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EFFECTS OF DIFFERENT PACKAGING MATERIALS ON THE NUTRITIONAL QUALITY OF OVEN DRIED KILISHI.

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Abstract

Meat is a good source of quality protein but undergoes rapid deterioration due to physical, chemical and microbial influence. Hence, the need to develop a nutrient-dense product like kilishi with significant storability and promote food security. Kilishi was produced from beef meat and was packaged using three packaging material which include High density polyethelene (HDPE), low density polyethelene (HDPE) and carton. The kilishi samples were analyzed for proximate composition and, physicochemical properties were carried out on the samples with standard methods over a period of 3 months. Moisture, ash, fat, protein, and carbohydrate contents of the kilishi samples ranged from 3.16-8.43 %, 6.22-8.82 %, 6.15 -

Introduction

Meat and meat products make valuable contributions to diets of developing countries due to its excellent source of high quality protein, large amounts of minerals, essential vitamins, fats and carbohydrates (Mgbemere *et al.*, 2011). However, in spite of its constant demand, meat is a highly perishable food item due to its abundant nutrients that favour the establishment, growth and proliferations of microorganisms (Wu *et al.*, 2017). Fonkem *et al.* (2010)

11.33 %, 46.49-55.24 % and 20.79-32.72 % respectively. pH, protein solubility, peroxide value, thioberbituric acid of the kilishi samples ranged from 5.55-6.55, 17.50-29.69 (mg/ml), 5.60-12.56 (mEq/kg) and 0.28-0.91 (mg/kg) respectively. The results showed that the packaging materials significantly affected the proximate, but the physicochemical properties were not significantly affected by the packaging materials during storage.

Keywords: kilishi, meat, carton, high density polyethylene, low density polyethylene.

noted that, the presence of some of these microorganisms may render it poisonous and unfit for human consumption. This may be the reason why man, has over the decades developed a number of meat preservation techniques that can maintain its stability and thus, shelf-life while at the same time protecting adequate nutritive value and desirable flavour. According to Apata *et al*. (2013), meat post-mortem processing in which properties of fresh meat are modified by the use of one or more seasoning, heat treatment or drying, is one of the ways of preserving it against microorganisms. Meat is an edible part of animal that comprised principally of fat, muscle, connecting tissues and used as food (Iheagwara and Okonkwo, 2016). Meat is rich in quality protein (Shamsudden, 2009), significant amount of minerals, appreciable essential vitamins as well as enough carbohydrate and fat for energy production (boost nutritive value on humans) (Ahmad *et al*., 2018). It contains a myriad of valuable nutrients which supports the proliferation of spoilage organisms when handled inappropriately, making meat very perishable, therefore preservation is required to ensure that the keeping quality is extended (Shamsudden, 2009).

Drying is one of the most antique processes to preserve foods and it consist of removal of water from the food product up to a level at which microbial spoilage and deterioration reactions are highly minimized (Krokida *et al*., 2013). This is one of the storage processes which have the ability to extend the shelf life of foods, still trying to maintain part of their nutritional properties. (Guiné and Barroca, 2012) and during this process foods may lose some nutrients, depending on drying conditions such as temperature or drying time.

Kilishi is a Hausa word, *Kussel* Fulani, which refers to beef, sheep or goat meat that is processed by slicing, dressing, sun-drying application of spiced slurry and roasting in a glowing fire. It contains varying constituents of protein, moisture, lipid, fibre and ash respectively, depending on the quality of meat used and amount of heat / drying. It is a rich snack (meat crackers) that has a nourishing and satisfying sensation. In areas where preservative and storage facilities are absent or limited such as in Northern Nigeria occupied by Fulani and Hausa herders, production of *kilishi* is a way of developing stable meat products with significant storage stability (Isah and Okunbanjo, 2012), however, the storage stability of *kilishi* is season, production and location dependent (Fonkem *et al*., 2010). Also, it has been shown that the quality of *Kilishi* produced by the traditional processors varies from one producer to the other and from one batch to another by the same producer (Olusola, 2006). According to Jones *et al*., (2001), some of the spices used in *Kilishi* processing such as onion, ginger and garlic have medicinal properties as well as being nutritious.

