CHANGES IN PHYSICOCHEMICAL AND SENSORY CHARACTERISTICS OF SMOKE-DRIED FISH SPECIES STORED AT AMBIENT TEMPERATURE

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ABSTRACT

This study assessed the comparative changes in the physical and chemical components of five different species of smoked freshwater fish: Bony tongue, *Heterotis niloticus,* African carp, *Labeo coubie,* Snake fish, *Parachanna obscura,* Nile Tilapia, *Oreochromis niloticus* and African mud catfish, *Clarias gariepinus* during storage. The fish were smoke-dried to average moisture content of $10.41 \pm 0.02\%$ and stored. Fish were packaged in black polythene bags and kept in perforated plastic containers. The fish were left in the plastic baskets for 56 days at ambient temperature (25-32°C).

Samples of fish were assessed weekly for physical attributes such as colour, fragmentation, odour, taste and texture. Proximate composition was assessed using changes in moisture content, crude protein, lipid and ash content. Biochemical indexes carried out were: Total Volatile Nitrogen (TVN), pH, Peroxide Value (PV) and Free Fatty Acid (FFA) levels. There was a general decline in physical attributes i.e. colour, fragments or cracks, odour, taste and texture of fish during storage. Fluffy woolly mat of moulds were noticed on the *Clarias gariepinus* from the 5th week of storage. There was a significant ($P < 0.05$) colour change in most species as from the 6th week. During this study, the moisture content increased weekly in the five smoked fish species from the initial average of $10.41 \pm 0.02\%$. This could be attributed to the difference in the moisture of the smoked fish relative to the surroundings.

Oreochromis niloticus and *Heterotis niloticus* had the best taste value. Apart from *Parachanna obscura*, the other fatty species, *C. gariepinus* and *L. coubie* became less firm as the weeks progressed. There were significant changes ($P < 0.05$) in most of the physical and chemical characteristics except odour from the 6th week (42 days) of storage. There were also significant differences ($P < 0.05$) between the initial and final values of the proximate and chemical constituents of the different species of fish. The study showed that keeping quality of smoked fish under ambient conditions decreases with increase in length of storage.

Keywords: Freshwater fish, hot smoking, storage

INTRODUCTION

The processing of a fish species inevitably entails a storage period for the finished product prior to marketing and consumption. Since fish are composed of perishable nutrients, storage period should be kept to a minimum with adequate storage conditions provided so as to prevent deteriorative changes occurring through oxidative damage and/or microbial, insect or rodent infestation. The most important environmental factors governing the storage or shelf life of fish are ambient temperature and humidity. These factors dictate the rate at which chemical changes take place. Smoking is one of the traditional fish processing methods aimed at preventing or reducing post-harvest losses.

Post-harvest losses in fish are represented by a net reduction in the amounts of nutrients potentially available to the consumer either by direct physical loss or nutritional loss. These factors have effect on consumer acceptability, commercial value and income of fish farmers/traders [1]. Also, the health implication of consuming spoilt fish cannot be quantified. The short shelf life of dead fish is due to changes in the chemical constituents of fish after death. Smoking enhances flavour and increase utilisation of the fish. Nonetheless, deterioration and spoilage still occur in smoked fish during storage. This study is therefore aimed at assessing the physical and chemical changes in stored smoked fish.

Furthermore, fish is an important protein food in the tropics. In Nigeria, fish constitutes 40% of the animal protein intake of the people [2]. But 40% of the total fish catch in Nigeria are lost annually due to inadequate or poor preservation, processing and handling [3].

The rate of fish spoilage depends on handling during processing, acidity level, species of fish, weather, mode of storage and temperature during transportation [4]. Chemical breakdown of protein, fat and water contents contribute to quick spoilage of fish.

Some preservation techniques currently used in the tropics include chilling, freezing, drying, salting and smoking. However, in Nigeria the most affordable and widely used method of fish preservation is smoking [5]. Meanwhile, the use of smoke from smouldering wood for the preservation of fish dates back to civilization [4]; it is one of the oldest means of preserving foods, especially fish.

MATERIALS AND METHODS

Five live fish species were purchased from the fish market at Akure, Ondo State, Nigeria. The species were *Heterotis niloticu*s (Cuvier, 1829), *Labeo coubie* (Ruppell, 1832), *Parachanna obcura* (Gunther, 1861), *Oreochromis niloticus* (Linne, 1758) and *Clarias gariepinus (*Burchell, 1822).

