with the processors during their smoking process of fish and evaluate the main factors that could explain the high level of PAHs in smoked fish.

The present study aimed to characterize the traditional smoking process, to evaluate the thermal and technological performances of the barrel kiln used, and to determine the effects on the physicochemical characteristics and the safety of end-products.

MATERIALS AND METHODS

Raw materials

Imported Atlantic mackerel (*Scomber scombrus*) (80 kg), purchased in fish-shop at Abomey-Calavi (Benin), and flying fish (*Cypselurus cyanopterus*) (60 kg), purchased from fresh fish sellers at fishing port Cotonou (Benin), were used for follow up trials because these two marine fishes were the main used for smoked fish and smoked-dried fish production, respectively.[19] The average mass, length, width and thickness recorded for *S. scombrus* were 266.6 ± 44.8 g, 27.4 ± 2.7 cm, 5.3 ± 0.5 cm and 3.8 ± 0.4 cm, respectively, whereas those recorded for *C. cyanopterus* were 219.7 ± 25.2 g, 24.8 ± 1.4 cm, 4.6 ± 0.3 cm and 3.3 ± 0.2 cm, respectively.

Methods

Experimental design and sampling

Three processors were selected on the basis of their experience (a minimum of 10 years) in hot smoking and smoking-drying practices. Each processor carried out both experiments of smoking and smoking-drying. Each smoking process (hot smoking or smoking-drying) was conducted in three independent experiments in duplicate. From each batch of fish, raw fish was collected as a control to assess the impacts of smoking or smoke-drying on PAHs and biogenic amine levels. Heat and smoke were generated from hard wood of *Acacia auriculiformis* using a traditional barrel kiln on which removable mesh trays were laid for fish loading. Raw Atlantic mackerel after washing was traditionally smoked (hot smoking), whereas raw flying fish after washing was scaled and bent before being hot smoked, and then dried in a traditional barrel kiln. Twenty-four samples (three raw and six smoked Atlantic mackerel; three raw, six smoked and six smoked-dried flying fish) were collected for laboratory analysis.

Determination of thermal and technological characteristics of the barrel kiln

The heat transfer phenomenon inside and outside the kiln during the smoking and smoking-drying processes, including combustion temperature in combustion place, temperature in combustion chamber, temperature at the contact point of metal tray with fish, temperature at the core of product and ambient temperature, were recorded every 30 min with a thermocouple (traceable digital thermocouple no. 620-2006, VWR, France) according to Kpoclou *et al.*[20] In addition, relative humidity was recorded using a thermo-hygrometer (PRO Digital, Model 9822B, rue Edouard Martel - Saint-Etienne Cedex - © SEFRAN) equipped with a probe. The kinetics of matter losses during smoking and smoking-drying processes was assessed. Thus, for each processing method, masses of three fresh fishes, which were randomly selected from different parts of a metal smoking tray and tagged, were tracked and recorded using an electronic balance (5 kg; Terraillon, Paris, France) every 30 min during the process and the yields were calculated at the end of the process.[21,22] Mass transfer associated with losses of water and fat during these processes was evaluated following Eqns (1) and (2) as proposed by Collignan and Raoult-Wack[23]:

$$\mathrm{WL} = X_t - X_{t+1}\left(\frac{m_{t+1}}{m_t}\right) \quad (1)$$

$$\mathrm{FL} = F_t - F_{t+1}\left(\frac{m_{t+1}}{m_t}\right) \quad (2)$$

Where WL is water loss between t and t + 1 (kg per 100 kg of product); FL is fat loss between t and $t + 1$ (kg per 100 kg of product); X_t is water content at t (kg per 100 kg of product) (wet basis); X_{t+1} is water content at $t + 1$ (kg per 100 kg of product) (wet basis); F_t is fat content at t (kg per 100 kg of product) (wet basis); F_{t+1} is fat content at $t + 1$ (kg per 100 kg of product) (wet basis); m_t is mass of product at t (kg); and m_{t+1} is mass of product at $t + 1$ (kg). The calculations were carried out between t and $t + 1$ time.

The durations of smoking and smoking-drying processes were determined using a chronometer.[24] The quantities of wood used during each experiment were determined by weighing with a commercial balance of 20-kg range.[5,20,24]

Determination of physicochemical characteristics

The pH of fresh fish samples was determined according to AFNOR NF V 04-408 using a digital pH meter (pH 730 WTW 82362; Inolab, Wellheim, Germany). The colour was measured with a chromameter (CR/DP 400/410; Minolta, Osaka, Japan) using L^*, a^*, b^* and ΔE coordinates. Dry matter contents of raw, smoked and smoked-dried fish were determined by oven drying the sample at 103 ± 2 °C to constant weight (ISO 1442:1997). Protein content was determined according to AOAC,[25] Method No. 981.10 and fat content was assessed following the method described by Folch *et al*.[26]

Determination of polycyclic aromatic hydrocarbons and biogenic amines

Fifteen PAHs [dibenzo[a,e]pyrene (DeP), dibenzo[a,l]pyrene (DlP), dibenz[a,h]anthracene (DhA), benzo[a]pyrene (BaP), benz[a]anthracene (BaA), benzo[j]fluoranthene (BjF), benzo[k]fluoranthene (BkF), benzo[ghi]perylene (BgP), chrysene (CHR), dibenzo[a,h]pyrene (DhP), benzo[b]fluoranthene (BbF), indeno[1,2,3-cd]pyrene (IcP), dibenzo[a,i]pyrene (DiP), 5-methylchrysene (5MC), benzo[c]fluorene (BcL)] were analysed in smoked fish and smoked-dried fish samples according to the analytical method described by Brasseur *et al*.,[27] whereas 10 biogenic amines, including histidine, were determined according the analytical method described by Douny *et al*.[28] These analytical methods are described as below.