Packaging surrounds and protects food after manufacturing, defend its integrity through transportation, handling, storage and retailing, and ensure its wholesomeness during consumption. Packaging maintains the benefits of food processing after the process is complete, enabling foods to travel safely from point of origin to the point of consumption (Prasad and Kochhar, 2014). Food packaging has evolved from simply a container to hold food to something today that can play an active role in food quality. Many packages are still simply containers, but they have properties that have been developed to protect the food (Risch, 2009). Packaging today plays an important role in the quality of food products by providing protection from environmental, chemical, and physical challenges. This protection can be as simple as

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preventing breakage of the product to providing barriers to moisture, oxygen, carbon dioxide, and other gases as well as flavors and aromas. Packaging can block light to protect nutrients and colors in a product from deteriorating. The aim of this research is to evaluate the effect of packaging materials on the nutritional quality of oven dried kilishi.

MATERIALS AND METHODS

Sources of Materials

Raw meat (beef) samples was bought from Malumfashi abattoir in Katsina state. The meat was purchased early in the morning to ensure that the meat is fresh and relatively hygienic for processing of *kilishi*. The ingredients for the slurry (groundnut cake, red pepper, black pepper, onion, ginger, alligator pepper big pepper, thyme, salt, sugar, cloves, curry, garlic, nutmeg and maggi) and the packaging materials (Carton, HDPE, and LDPE) were purchased from Malumfashi central market, Katsina State.

Samples preparations

Meat preparation

The meat obtained was thoroughly washed with water in order to remove dirt and blood stains, Fat and connecting tissues which are not required for the production of *kilishi* were also removed. After cleaning and trimming of the unwanted materials, the meat was cut into thin slices measuring 1-2 cm thick and 60-80 cm long as described by Iheagwara and Okonkwo (2016).

Preparation of the ingredients

Infusion slurry was prepared following the procedures of Muhammad and Muhammad, (2010) with slight modification. The fresh groundnut paste was prepared from grains of dry uncooked groundnut after extraction of oil by pressing. The various ingredients (groundnut cake, red pepper, black pepper, onion, ginger, alligator pepper big pepper, thyme, salt, sugar, water, cloves, curry, garlic, nutmeg and maggi) in propotions as presented in Table 1 were ground and mixed thoroughly with water to form slurry.

Table 1 ingredient formulation for *kilishi*

Name of ingredients		% Composition
Groundnut cake (Tunkusa)		38.0
Red pepper		1.10
Black pepper		3.40
Onion		5.70
Ginger		2.50
Alligator pepper		1.80
Big pepper		1.10
Thyme seasoning		1.00
Salt		0.10
Sugar		2.50
Water (ml)		36.0
Cloves		2.50
Curry		0.70
Garlic		0.50
African nutmeg		1.00
Maggi (Star)		2.10
Total		**100.0**

Source: Muhammad and Muhammad (2010)

Production of *Kilishi*

The method described by Ogunsola and Omojola (2008) was adopted with slight modifications for *kilishi* production. First the slice meat were spread on the tray in oven at 71°C for 2-4 hours. The dried thin sheets of meat were soaked in the infusion slurry for about 30 minutes, after which they were taken out and were spread out on flat steel trays in oven at 50 °C for 30 minutes and dried to generate the *kilishi.* After final drying process, the infused meat was transferred to where it was gently roasted with a glowing fire.

The height at which the meat was placed on the wire mesh allowed the heat of contact on the meat to be around 100-120 °C for 10-15minutes. This enormously reduced the moisture content of the product and the *kilishi* acquires its special flavour from the smoke that was produced from the fire wood during this period. The hot, sizzling, brown colored *kilishi* samples were then removed, cooled to room temperature and packaged in high density polyethylene (HDPE), low density polyethylene (LDPE) bags and Carton (CTN) and stored at ambient temperature until it is required for analyses (Ogunsola and Omojola, 2008). The proximate and physicochemical, were determined after 30 days for three months.