The fish were gutted, washed, cut into chunks (50 g) and soaked in a 7.5% brine solution (75 grams Sodium chloride in 1 litre of water), drained and placed on wire guaze. Smoking was done using red-hot charcoal obtained from Gmelina (*Gmelina aborea)* wood. The fish chunks were turned at intervals and smoked to an average moisture content of 10.41±0.02%. The smoked fish samples were kept in covered plastic baskets, under laboratory conditions with

no preservative. Physical and chemical evaluations were carried out at the beginning of the experiment (week 0) and at weekly interval thereafter for 56-day period.

Physical assessment was carried out using a five-man trained panel through a 10-point hedonic scale (grading sheets) to evaluate changes in colour, odour, texture, taste and level of fragmentation. Proximate analysis (moisture, ash, crude protein and fat content) was carried out according to Association of Official Analytical Chemists (A.O.A.C) methods of analysis [6]. Chemical changes in stored fish were assessed using pH, total volatile nitrogen, peroxide value and free fatty acid levels. The pH was determined using Jenway pH meter-3015 model, while Total Volatile Nitrogen (TVN), Peroxide Value (PV) and Free Fatty Acids (FFA) were determined using methods described by Pearson [7]. In each analysis, an average of triplicate trials or values was recorded.

Data Analysis

Analysis of Variance (ANOVA) was carried out on all the physical and chemical parameters measured to test for variability at 5% level of significance. Duncan Multiple Range Test was used to separate means. Statistical Package for Social Science (Version 10.00) was used [8].

RESULTS

Chemical analyses

The results of pH, TVN, PV and FFA analyses are presented in Figures 1-4. The pH values decreased over the weeks and all the species became more acidic.

Total Volatile Nitrogen increased during storage. The Peroxide value which is a primary indicator of oxidation of fat (rancidity) increased weekly ranging from 21.3 to 27.7 ml/Kg at the end of the 56-day storage period.

Lipid loss was inversely proportional to the initial fat content (Table 2). In this study, the percentage FFA for the five fish species at the end of the 56 days of storage ranged between 0.91-1.96%, showing a high level of FFA production.

Physical Parameters

The result of the physical assessment is presented in Table 1. There was a general decline in the physical attributes such as colour, fragmentation, odour, taste and texture of fish during storage. Fluffy woolly mat of moulds was noticed on *Clarias gariepinus* from the 5th week of storage. There was a significant colour change in most species as from the 6th week. *Oreochromis niloticus* and *Heterotis niloticus* had the best taste while *L. coubie* had the poorest. Apart from *Parachanna obscura*, the other fatty species, *C. gariepinus* and *L. coubie* became less firm as the weeks progressed.

Table 2 shows the mean proximate composition of the five fish species during storage. In this study, crude protein and fat content reduced while the ash and moisture percentage increased