For PAH, extraction was carried out with 1 g of lyophilized sample of smoked fish product with hexane/acetone (50:50, v/v) using accelerated solvent extractor (ASE 200; Dionex Corporation, Sunnyvale, CA, USA). The extract was purified using solid phase extraction columns cartridges (Chromabond HR-X 6 mL/500 mg; Macherey-Nagel, Düren, Germany) as described by Veyrand *et al*.[29] The high-performance liquid chromatography system, commprising a Model 600 E solvent delivery system, equipped with a Model 717 automatic injector, a Mistral TM oven and a 2475 Fluorescence detector (all from Waters Corp., Milford, MA, USA), was used for injection and quantification as described by Brasseur *et al*.[27] and Danyi *et al*.[30] The deuterated (DiP-D14) was used as injection standard and was spiked at a constant concentration (i.e. 250 pg μL^{-1} in acetonitrile) in each calibration solution. For each series of injections, seven calibration solutions containing the 15 PAHs at increasing concentrations from 2.5 to 400 pg μL^{-1}, except for BjF and IcP (from 10 to 1600 pg μL^{-1}), were injected. The calibration curve was used to calculate the PAH concentration from the response factor (ratio between both native and injection standard PAHs peak areas. Any concentration below the concentration of the first point of standard curve was not quantifiable. Accordingly, the concentration of the first point of calibration curve was set as limit of quantification (LOQ). The PAH LOQ expressed on fresh weight (based on average of 50% of moisture) was 0.1 µg kg^{-1}, except for IcP and BjF, which displayed a LOQ of 0.5 µg kg^{-1} fresh weight.

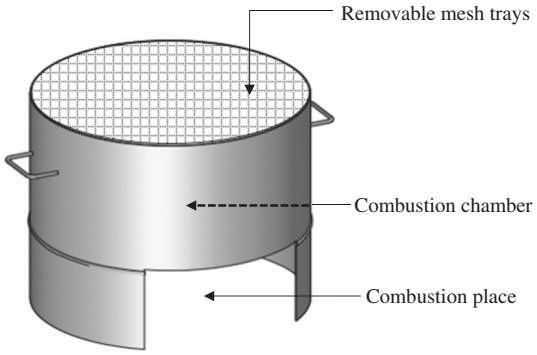

Figure 1. . Barrel kiln.

For the biogenic amines, extraction was performed using 2 g of ground fresh fish sample by adding 0.3 mL of perchloric acid (0.4 mol L^{-1}) and 200 µL of internal standard (1,7-diaminoheptane) of 100 ng μL^{-1} solution in 5% trichloroacetic acid. The derivatisation was realized by adding 2 mL of dansyl chloride (10 mg mL^{-1} in acetone) and tubes were incubated at 70 °C for 15 min in a water bath followed by centrifugation (3700 × g for 10 min at room temperature). Injection and quantification were performed using an UPLC Acquity system integrated autosampler (Acquity Sample Manager FTN), a solvent delivery system (Acquity QSM H Class) and a column heater coupled to an Acquity Fluorescence detector (FLD) (all from Waters Corp.). The column used was an Acquity UPLC BEH C18 (2.1 × 100 mm, 1.7 µm) with a UPLC BEH C18 VanGuard precolumn (2.1 × 5 mm, 1.7 µm) (Waters Corp.). The LOQ, which is also the first point of the calibration curve, ranged between 1.3 and 10 mg kg^{-1} depending on the biogenic amines

Statistical analysis

Data were entered and processed in Excel (Microsoft Corp., Redmond, WA, USA) to determine the descriptive statistics (mean ± SD). STATISTICA, version 7.1 (StatSoft, Paris, France) was used to carry out the comparison of means; in particular, Student's t-test and one-way analysis of variance (Newman–Keuls test), as well as nonparametric tests, namely the Mann–Whitney U-test and the Kruskal–Wallis test, at a threshold of significance $\alpha = 5\%$.

RESULTS AND DISCUSSION

Characteristics of the barrel kiln used for fish smoking and smoking-drying

The barrel kiln (Fig. 1) is made from a recycled iron barrel and has a cylindrical shape with characteristics summarized in Table 1. The barrel kiln is similar to those described by Idah and Nwankwo,[31] Degnon *et al*.,[24] Kpoclou *et al*.[20] and Assogba *et al*.[19] The barrel kiln is used with wood from *A. auriculiformis*. It has a direct functioning mode because of direct exposition of processed products to smoke and heat from firewood.

Processing of fresh fish into smoked and smoked-dried fish and mass balance

In the present study, the smoking process was carried out in three main steps. The fish (*S. scombrus*) was washed twice and then spread on the metal smoking tray before being smoked (Fig. 2a). The smoking-drying process was carried out in five steps.

Table 1. Characteristics of the barrel kiln

Characteristics	Size			
	Width	Height	Diameter	Volume
External size (cm)	–	48–51	67–79.5	
Opening combustion place size (cm)	36–64.5	19.5–33	–	
Size of smoking tray (cm)	–	–	82–93	
Covering surface of smoking tray (m^2)		0.5–0.7		
Thickness of kiln walls (cm)		0.7–0.8		
Mesh size of smoking tray wire (cm × cm)		3.3 × 2.5		
Surface of the wire section (cm^2)		0.2–0.2		
Total volume of kiln (liter)				169.1–253.0

Fresh fish (*C. cyanopterus*) was scaled, washed, bended and spread on the metal smoking tray and smoked-dried (Fig. 2b). The mass balances during smoking and smoking-drying were determined, as shown in Fig. 2. After the smoking process, the average yield of smoked fish obtained from 100 kg of fresh fish (water content of 70.1 ± 1.5%) was 68.1 ± 4.8% (n = 6). After the smoking-drying process, the average yield of smoked-dried fish obtained from 100 kg of fresh fish (water content of 75.7 ± 0.9%) was 50.9 ± 18.5% (*n* = 6). The weight loss during these two processes was essentially a result of water and fat losses under the effect of heat.