Proximate analysis of *kilishi*

The *kilishi* samples was analyzed for proximate composition (moisture, fat, crude protein, ash and nitrogen free extract) using the methods described by AOAC (2018).

Determination of moisture content

The moisture content of the samples was determined according to the method of AOAC (2018). Two grammes (2 g) of the sample was weighed into a dry crucible of known weight and placed in an oven set to 105 °C for three hours. The sample was weighed using an electronic analytical balance after cooling in a desiccator. They were placed back into the oven to continue drying. It was removed, allowed to cool and weighed until the sample reached a constant weight. The change in weight was determined by weighing the amount of moisture that is lost as a percentage calculated as follows: using equation (i)

$$Moisture\ content(\%) = \frac{Weight\ loss\ (W_2 - W_3)}{Weight\ of\ sample\ (W_1)}\ x\ 100 \ldots\ldots\ldots\ldots\ldots\ldots \text{(i)}$$

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Where

W_3 = weight of crucible + weight of sample after reaching constant weight, W_2 = weight of crucible + original weight of sample and W_1 = weight of empty crucible

Determination of ash content

The ash content of the samples was determined according to the method of AOAC (2018).Two grammes (2 g) of each sample was weighed and transferred into a porcelain crucible of known weight and it was heated in a Muffle furnace at 550 °C for three hours until it completely turned to white ash free of carbon residue. After taking out of the furnace, the sample was cooled to room temperature in a desiccator and then promptly reweighed. Thereafter, the residual percentage ash content was determined as shown in equation (ii)

$$Ash\ content\ (\%) = \frac{Weight\ loss\ (W_2 - W_3)}{Weight\ of\ sample\ (W_1)}\ x\ 100 \ldots\ldots\ldots\ldots \text{(ii)}$$

Where

W_3 = crucible (at constant weight after drying) + weight of sample, W_2 = crucible + sample weight and W_1 = sample weight

Determination of crude protein content

The Kjeldahl technique was used to determine the crude protein concentration. The sample (1 g) was weighed into a Kjeldahl flask for digestion. The samples were then mixed with 3.0 g of hydrated cupric sulphate, 20 ml of sodium sulphate solution (Na_2SO_4) and 1.0 mL of concentrated sulfuric acid (H_2SO_4). The solution was heated in the flask while it was clamped inside of a fume cupboard.

Distillation stage: After cooling the clear solution, 100 ml of distilled water was added to dilute it. In order to produce ammonia, ten (10) ml of the

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resultant solution was combined with five (5) mL of a 40 % sodium hydroxide solution in a distillation flask.

Titration: Hydrochloric acid (0.1 ml) was added to the resultant solution above during this stage (Hcl).The crude protein was computed using the expression utilizing the titre value, or the point at which the color shifted from green to pink. It is calculated as shown in equation (iii)

$$\text{Crude Protein } (\%) = \frac{14.01 \text{ x } 6.25 \text{ x } 25 \text{ x T}}{\text{W x } 10} \text{ x } 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(iii)}$$

Where

VF = total volume of the digest = 100 ml, W = weight of the sample digested,T = titre Value, 10 = aliquot volume distilled, 6.25 = conversion factor and N = Normality of Hcl in moles per 100 ml (0.1N)

Determination of crude fat content

Solvent extraction method was used. The setup included a reflux condenser and a soxhlet extraction machine. A round-bottomed flask was weighed after washing, drying, then filled to half with petroleum ether (boiling point 40-60 ^{o}C) and put into the apparatus. The round bottom flask with 120 ml of petroleum ether was cleaned, dried and weighed. Two grammes of each sample was wrapped in Whatmann filter paper and slowly lowered into the thimble. Petroleum ether was used to extract fat for 6 hours and then distillation was used to recover it. To calculate the amount of lipid extracted, the flask was weighed after the solvent evaporated from it and dried in an oven at 105 °C. The weight of oil extracted was determined by difference and thus expressed as shown in equation (iv)

$$Crude\ fat\ content\ (\%) = \frac{Weight\ loss\ (W_2 - W_3)}{Weight\ of\ sample\ (W_1)}\ x\ 100 \ldots\ldots\ldots\ldots\ldots\ldots \text{(iv)}$$