during storage. The moisture content increased weekly in the five smoked fish species from the initial average of 10.41±0.02% to 10.62±0.05%. *Oreochromis niloticus* had the highest final moisture content of 10.62%. This species had the least fragmentation. Highest fragments/cracks were observed in *Heterotis niloticus*. Percentage lipid content was observed to decrease over time while crude protein formed the largest quantity of the dry matter in all the samples.
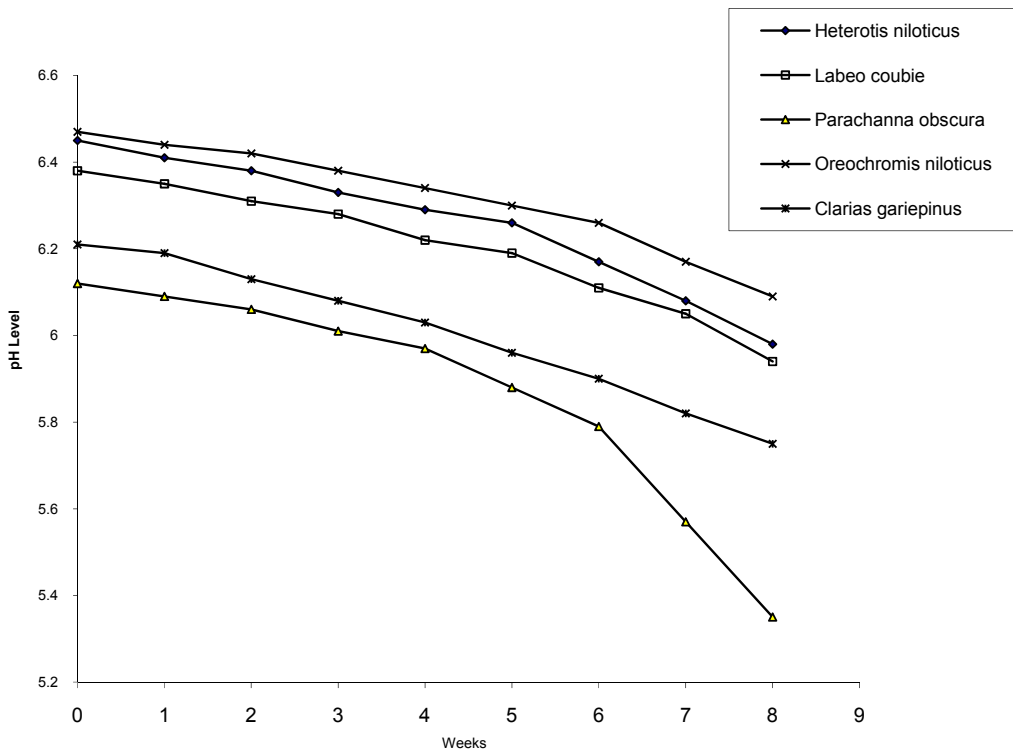