Heat and mass transfer and technological performances of the barrel kiln

Changes in air conditions, kiln and product temperature during smoking and smoking-drying of fish

Changes in temperature in various parts of the barrel kiln during smoking of *S. scombrus* and smoking-drying of *C. cyanopterus* were recorded and are shown in Figs 3 and 4, respectively. During all the smoking process time of *S. scombrus* (90 min), the ambient temperature showed an increasing trend from 30.2 ± 1.6 °C to 34.4 ± 2.3 °C, but without significant change (Fig. 3). However, the air relative humidity decreased significantly from 31.8 ± 5.1% to 23.5 ± 5.0% between 5 and 95 min as a result of heat exchange between the kiln and ambient air.

At the beginning of smoking process (i.e. after 5 min of smoking time), the average temperatures in the combustion place, combustion chamber, at the contact point of the metal smoking tray with fish and at the core of the product were 322.1 ± 83.7 °C, 198.8 ± 30.1 °C, 56.3 ± 12.4 °C and 32.9 ± 11.4 °C, respectively (Fig. 3). From 5 to 65 min of smoking, the average temperature in the combustion place increased and reached 348.4 ± 70.9 °C, with a maximal value of 456.4 °C, whereas the average temperatures in the combustion chamber, at the contact point and at the core of the product increased significantly from 198.8 ± 30.1 °C to 235.9 ± 38.7 °C, from 56.3 ± 12.4 °C to 87.2 ± 20.7 °C and from 32.9 ± 11.4 °C to 75.9 ± 9.2 °C, respectively (Fig. 3). This increase in temperature could be explained by the increase of the intensity of wood combustion in the combustion place. This confirmed the occurrence of heat transfer phenomenon inside the barrel kiln. From 65 to 95 min, the average temperatures in the combustion place, combustion chamber, at the contact point and at the core of product decreased significantly to 333.5 ± 67.2 °C, 226.7 ± 37.5 °C, 77.3 ± 19.3 °C and 70.9

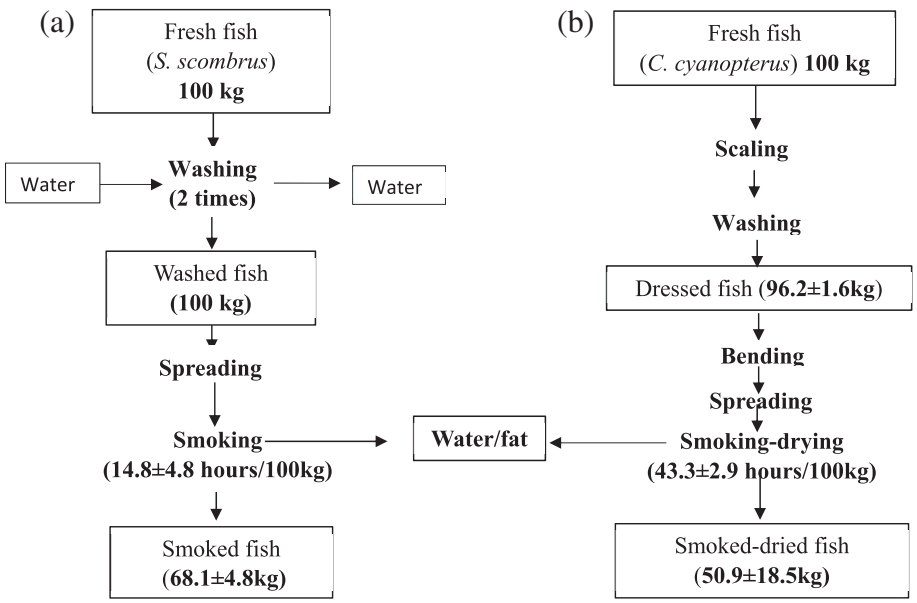

Figure 2. Flow diagram of smoking (a) and smoking-drying (b) with mass balance.

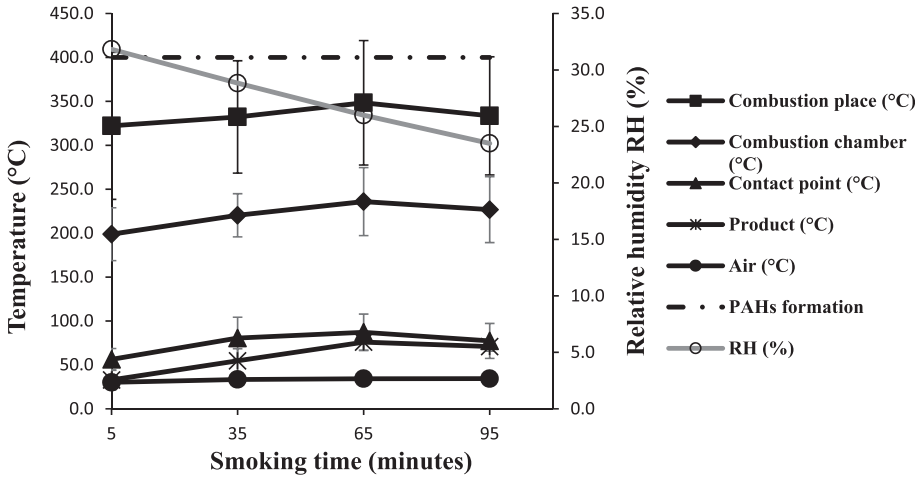

Figure 3. Changes in temperature and relative humidity during the smoking process. PAHs, polycyclic aromatic hydrocarbons; RH, relative humidity.