Where

W_3 = weight of sample + filter paper (constant weight after drying), W_2 = weight of sample + filter paper and W_1 = weight of sample

Determination of crude fibre content

The crude fibre content of the samples was determined according to the method of AOAC (2018). Defatted sample (2 g) was heated for 30 minutes at reflux in a solution containing 200 ml of 1.25 % H_2SO_4. The sample was first boiled for 30 minutes under the same conditions, and then washed several times with hot water using a two-fold muslin cloth to trap the particles. The sample was returned to the flask and boiled for another 30 minutes in 200 ml of 1.25 % NaOH solution. This time, it was allowed to drain dry before being transferred to a weighed crucible where it was dried at 105 °C to a constant weight. Following that, it spent 4 hours at 550 °C in a muffle furnace before being cooled in a desiccator and reweighed. By difference in weight, the weight of the fibre was calculated by the expression as shown in equation (v)

$$Crude\ fibre\ content\ (\%) = \frac{Weight\ loss\ (W_2 - W_3)}{Weight\ of\ sample\ (W_1)}\ x\ 100 \ldots\ldots\ldots\ldots...(v)$$

Where

W_3 = weight of sample + crucible after ashing (constant weight after drying), W_2 = weight of sample + crucible before ashing and W_1 = initial weight of sample

Determination of carbohydrate content

The nitrogen free technique was employed. The carbohydrate was estimated as weight by the difference between 100 and the addition of the percentages of other factors as Nitrogen Free Extract percentage carbohydrate.

$$\text{Carbohydrate content } (\%) = 100 - (\text{M} + \text{P} + \text{F} + \text{A} + \text{F2}) \ldots\ldots\ldots\ldots(vi)$$

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Where

M = moisture, P = protein, F = fat, A = ash and F2 = crude fibre

Physicochemical analysis of *kilishi*

Physicochemical analysis was determined by AOAC (2018)

Determination of pH

The pH was determined by AOAC (2018). The *kilishi* was ground into a fine powder using mortar and pestle. A 10g portion of the powdered *kilishi* was mixed with 100mL of distilled water in a beaker. The mixture was stirred for 5 minutes to ensure complete dissolution. The pH electrode was calibrated using pH 4, 7 and 10 buffer solutions. The calibrated pH electrode was inserted into the *kilishi* mixture and the pH reading was taken after stabilization. The pH reading was recorded and repeated three times to ensure accuracy. The average values were recorded.

Determination of protein solubility

The protein solubility was determined by AOAC (2018). The *kilishi* sample was ground into a fine powder using mortar and pestle. A 5 g portion of the powdered *kilishi* was mixed with 50 mL of distilled water in a centrifuge tube. The mixture was vortexed for 1 minutes to ensure complete dispersion. The mixture was then centrifuged at 3000 rpm for 10 minutes. The supernatant was collected and mixed with 10 mL of buffer solution (pH 7.0). The mixture was vortexed again for 1 minute and then centrifuged at 3000 rpm for 5 minutes. The protein content of the supernant was determined using a spectrophotometer at 280 nm. The protein solubilty was calculated as the percentage of soluble protein in the total protein content using using equation (xii)

$$\text{Protein solubility (mg/ml)} = \frac{\text{Soluble Protein } X \text{ } 100}{\text{Total Protein}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots..(xii)$$

Where: mg =miilgram mL=mililiter

Determination of peroxide value

The peroxide value was determined by AOAC (2018). The *kilishi* sample was ground into a fine powder using mortar and pestle. A 5 g portion of the powdered *kilishi* was mixed with 30 mL of chloroform in a glass tube. The mixture was shaken for 1 minute and then centrifuged at 3000 rpm for 5 minutes. The supernatant was collected and mixed with 0.5 mL of sodium thiosulfate solution (0.1 N) in a glass tube. The mixtures was then placed in a water bath at 60 ^{o}C for 10 minutes. After cooling, 1mL of starch indicator solution was added to the mixture. The mixture was titrated with sodium thiosulphate solution (0.1 N) until the colour changed from blue to colourless. The peroxide value was calculated as shown in equation (xiii)

$$\text{Peroxide Value (meq/kg)} = \frac{\text{Volume of sodium thiosulfate x Normality } X \text{ 1000}}{\text{Sample weight}} \ldots\ldots\text{(xiii)}$$