Fig.1: Weekly Changes in pH level of the smoked fish species during storage

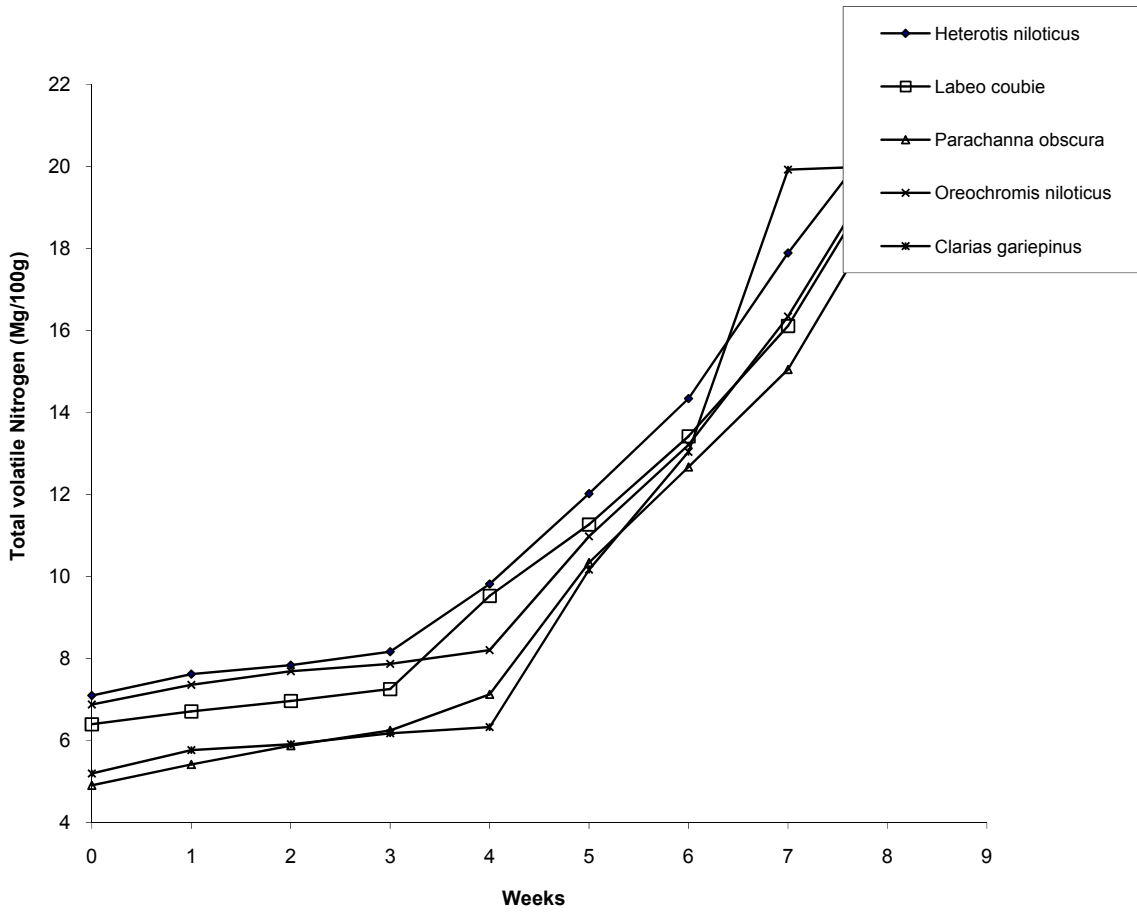**Fig. 2: Weekly Changes in TVN of the smoked fish species during storage**
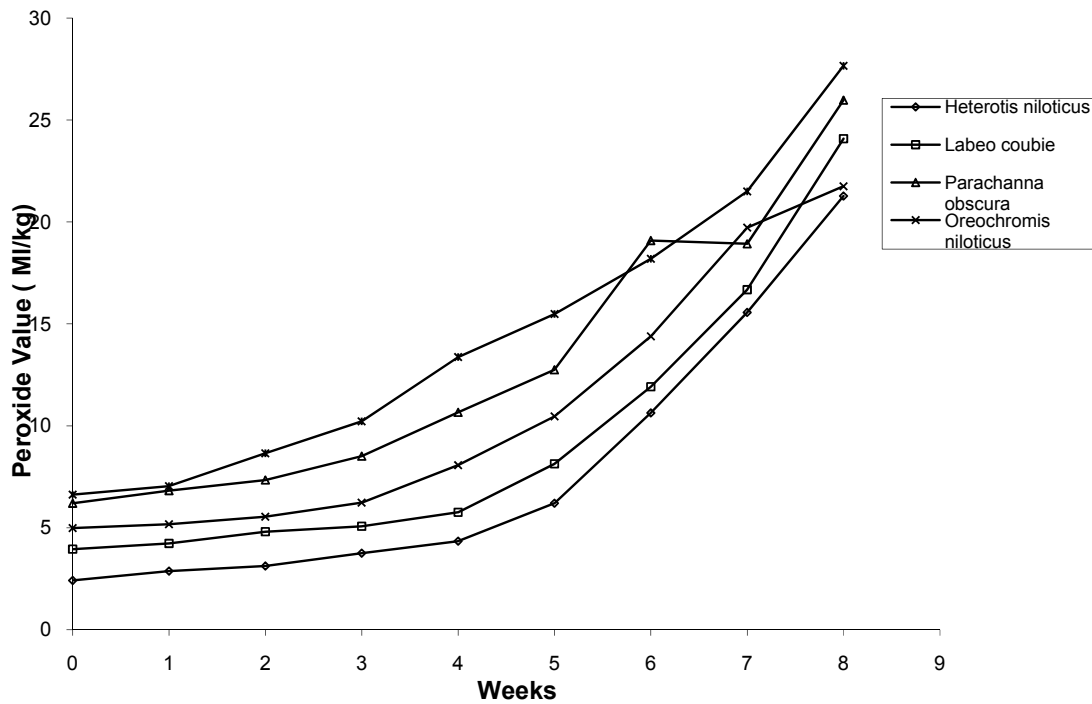
Fig.3: Weekly Changes in Peroxide values of the smoked fish species
during storage