± 5.2 °C, respectively. The decrease of temperature was a result of the processor's intervention to reduce the fire intensity by adding water or by withdrawing wood from the combustion place aiming to avoid product carbonization and consequently preserving the market quality of the end product. The increase and decrease of combustion and contact point temperatures induced, respectively, an increase and decrease of the temperature at the core of the product. Thus, monitoring the temperature in the combustion place would allow control of the temperature at the core of fish and ensure good cooking and a good quality product. The temperature at the core of *S. scombrus* fish exceeded 70 °C during 30 min of the smoking process. It can therefore be assumed that the smoking conditions of fish could be assimilated to pasteurization with the possibility of the elimination of Gram-negative bacteria such as *Pseudomonas* spp., *Escherichia coli* and *Salmonella* spp.[7,20,32]

During all of the smoking-drying time of *C. cyanopterus* (210 min), the ambient temperature ranged between 33.3 ± 1.0 °C and 36.5 ± 3.3 °C without a significant change, whereas the air relative humidity decreased significantly from 33.7 ± 6.2% to 24 ± 4.2% (Fig. 4). Indeed, the air conditions observed during smoking-drying were not significantly different ($P > 0.05$) from those of the smoking process because of the use of the same barrel kiln. During the first step (i.e. smoking) of the smoking-drying process of *C. cyanopterus* (5–95 min), the combustion temperature increased from 300.2 ± 74.6 °C to 385.2 ± 64.5 °C with a maximal value of 482.8 °C (Fig. 4). Consequently, the average temperatures in the combustion chamber, at the contact point and at the core of the product, increased significantly from 174.5 ± 15.6 °C to 246.6 ± 17.0 °C, from 54.6 ± 15.5 °C to 87.9 ± 11.6 °C and from 36.2 ± 4.2 °C to 72.3 ± 10.4 °C, respectively. Similar to the smoking process of *S. scombrus*, the increase in temperature inside the barrel kiln and at the core of product during the first step (smoking) of the smoking-drying process of *C. cyanopterus* could also be explained by the increase of the intensity of wood combustion in the combustion place. From 95 to 215 min (end of the process), corresponding to the drying step, the average temperatures in the combustion place, the combustion chamber, at the contact point and at the core of the product decreased slowly to 287.3 ± 62.5 °C, 175.9 ± 23.0 °C, 59.3 ± 11.4 °C and 49.1 ± 13.3 °C, respectively. This phase (95–215 min) is considered by processors as the drying step during which the decrease of temperature is the result of the

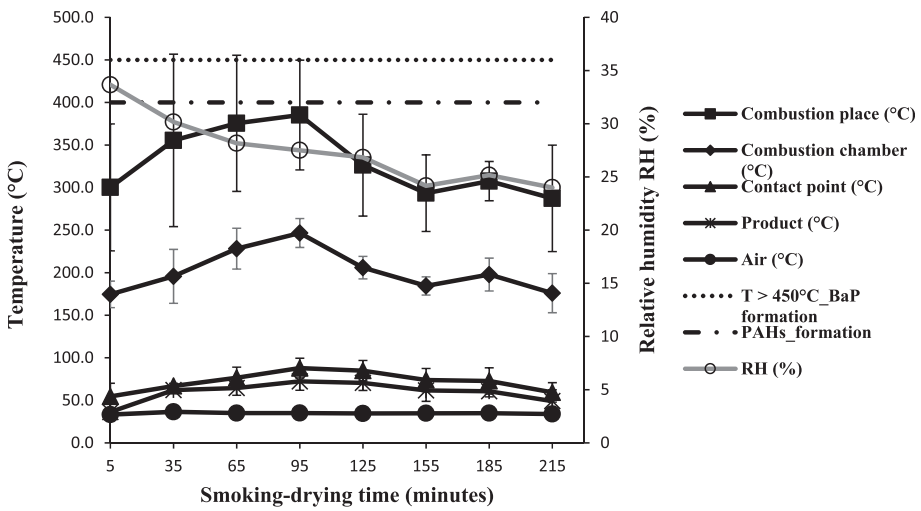

Figure 4. Changes in temperature and relative humidity during the smoking-dying process. PAHs, polycyclic aromatic hydrocarbons; BaP, benzo(a)pyrene; RH, relative humidity.

diminution of fire intensity by processors as a result of the removal some quantity of wood from the combustion place and sometimes by the addition of water to reduce the fire and heat transfer. During the drying step, the average temperature at the core of fish also exceeded 70 °C between 95 and 125 min, before progressively decreasing until reaching 49.1 ± 13.3 °C at 215 min.

The maximal values of combustion temperature recorded during the smoking process (5–95 min) of *S. scombrus* (456.4 °C) and *C. cyanopterus* (482.8 °C) were higher than 450 °C, which is the temperature of wood pyrolysis for which Horne and Williams[33] reported the formation and detection of some PAHs such as naphthalene, fluorene, phenanthrene, anthracene, pyrene, chrysene and benzo[a]pyrene. This high combustion temperature could result in exposure of smoked fish and smoked-dried fish to chemical contaminants such as PAH. Indeed, Toth and Blaas[34,35] observed a linear increasing of PAHs levels in the smoke with increasing combustion temperatures, from 400 to 1000 °C. Hajaligol *et al.*[36,37] also reported the formation of various aliphatic and aromatic hydrocarbons during pyrolysis between 350–600 °C when cellulosic materials such as solid char residues derived from cellulose, glucose, pectin and xylan were used. In addition, a number of studies[38-41] observed that a high temperature (> 450 °C) was sufficient for the formation of benzo(a)pyrene which is considered by European Food Safety Authority (EFSA) as one of the markers for the occurrence and toxicity of carcinogenic PAHs in food.[42]

Kinetics of mass loss of fish during smoking and smoking-drying

A significant decrease ($P < 0.05$) of product mass was observed after each 30 min of sampling during the smoking process and smoking-drying process (Fig. 5). During the two smoking processes (5–95 min), the mass of fish decreased from 100 kg to 61.9 ± 5.2 kg for *S. scombrus* and from 100 kg to 62.5 ± 7.9 kg for *C. cyanopterus*, corresponding to a total weight loss of 38.1 kg per 100 kg and 37.5 kg per 100 kg, respectively. Because no significant difference was found between the weight losses during smoking of *S. scombrus* and smoking of *C. cyanopterus*, it is assumed that the type of fish species did not influence the mass transfer phenomenon during the smoking process of fish. The raw