Where: meq =milliequivalent kg=kilogram

Determination of thiobarbituric acid

The thioberbioturic acid was determined by AOAC (2018). The *kilishi* sample was ground into a fine powder using a mortar and pestle. A 10 g portion of the powdered *kilishi* was mixed with 50 mL of distilled water in a test tube. The mixture was heated in a water bath at 90 ^{o}C for 30 minutes. After cooling, 5 mL of TCA solution (20%) was added to the mixture. The mixture was then centrifuged at 3000 rpm for 10 minutes. The supernatant was collected and mixed with 5 mL of TBA solution (0.6%). The mixture was then heated in a water bath at 90 ^{o}C for 30 minutes. After cooling, the absorbance was measured at 532nm using a spectrophotometer. The TBA value was calculated using equation (xiv)

$$\text{TBA Value (mg/kg)} = \frac{\text{(Absorbance x 100)}}{\text{Sample weight}} \ldots\ldots\ldots\ldots\ldots\ldots \text{(xiv)}$$

Where: mg =miilgram kg =kilogram

Statistical analysis

Data obtained was subjected to descriptive statistics and means subjected to one-way analysis of variance (ANOVA). Means were separated using Duncan's Multiple Range Test (DMRT) at $p<0.05$ level of significance using Statistical Package for the Social Science Version 15.0.

RESULTS AND DISCUSSION.

Table 2: Proximate composition of fresh kilishi and *kilishi* stored for 5 months

Sample	Moisture (%)	Ash (%)	Fat (%)	Protein (%)	Nitrogen Free Extract (%)
AFTER 1 MONTH OF STORAGE					
A	8.43 ± 0.04^{a}	6.22 ± 0.02^{c}	6.15 ± 0.05^{d}	46.49 ± 0.67^{c}	32.72 ± 0.76^{a}
B	5.65 ± 0.01^{c}	7.66 ± 0.05^{a}	9.13 ± 0.07^{a}	50.84 ± 0.31^{a}	25.21 ± 0.38^{c}
C	6.46 ± 0.05^{b}	6.78 ± 0.06^{b}	7.71 ± 0.04^{c}	47.17 ± 0.42^{b}	31.76 ± 0.64^{a}
D	4.36 ± 0.03^{d}	7.12 ± 0.03^{b}	8.59 ± 0.03^{b}	48.58 ± 0.01^{b}	28.12 ± 0.02^{b}
AFTER 2 MONTHS OF STORAGE					
A	8.43 ± 0.04^{a}	6.22 ± 0.02^{c}	6.15 ± 0.05^{d}	46.49 ± 0.67^{d}	32.72 ± 0.76^{a}
B	4.18 ± 0.02^{c}	8.66 ± 0.05^{a}	10.63 ± 0.03^{a}	53.34 ± 0.17^{a}	23.19 ± 0.46^{c}
C	5.06 ± 0.05^{b}	7.07 ± 0.05^{b}	7.95 ± 0.04^{c}	48.78 ± 0.65^{c}	31.02 ± 0.34^{a}
D	4.01 ± 0.03^{c}	7.42 ± 0.05^{b}	9.02 ± 0.01^{b}	51.40 ± 0.02^{b}	27.97 ± 0.03^{b}
AFTER 3 MONTHS OF STORAGE					
A	8.43 ± 0.04^{a}	6.22 ± 0.02^{c}	6.15 ± 0.05^{d}	46.49 ± 0.67^{d}	32.72 ± 0.76^{a}
B	3.58 ± 0.01^{b}	9.06 ± 0.07^{a}	11.33 ± 0.04^{a}	55.24 ± 0.57^{a}	20.79 ± 0.78^{c}
C	4.06 ± 0.07^{b}	7.38 ± 0.07^{b}	8.01 ± 0.01^{c}	49.77 ± 0.75^{c}	30.79 ± 0.64^{ab}
D	3.16 ± 0.06^{b}	8.82 ± 0.07^{ab}	9.50 ± 0.01^{b}	52.50 ± 0.01^{b}	26.02 ± 0.01^{b}