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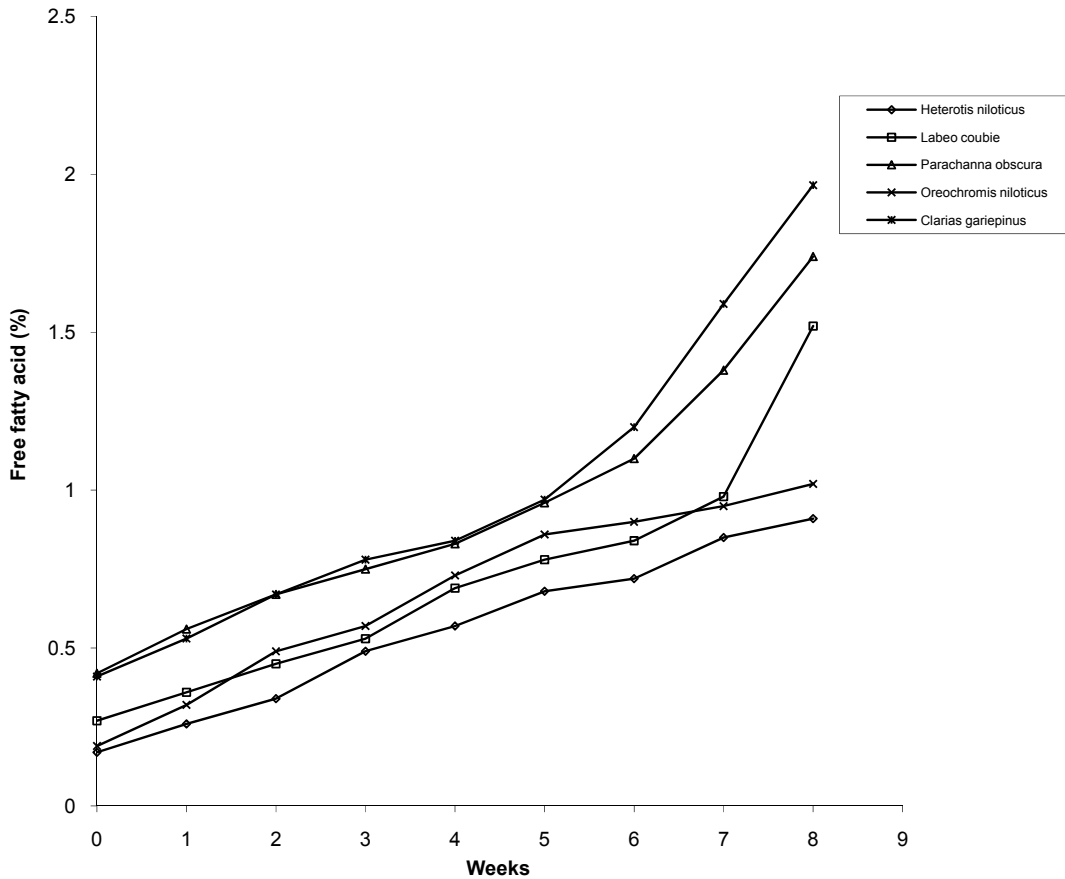
Fig. 4: Weekly Changes in Free fatty acid of the smoked fish species during storage

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DISCUSSION

The results from the physical assessments corroborated the assertion of Sefa-Dedeh [9] that differences between fish species may be reflected in the quality of the smoked fish. The reduction in the physico-chemical qualities with increasing storage period could be attributed to higher activities of the spoilage agents. Results of research into storage of crustaceans (Oyster and Shrimps) revealed quality loss during storage both at ambient temperature and chilling [10, 11]. After storing the smoked fish for five weeks, attacked by moulds was evidenced in *Clarias gariepinus*, a fatty fish. This is known to have the potential of causing mycotoxin production.

The water activity determines the storage life of fish. Smoking decreases the water activity in fish tissue [12]. If not properly stored the efforts involved in smoking would not yield the expected preservative effect. However, the result of the proximate composition (Table 2) shows increase in the moisture content of the fish on storage. Hence, it may be assumed that the fish species used in this study absorbed moisture during storage. Therefore, storing smoked fish at low initial moisture content (<20%) under ambient conditions ($25^{o}C$) would not be advisable or considered as proper storage. Moisture content of 12% is the level beyond which fish products begin to grow moulds after few days [13]. The final moisture of all the species of fish under study was less than 12%. Furthermore, apart from temperature at the point of storage and storage conditions, the species of fish should also need to be considered. Meanwhile, the different arrangements of fish muscles vis-a-vis free/bound waters and the fat content of fish species could be important factors to storage. Also, the species of fish used or fat content could have been responsible for the presence of moulds.

In the mean proximate composition (Table 2), the crude protein formed the largest quantity of the dry matter in all the fish samples. This is in-line with the report that protein forms the largest quantity of dry matter in fish [14]. There was reduction in the percentage of crude protein of the species during the period of storage. This could be due to gradual degradation of the initial crude protein to more volatile products such as Total Volatile Bases (TVB), Hydrogen sulphide and Ammonia [15]. Changes observed in protein and lipid content during storage may have been due to leaching out of some extractable soluble protein fraction and hydrolysis of some of the lipid fractions [16].