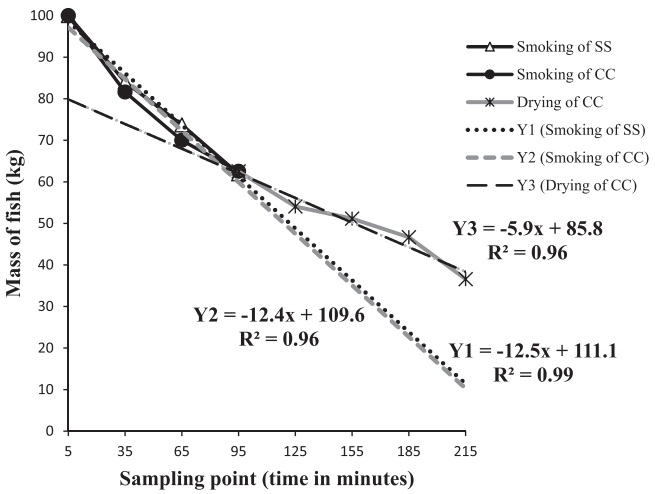

Figure 5. . Mass loss kinetic of fish during smoking and smoking-drying processes. Sampling was performed between 5 and 215 min of processing. Sampling point number 1 corresponds to 5 min of processing. Then, each sampling was performed at intervals of 30 min. SS, *Scomber scombrus*; CC, *Cypselurus cyanopterus*.

king fish (*S. scombrus*) had a moisture content of 70.1 ± 1.5 kg per 100 kg and a fat content of 9.7 ± 0.2 kg per 100 kg, whereas the smoked *S. scombrus* had a moisture content of 61.2 ± 3.4 kg per 100 kg and a fat content of 12.4 ± 0.6 kg per 100 kg. The water and fat losses during this process were evaluated as 36.1 ± 3.1 kg per 100 kg and 2.2 ± 0.6 kg per 100 kg, respectively. During the first step (smoking) of smoking-drying of *C. cyanopterus*, the raw flying fish had a moisture content of 75.7 ± 0.9 kg per 100 kg and fat content of 5.9 ± 0.1 kg per 100 kg, whereas the smoked fish of *C. cyanopterus* had a moisture content of 60.9 ± 10.6 kg per 100 kg and fat content of 8.6 ± 1.5 kg per 100 kg. Thus, the moisture and fat losses during the first step (smoking) of smoking-drying of *C. cyanopterus* were evaluated as 37.6 ± 4.8 kg per 100 kg and 0.5 ± 0.6 kg per 100 kg, respectively. These results showed that the moisture contents of smoked *S. scombrus* and smoked *C. cyanopterus* decreased, whereas their fat contents appeared to increase certainly as a result of moisture loss and concentration of fish dry matter.

During the second step (drying) of smoking-drying (95–215 min) of *C. cyanopterus*, it was observed that the mass of fish decreased from 62.5 ± 7.9 kg to 36.6 ± 5.5 kg. The moisture content also decreased from 60.9 ± 10.6 kg per 100 kg to 48.3 ± 12.7 kg per 100 kg, whereas the fat content appeared to increase from 8.6 ± 1.5 kg per 100 kg to 11.1 ± 1.1 kg per 100 kg. The moisture and fat losses during the drying step were evaluated as 20.4 ± 2.7 kg per 100 kg and 1.3 ± 0.6 kg per 100 kg, respectively. Thus, all the smoking-drying process of *C. cyanopterus* led to water and fat losses evaluated as 58.0 kg per 100 kg and 1.8 kg per 100 kg, respectively.

As shown by the regression line equations, Y1 and Y2, which are overcome (Fig. 5), the rate of mass loss during smoking of *S. scombrus* (12.5 kg per 30 min) is similar to that obtained during the first step (smoking) of smoking-drying of *C. cyanopterus* (12.4 kg per 30 min). The similarity of the rates of mass loss during the smoking of the two fish species is justified by the use of the same barrel kiln, wood fuel and duration. At the opposite, the rate of mass loss of *C. cyanopterus* during the second step (drying) of smoking-drying shown in the regression line equation Y3 (5.9 kg/30 min) was twice as low compared to those recorded during the smoking process. This significant decrease in the rate of mass loss of *C. cyanopterus* during the drying step of smoking-drying could be explained by the fact that the maximal water in this fish was lost during the smoking step. In addition, the modification of the histologic structure of the fish tissue as a result of protein aggregation could decelerate the water loss of fish.

Technological performances of the barrel kiln during smoking and smoking-drying of fish

The main technological parameters recorded during smoking and smoking-drying processes using the barrel kiln are shown in Table 2. The load capacity of the barrel kiln was evaluated as 14.6 ± 4.7 kg for fresh *S. scombrus* and to 8.1 ± 0.6 kg for *C. cyanopterus*. The smoking of fresh *S. scombrus* lasted an average time of 1.4 ± 0.2 h corresponding to 11.1 ± 4.3 kg of fresh fish processed every 1 h with a specific fuel consumption estimated as 0.38 ± 0.18 kg kg^{-1} (kg of wood per kg of product). As regards the smoking-drying of fresh *C. cyanopterus*, this lasted for an average duration of 3.5 ± 0.2 h corresponding to 2.4 ± 0.2 kg of fresh fish processed every 1 h with a specific fuel consumption of 0.93 ± 0.29 kg kg^{-1}. The lengthy time of the smoking-drying process could explain the high specific fuel consumption needed.

Table 2. Technological performances of the barrel kiln (mean ± SD)

Technological performances	Types of process: Smoking of SS ($n = 6$)	Types of process: Smoking-drying of CC ($n = 6$)
Load capacity (kg of fresh fish)	14.6 ± 4.7	8.1 ± 0.6
Duration (h)	1.4 ± 0.2	3.5 ± 0.2
Quantity of fresh fish processed per time unit (kg h^{-1})	11.1 ± 4.3	2.4 ± 0.2
Specific fuel consumption (kg kg^{-1} of fresh fish)	0.38 ± 0.18	0.93 ± 0.29
Yield (%)	68.1 ± 4.8	51.0 ± 18.5

SS, *Scomber scombrus*; CC, *Cypselurus cypnopterus*.