Increase in moisture content could be attributed to the difference in the moisture of the smoked fish relative to the surroundings. Fish at 10-15% moisture content, reportedly had a shelf life of 3-9 months when stored properly [17]. Reduction in lipid content could be attributed to oxidation of poly-unsaturated fatty acids (PUFA) contained in the fish tissue to products such as peroxides, aldehydes ketones and the free fatty acids [18]. However, the rate of fat deterioration was very gradual. Fish oil has been found to be more liable to spoilage than other oils due to their greater number of unsaturated fatty acids as shown by

the lower specification number and higher iodine value [5]. The greater the degree of un-saturation, the greater would be the tendency for fat oxidation (rancidity). There might be high risks of rancidity during prolonged storage conditions due to the fatty nature of fish [18]. In the freshly smoke–dried fish except *Heterotis niloticus*, the crude protein was lower than values recorded for these species even at higher moisture content of between 11.2-12.8% [19]. This difference could be due to the size of the fish and their nutritional status of the raw samples. The trend was reversed in fat content with *Heterotis niloticus* having lower value. Protein is inversely proportional to fat content in fish. *Oreochromis niloticus* and *Clarias gariepinus* had higher ash percentage while the three other species had lower values compared with the findings of Ikeme [20]. Smaller sized fish specie has higher ash content due to the higher bone to flesh ratio.

Furthermore, Eyo [5] stated that pH is an indicator of the extent of microbial spoilage in fish and that some proteolytic microbes produce acid after decomposition of carbohydrate, thereby increasing the acid level of the medium. The pH value is a reliable indicator of the degree of freshness or spoilage. Decrease in the pH level is due to the fact that carbohydrate of the fish was fermented to acids.

The Total Volatile Nitrogen increased during storage. The ratio of Volatile Basic Nitrogen to the Total Nitrogen has been recommended as a useful index of quality in fish [21]. Pearson [7] recommended that the limit of acceptability of fish is 20-30mg N per 100g while Kirk and Sawyer [22] suggested a value of 30-40mg N per 100g as the upper limit. Also the limit of acceptability of fish is reported to be 30mg N per 100g by Connell [23]. Beyond this level, white fish and prawns are regarded as unacceptable. However, result from this study shows that stored smoked fish species still have their final TVN within acceptable limits, since they all have values less than 30 mg N per 100g. Increase in final values of TVN in this study is similar to the result of Trinidad and Estrada [24] who reported an increase in the value of TVN produced in smoked Tilapia fish from 4.2 mg to 11.00 mg N per 100g in less than fifteen hours. In this study, the highest value of Total Volatile Nitrogen (21.03 mg N per 100g) was in *Heterotis niloticus* in the 8th week.

The Peroxide value which is a primary indicator of oxidation of fat (rancidity) increased weekly ranging from 21.3 to 27.7ml per Kg at the end of the 56-day storage period. The peroxide values corresponding to incipient spoilage are usually in the order of 20-40 milliequivalents of oxygen per Kg of sample (ml per Kg). However, Connell [23] reported that when peroxide value is above 10-20, fish develop rancid taste and smell. Thus, it can be concluded that the values from this research indicated the beginning of spoilage.

Free Fatty Acid (FFA), a tertiary product of rancidity, increased during storage. The FFA is a measure of hydrolytic rancidity-the extent of lipid hydrolysis by lipase action. In most fish oils, rancidity is noticeable when the FFA (calculated as oleic acid) is in

between 0.5-1.5% [5]. It was noted that the PV and FFA results followed the same trend in this study. They were both produced as a result of fat oxidation (rancidity).

During storage, reduction in protein was corroborated with increase in TVN while low pH confirms increased putrefaction (spoilage). Reduction in fat content could have been due to oxidation and fat break down to other components [18]. This was confirmed by higher peroxide value (Fig. 3) and free fatty acid (Fig 4) of the stored products.