Changes in the physicochemical characteristics of fish

The physicochemical characteristics of the fresh, smoked and smoked-dried fish are presented in Table 3. The pH values of the smoked *S. scombrus* (6.5 ± 0.3) and smoked-dried *C. cyanopterus* (6.8 ± 0.1) were not significantly different ($P > 0.05$) from the pH values recorded for the fresh fishes (6.3 ± 0.0 and 6.2 ± 0.1, respectively). Therefore, the smoking and smoking-drying processes did not influence the pH of the end products. On the other hand, the smoking process induced a significant ($P < 0.05$) change in colour parameters (L^* and ΔE) of *S. scombrus*, whereas the smoking-drying process increased significantly ($P < 0.05$) the red and yellow colour parameters (a^* and b^*) of *C. cyanopterus*. The decrease of the lightness parameter (L^*) could be explained by the occurrence of the Maillard reaction and the deposition of phenolic compounds from smoke on the surface of the products during the smoking process. The concomitant increase of red (a^*) and yellow (b^*) colour index after the smoking-drying process of *C. cyanopterus* could justify the golden colour of the end product as appreciated by fish processors.[19] It shoud be to noted that smoked *S. scombrus* is a product with a high water content (61.2 ± 3.4%) and this is a major constraint for its preservation. Also for *S. scombrus* and *C. cyanopterus*, it was observed that the smoking and smoking-drying processes did not significantly influence ($P > 0.05$) the protein and fat contents (dry weight) of the end-products. The protein content (dry weight) recorded in *S. scombrus* (63.1 ± 4.6% and 61.9 ± 1.5%, for fresh and smoked fish, respectively) was higher than that reported (44.3 ± 0.5% and 39.4 ± 0.4%, respectively) by Aminullah *et al.*[43] for the same species caught in low season, whereas the fat content (dry weight) recorded (32.5 ± 1.3% and 32.0 ± 1.4% for fresh and smoked fish, respectively) was much lower than that (52.2 ± 0.7% and 48.6 ± 0.6%, respectively) reported by Aminullah *et al.*[43] The differences in protein and fat contents between the results reported in the previous study by Aminullah *et al.*[43] versus those of the present study could be a result of the fresh fish (*S. scombrus*) used for the two studies not originating from the same area, not being caught during the same season, not having the same nutritional food supply and not being submitted to the same storage conditions, which can cause different levels of alteration.

Changes in biogenic amines content

The biogenic amines concentrations of fish samples are presented in Tables 4 and 5. The raw fish, the smoked fish and the smoked-dried fish contained at least one biogenic amine. Histamine in the raw king fish (*S. scombrus*) was below the LOQ (< 10 mg kg^{-1}), whereas the concentration of histamine in the raw flying fish (*C. cyanopterus*) ranged between 118 and 142 mg kg^{-1} with an average of 127.5 ± 12.6 mg kg^{-1} (Table 5). All samples of raw flying fish had a histamine concentration exceeding 100 mg kg^{-1}, comprising the maximal limit of EU and Benin for fish and fishery products.[44] The contamination of the raw flying fish might be a result of fish contamination with histidine-decarboxylase producing bacteria, which can occur on the vessel after fish capture.[45] The raw, smoked and smoked-dried fish samples collected during the present study showed high contamination with respect to aerobic mesophilic bacteria for which 66.7% of the samples were not in compliance with the acceptable limit (< 7 $\log_{10}$ CFU g^{-1}) fixed by the Health Protection Agency.[32] The presence of aerobic mesophilic bacteria in fish producing free amino acid decarboxylase could explain the histamine content found in fish. Altieri *et al.*[46] also reported high histamine levels in fresh tuna (478 mg kg^{-1}) and in fresh anchovy (> 720 mg kg^{-1}) during the European official control of food. The histamine concentration of the smoked and smoked-dried flying fish found in this study

Table 3. Physicochemical characteristics of fishes (mean ± SD)

Parameters	*Scomber scombrus*: Fresh ($n = 3$)	*Scomber scombrus*: Smoked ($n = 6$)	*Cypselurus cyanopterus*: Fresh ($n = 3$)	*Cypselurus cyanopterus*: Smoked ($n = 6$)	*Cypselurus cyanopterus*: Smoked-dried ($n = 6$)
pH	6.3 ± 0.0 a	6.5 ± 0.3 a	6.2 ± 0.0 a	6.5 ± 0.1 a	6.8 ± 0.1 a
L^*	78.3 ± 5.3 a	69.9 ± 1.6 b	72.8 ± 0.9 a	71.8 ± 0.8 a	71.7 ± 1.1 a
a^*	2.9 ± 0.8 a	9.3 ± 9.3 a	3.6 ± 0.0 a	5.4 ± 0.8 b	4.7 ± 0.5 b
b^*	4.1 ± 2.2 a	3.8 ± 1.7 a	1.4 ± 0.6 a	3.4 ± 0.9 b	2.5 ± 0.8 ab
ΔE	9.5 ± 0.7 a	12.8 ± 1.8 b	11.8 ± 0.8 a	11.5 ± 0.7 a	11.8 ± 1.1 a
Dry matter (%)	29.9 ± 1.5 a	38.8 ± 3.4 b	24.3 ± 0.9 a	39.1 ± 10.6 ab	51.7 ± 12.7 b
Protein (%)	63.1 ± 4.6 a (18.8 ± 0.5 a)*	57.1 ± 4.9 a (22.0 ± 0.8 b)	66.5 ± 2.3 a (16.2 ± 0.8 a)	61.0 ± 9.7 a (22.9 ± 2.1 b)	63.5 ± 7.6 a (32.1 ± 3.7 c)
Fat (%)	32.5 ± 1.3 a (9.1 ± 0.2 a)	31.9 ± 1.4 a (12.4 ± 0.6 b)	24.6 ± 0.9 a (5.9 ± 0.1 a)	22.4 ± 1.9 a (8.6 ± 1.5 b)	22.0 ± 3.0 a (11.1 ± 1.1c)

*Values in brackets represent the percentage on a wet basis; values with different lowercase letters in the same row limited to each fish species (*S. scombrus* and *C. cyanopterus*) are significantly different at the α threshold value of 5%.