CONCLUSION

Although chemical quality indices of smoked-dried fish could predict their biological utilisation by consumers [24], physical assessment is the method that is easily available to an intending fish buyer. While physical or subjective methods of analysis may suffer from the limitation of being subject to bias by the assessor and hence may not be reproducible, the chemical methods are reliable measures of freshness or state of deterioration of product. This is so as the concentrations of chemicals are dependent on storage time and temperature. Therefore, based on both physical and chemical methods of assessment it was observed that the five smoked fish species underwent loss in quality. The optimum storage period of the smoked fish under natural condition at 10% moisture content is 4-5weeks. This shows that when stored at ambient temperatures, these smoked fish should be consumed within four weeks (One month). Under the ambient conditions, the cost of storage was low since there was neither special temperature regulator nor additive. Hence, in spite of being physically firm, caution should be exercised in consuming fish stored on open shelf for very long weeks. Such fish could contain microbial cells; re-heating may be necessary to destroy or inactivate such cells. A longer storage period could be attained under improved storage conditions, though more expensive.

RECOMMENDATIONS

Based on the results of this study, the following recommendations are made:
-   Intermittent sundrying or mild smoking can be carried out on smoked fish to extend its shelf life.
-   Preservatives such as pirimiphos - methyl (actelic) can be applied to preserve smoked fish.
-   The salt concentration could be raised to a level above 30% as this inhibits growth of insect larvae.
-   In areas where electricity supply is constant, smoked fish can be oven-dried, refrigerated or frozen, inhibiting some spoilage bacteria.
-   Moisture content less than 10% should be maintained in stored smoked fish to reduce the growth of bacteria and moulds.

Table 1: Physical (Organoleptic) Characteristics of the smoked fish species during storage.

Quality Parameters	Fish Species	Length of Storage (Weeks)									Means
		0	1	2	3	4	5	6	7	8	
Taste	*H.niloticus*	4.60±0.55	4.40±0.55	4.20±0.45	3.80±0.55	3.60±0.55	3.40±0.55	2.80±0.45	2.60±0.45	2.40±0.71	3.53±0.89[a]
	L. coubie	4.40±0.55	4.20±0.45	4.00±0.71	3.60±0.45	3.40±0.55	2.80±0.55	2.60±0.55	2.00±0.55	1.40±0.71	3.16±1.09[c]
	P.obscura	4.60±0.55	4.20±0.45	4.00±0.71	3.80±0.45	3.40±0.55	2.80±0.55	2.60±0.45	2.40±0.71	1.80±0.71	3.29±1.01[bc]
	O. niloticus	4.80±0.45	4.60±0.55	4.00±0.45	3.80±0.45	3.60±0.55	3.20±0.45	3.00±0.55	2.80±0.84	2.60±0.55	3.60±0.84[a]
	C.gariepinus	5.00±0.45	4.80±0.55	4.20±0.45	3.80±0.71	3.60±0.45	3.40±0.55	2.80±0.55	2.20±0.55	1.60±0.84	3.49±1.16[ab]
Odour	*H.niloticus*	4.60±0.55	4.40±0.55	4.40±0.55	4.00±0.55	3.60±0.71	3.60±0.55	3.00±0.55	2.60±0.55	2.40±0.55	3.62±0.94[a]
	L. coubie	4.80±0.45	4.40±0.55	4.00±0.71	3.60±0.55	3.40±0.55	3.20±0.45	2.80±0.55	2.40±0.55	1.80±0.84	3.38±1.05[a]
	P.obscura	5.00±0.45	4.40±0.55	4.00±0.71	3.80±0.71	3.60±0.45	3.40±0.55	2.80±0.45	2.60±0.55	2.00±0.71	3.51±1.01[a]
	O. niloticus	4.60±0.55	4.40±0.55	4.00±0.71	3.80±0.45	3.60±0.55	3.40±0.45	2.80±0.55	2.40±0.55	2.20±0.45	3.47±0.94[a]
	C.gariepinus	4.80±0.55	4.40±0.55	3.80±0.45	3.60±0.55	3.40±0.55	3.20±0.55	2.80±0.55	2.20±0.45	2.00±0.84	3.36±1.00[a]
Colour	*H.niloticus*	4.60±0.55	4.40±0.55	4.20±0.55	3.60±0.84	3.40±0.84	3.20±0.71	3.00±0.71	2.60±0.55	2.40±0.55	3.49±0.89[bc]
	L. coubie	4.40±0.55	4.00±0.89	3.80±0.55	3.40±0.71	3.20±0.55	3.00±0.71	2.80±0.84	2.00±1.30	1.80±0.84	3.13±1.01[d]
	P.obscura	4.20±0.84	4.00±0.55	3.80±0.55	3.60±0.55	3.40±0.71	3.00±0.71	2.80±0.84	2.60±0.71	2.20±1.14	3.29±0.89[cd]
	O. niloticus	4.60±0.55	4.40±0.55	4.00±0.55	4.00±0.71	3.80±0.55	3.60±0.71	3.40±0.84	3.00±0.84	3.00±0.55	3.76±0.74[a]
	C.gariepinus	4.80±0.45	4.60±0.55	4.40±0.55	3.80±0.55	3.60±0.55	3.40±0.55	3.00±0.84	2.80±0.71	2.00±0.84	3.60±1.01[ab]
Texture	*H.niloticus*	4.80±0.45	4.60±0.55	3.80±0.45	3.80±0.71	3.60±0.55	3.40±0.55	3.20±0.55	3.00±0.55	2.60±0.45	3.64±0.80[b]
	L. coubie	4.40±0.45	4.00±0.55	3.80±0.45	3.40±0.55	3.20±0.55	3.00±0.55	2.60±0.55	2.40±0.55	2.00±0.45	3.24±0.91[c]
	P.obscura	5.00±0.55	4.80±0.55	4.60±0.45	3.80±0.55	3.60±0.55	3.40±0.45	3.40±0.89	3.20±0.45	3.00±0.55	3.87±0.81[a]
	O. niloticus	4.80±0.45	4.60±0.55	3.80±0.55	3.60±0.45	3.40±0.55	3.20±0.55	3.00±0.55	3.00±0.55	2.80±0.45	3.58±0.78[b]
	C.gariepinus	4.60±0.45	4.40±0.45	4.20±0.45	4.00±0.55	3.60±0.55	3.20±0.55	2.80±0.89	2.60±0.55	2.20±0.45	3.51±0.92[b]
Fragment	*H.niloticus*	4.60±0.55	4.60±0.55	4.40±0.84	4.20±0.71	3.80±0.55	3.40±0.71	2.80±0.55	2.60±0.55	2.40±0.55	3.64±0.96[a]
	L. coubie	4.80±0.45	4.40±0.45	3.80±0.55	3.60±0.55	3.60±0.55	3.40±0.55	2.80±0.55	2.60±0.71	2.40±0.55	3.49±0.89[ab]
	P.obscura	4.60±0.55	4.40±0.55	4.20±0.55	3.60±0.55	3.40±0.55	3.20±0.55	3.00±0.45	2.60±0.84	2.20±0.84	3.47±0.92[ab]
	O. niloticus	4.80±0.45	4.60±0.55	4.40±0.55	3.60±0.55	3.40±0.55	3.00±0.45	2.80±0.45	2.60±0.55	1.60±0.84	3.40±1.10[b]
	C.gariepinus	4.60±0.55	4.60±0.55	4.00±0.71	3.80±0.84	3.40±0.55	3.20±0.45	3.00±0.55	2.80±0.55	2.60±0.45	3.56±0.87[ab]

- **Means of replicate values with standard deviation**

- **Means with different superscripts differs significantly (P< 0.05).**

Table 2 : Mean Proximate composition (Initial and Final %) of stored smoked fish species

Fish Species	Moisture Content			Crude Protein			Fat Content			Ash Content		
	Initial (Wk 0)	Final (Wk 8)	Percentage Difference	Initial (Wk 0)	Final (Wk 8)	Percentage Difference	Initial (Wk 0)	Final (Wk 8)	Percentage Difference	Initial (Wk 0)	Final (Wk 8)	Percentage Difference
Heterotis niloticus	10.40	10.59	1.83%	72.78	69.01	5.18%	11.32	10.94	3.37%	8.50	8.72	2.59%
Labeo coubie	10.42	10.58	1.54%	58.95	56.87	3.53%	16.12	15.31	5.03%	9.53	9.74	2.20%
Parachanna Obscura	10.41	10.58	1.63%	54.71	53.02	3.09%	16.58	15.68	5.43%	8.31	8.49	2.17%
Oreochromis niloticus	10.41	10.62	2.02%	69.24	66.35	4.17%	14.22	13.60	4.36%	9.14	9.36	2.41%
Clarias gariepinus	10.39	10.56	1.64%	56.82	54.74	3.66%	20.24	18.96	6.32%	8.54	8.75	2.46%

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