Table 4. Individual concentration (mg kg^{-1}) of biogenic amines in smoked fish and smoked-dried fish

Samples	Methylamine	Tryptamine	2-phenylethylamine	Putrescine	Cadaverine	Histamine	Serotonine	Tyramine	Spermidine	Spermine
1	17.1	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	10.4	22.5
2	< LOQ	13.2	< LOQ	< LOQ	< LOQ	< LOQ	10.9	< LOQ	11.0	23.1
3	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	11.5	24.4
4	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	7.5	19.8
5	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	2.5	< LOQ
6	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	11.0	23.6
7	< LOQ	41.2	< LOQ	< LOQ	< LOQ	< LOQ	16.4	< LOQ	10.4	20.0
8	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	14.6	34.6
9	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	8.4	18.0
10	14.2	8.8	< LOQ	< LOQ	33.4	122.6	< LOQ	< LOQ	16.6	43.8
11	< LOQ	9.0	< LOQ	< LOQ	60.4	118.1	< LOQ	60.1	8.2	24.0
12	< LOQ	111.5	6.2	615.9	9.0	141.8	11.8	397.5	42.5	57.8
13	< LOQ	< LOQ	< LOQ	< LOQ	144.8	1139.4	< LOQ	< LOQ	24.1	65.4
14	< LOQ	< LOQ	0.1	0.1	107.6	549.8	< LOQ	18.5	18.5	47.7
15	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	101.9	< LOQ	< LOQ	20.7	58.3
16	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	41.3	< LOQ	< LOQ	15.4	48.9
17	< LOQ	304.0	52.6	2224.0	59.7	676.1	3.7	1766.5	29.9	18.3
18	< LOQ	180.9	34.9	855.4	42.0	321.9	14.9	647.8	93.8	115.0
19	< LOQ	< LOQ	< LOQ	4.7	12.7	260.9	3.8	< LOQ	24.1	65.5
20	< LOQ	< LOQ	< LOQ	71.2	357.8	77.4	18.6	20.6	50.0	131.5
21	< LOQ	17.3	< LOQ	70.4	319.4	82.4	11.8	< LOQ	29.1	61.4
22	< LOQ	< LOQ	< LOQ	60.3	282.6	115.9	21.5	17.5	49.6	147.9
23	< LOQ	37.1	< LOQ	< LOQ	99.8	2255.1	26.4	< LOQ	25.5	71.5
24	< LOQ	36.2	< LOQ	< LOQ	102.1	1734.0	26.8	< LOQ	26.5	72.2

Sample numbers 1–3: fresh *Scomber scombrus*; 4–9: smoked *Scomber scombrus*; 10–12: fresh *Cypselurus cyanopterus*; 13–24: Smoked (13–18) and smoked-dried (19–24) *Cypselurus cyanopterus*. LOQ ranged between 1.5 and 10 mg kg^{-1} depending on biogenic amines.

ranged between 41.3 and 2255.1 mg kg^{-1} (Table 4) with nine samples having a histamine concentration > 100 mg kg^{-1}. Moreover, cadaverine and putrescine were found in most samples in which histamine was found. No significant differences ($P > 0.05$) were recorded for histamine concentrations of flying fish before smoking (raw fish: 127.5 ± 12.6 mg kg^{-1}), after smoking (smoked fish: 471.7 ± 409.4 mg kg^{-1}) and after smoking-drying (smoked-dried fish: 754.3 ± 977.0 mg kg^{-1}) (Table 5). This means that the histamine concentration found in the processed fish is mainly a result of the histamine concentration in raw fish. However, poor handling practices at the post smoking and post smoke-drying steps may lead to a recontamination with histidine-decarboxylase producing bacteria. Thus, it is useful to respect good hygiene and good handling practices after processing aiming to preserve the safety of the processed fish.

Table 5. Concentration (mg kg^{-1}) of histamine in raw and processed *Scomber scombrus* and *Cypselurus cyanopterus* (mean ± SD)

Species of fish	Histamine concentration (mg kg^{-1})
Scomber scombrus	
Raw fish ($n = 3$)	< 10 a
Smoked fish ($n = 6$)	< 10 a
Cypselurus cyanopterus	
Raw fish ($n = 3$)	127.5 ± 12.6 a
Smoked fish ($n = 6$)	471.7 ± 409.4 a
Smoked-dried ($n = 6$)	754.3 ± 977.0 a

Values with the same lowercase letters according to each species of fish are not significantly different ($P > 0.05$); *n*, number of samples analysed.

Effect of a traditional barrel kiln on levels of PAHs in smoked and smoked-dried fishes

PAH contents in raw fish

Raw fish contained very low levels of individual PAHs concentration (< 1 μg kg^{-1}) with most results being below the LOQ (data not shown). Before smoking, the mean BaP levels in raw *S. scombrus* and *C. cyanopterus* were 0.1 ± 0.0 μg kg^{-1} and < 0.1 μg kg^{-1}, respectively (Table 6). Fish and marine organisms may naturally contain a low quantity of different PAHs absorbed from a polluted environment.[39,47,48] Soclo *et al.*[47] reported a BaP concentration ranging between 1.1 and 3.3 μg kg^{-1} (dry weight) in raw fish species collected from Cotonou Lagoon in Benin, namely *Pseudotolithus senegalensis*, *Tilapia* sp., *Drepane africana* and *Lutjanus flugens*. Essumang *et al.*[49] reported similar results in mackerel, a marine fish from Ghana.

PAH contents in fish after smoking

PAHs concentrations in smoked fish and smoked-dried fish are shown in Tables 6 and 7. The BaP concentration of smoked fish and smoked-dried fish ranged between 2.8 and 52.7 μg kg^{-1}, whereas the PAH4 (sum of BaP, CHR, BbF and BaA) concentration ranged between 16.5 and 290.9 μg kg^{-1} (Table 7). None of the samples of smoked fish and smoked-dried fish met the EU regulation that set maximal limits of BaP and PAH4 at 2 and 12 μg kg^{-1}, respectively,[50] whereas 78% of samples were not compliant with

Table 6. Comparison of individuals and the sum of PAH4 concentrations (μg kg^{-1}) of raw (Control), smoked fish and smoked-dried fish from different species (mean ± SD)

Type of fish	PAHs concentration (μg kg^{-1} wet weight)				
	BbF	BaA	CHR	BaP	∑PAH4
Scomber scombrus					
Raw fish ($n = 3$)	< 0.1 a	0.7 ± 0.1 a	0.3 ± 0.0 a	0.1 ± 0.0 a	1.2 ± 0.0 a
Smoked fish ($n = 6$)	6.7 ± 3.4 b	19.2 ± 6.5 b	21.2 ± 8.6 b	**5.6 ± 2.4 b**	**52.6 ± 20.4 b**
Cypselurus cyanopterus					
Raw fish ($n = 3$)	< 0.1 a	< 0.1 a	0.1 ± 0.0 a	< 0.1 a	0.1 ± 0.0 a
Smoked fish ($n = 6$)	17.6 ± 14.7 b	23.5 ± 32.7 b	26.0 ± 30.1 b	**23.0 ± 19.3 b**	**90.1 ± 93.3 b**
Smoked-dried fish ($n = 6$)	24.9 ± 14.4 b	44.9 ± 31.7 b	53.1 ± 28.7 b	**30.9 ± 16.2 b**	**153.8 ± 85.8 b**
Smoked fish ($n = 6$), DW	37.7 ± 36.8 A	56.1 ± 89.0 A	60.1 ± 82.7 A	**49.5 ± 48.7 A**	**203.3 ± 253.6 A**
Smoked-dried fish ($n = 6$), DW	37.3 ± 17.6 A	66.6 ± 36.6 A	79.9 ± 31.5 A	**46.3 ± 18.7 A**	**230.2 ± 94.0 A**

Value with different letters (lowercase and uppercase) in the same column for the same species are significantly different ($P < 0.05$).
DW, dry weight; BaP, benzo(a)pyrene; CHR, chrysene; BbF, benzo[b]fluoranthene; BaA, benz[a]anthracene.

the Benin regulation. The maximal levels of BaP and PAH4 exceeded the EU maximal limits approximately 25 times. The significant differences ($P < 0.05$) recorded between individual and the sum of PAH4 concentrations of the raw and smoked fish for both species (Table 6) indiacated that the smoking process was the main source of smoked fish contamination. However, no significant difference ($P > 0.05$) was recorded between individual and the sum of PAH4 concentrations of the smoked fish and the smoked-dried fish from the same species (*C. cyanopterus*) for fresh and dry weight (Table 6). This indicates that the drying process following smoking did not involve the extra contamination of fish. Kpoclou *et al.*[20] reported a high BaP and the sum of PAH4 contamination in smoked shrimp processed with the barrel kiln using Acacia wood with BaP (91.8 ± 21 μg kg^{-1}) and PAH4 (481.7 ± 62.6 μg kg^{-1}) concentrations exceeding EU maximum limits. Amos and Sambo[51] also reported the effect of traditional smoking on fish contamination with PAHs. Kpoclou *et al.*[20] and Essumang *et al.*[50] reported that Acacia wood used during the follow-up trials in the present study had significant lignin content. According to the guidelines of the Codex Alimentarius Commission, a high lignin content in fuel involved a high amount of PAH.[13] Additionally, high levels of BaP (from 35.3 to 1489.6 μg kg^{-1}) were also reported in some fish species traditionally smoked in Sri Lanka.[52] An improvement of the smoking technique based on the type of fuel, smoking time, smoking temperature and type of kiln (new or improved) could be investigated aiming to control and limit the contamination of smoked and smoked-dried fish by PAHs, as suggested by Iko Afé *et al.*[53]

Table 7. Minimum, median and maximum PAH contents (μg kg^{-1}) in 18 samples of smoked fish and smoked-dried fish

PAHs	Minimum	Maximum	Median
BbF	**2.5**	**47.3**	**12.7**
DlP	< 0.1	1.2	0.4
DhA	0.4	8.3	2.1
BgP	1.8	35.5	7.5
DeP	0.3	7.3	1.6
BjF	1.9	33.0	7.8
BcL	2.4	63.5	15.9
BaA	**5.1**	**104.6**	**19.1**
CHR	**5.2**	**101.6**	**23.3**
5MC	< 0.1	7.8	0.7
BkF	1.4	20.1	5.1
BaP	**2.8**	**52.7**	**13.2**
IcP	1.6	25.7	8.0
DiP	< 0.1	7.0	0.6
DhP	< 0.1	1.3	0.2
PAH4	**16.5**	**290.9**	**67.2**

PAH4: Sum of benzo(a)pyrène (BaP), benz(a)anthracène (BaA), benzo(b)fluoranthène (BbF) and chrysene (Chr).

CONCLUSIONS

The technological performances of the barrel kiln during smoking and smoking-drying process were evaluated. A significant change in temperatures was observed at different levels with respect to the barrel kiln and processed fish. The extreme values of temperature recorded at the combustion place during the two processes attained a threshold value favorable to formation of PAHs (450 °C) indicating a risk of PAH deposition on the fish during processing. As a result, concentrations of BaP and PAH4 were much higher than the maximal limit values fixed by standard regulations. The smoking and smoking-drying processes significantly influenced some mechanical and physicochemical characteristics of the end products. All of the information collected throughout the present study could be used to improve the processing steps, as well as design and manufacture an improved kiln that must take into account the technological parameters for controlling and limit PAH contamination and ensuring good sensory characteristics of the end-product.

CONFLICT OF INTERESTS

The authors declare that they have no conflicts of interest.

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