Means within same column with different superscripts differed significantly ($p\leq0.05$)

Keys: (A = Fresh *kilishi*, B = Carton Storage, C = High density polyethelene Storage, D = Low density polyethelene Storage

The proximate composition of the *kilishi* is presented in Table 2. The result showed a promising importance of all the packaging material within the length of the storage period. The moisture content of the fresh *kilishi* was 8.43

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% which is less than 10.00% reported by Olusola *et al*., (2012) but it's in agreement with 8.97 % reported by Ndife *et al*., (2022) for quality of *kilishi* similar results were reported by (Falowo, 2023; Inusa and Said 2017) with moisture content of 7-12 % and 8.70- 9.87%. For a good *kilishi ,* according to Thomas, (2018) and Gao *et al*., (2003), *kilishi* with moisture content of 20 and 15 % has the ability of inhibiting some bacteria and fungi species respectively hence good shelf stability is not guaranteed. This variation in moisture content with previous research could be attributed to the initial meat type, age of the meat, the length of the drying process and the overall processing technique (Mbofung, 1993). It could also be as a result of the environmental conditions and the infusion ingredients paste used during its production, and or other nutrients in meat products (Iheagwara and Okonkwo, 2016). After 3 months storage, the moisture content of the *kilishi* sample reduce to 3.58, 4.06 and 3.16 % in carton, high density polyethylene and low density polyethylene packaging indicating loss of moisture which is not desirable for a good packaging material, though the *kilishi* samples after 3 months did not differ significantly ($p>0.05$). Packaging material are expected to provide a total barrier to external environment, protect the food and reduce sorption or adsorption (Garba, 2023). Even though the overall moisture content of the sample was below the acceptable limits for *kilishi* (15%) according to the Nigerian Industrial Standard (NIS, 2008), recommend a moisture content of less than 18% for dried meat product like *kilishi* to ensure food safety and quality (Ajibola *et al*., 2016). Akpinar *et al*., (2006) suggests that a moisture content of 12-14% is optimal for *kilishi* to maintain its texture and shelf life. This effect of packaging is not desirable as it might tend to affect the other organoleptic or physicochemical properties of the meat.

The ash content depicts the concentration of elemental properties of a food material. Ash content is an indication of food quality such that, the higher the mineral composition the higher the propensity of the food material to supply micronutrients. The ash content of the control sample was significantly ($p\leq0.05$) lower (6.22 %) than 9.06 % in carton packaging, 7.32 % in high density polyethylene packaging and 8.82% in low density polyethylene packaging but in agreement with 1.2 – 24.5% reported by (Falawo, 2023) for a quality *Kilishi,* and higher than 3.99-6.31 % reported by Ndife *et al*., (2022). The high ash content of the *kilishi* is important and the variation in this results could be due meat type, specie, age, sex and contribution of infused ingredients, which is in agreement to the observation reported by Khalid *et al*., (2012)*.* The processing of beef into *kilishi* alters the ash content of the meat as opined by Thomas, (2018). Each spice used contains an individual mineral content which when aggregated, excluding the losses accounted for during processing, results to increased mineral level which is represented by high ash content. Previous research showed that, the processing steps in *Kilishi* production affect its ash content (Olagunju and Taiwo, 2020). Jones *et al*., (2001) reported ash content of 6.72% while Khalid *et al*., (2010) reported 9.6% for finished *kilishi* product and 7.83% for dried infused product before roasting. The values obtained in this study showed that the *kilishi* had the higher ash content which may signifies the presence of a higher amount of mineral content compared to other reported results. Probably due to the effect of the packaging that allow moisture loss, the ash content of the *kilishi* increases to compensate the amount of water/moisture loss, the ability of carton packaging to cause more increase in ash might also be explained of the characteristics porosity of the material if compared with high density polyethylene packs, this affect the rate of moisture loss and the interaction of the packaging with external environmental factors.

The fat contents of the *kilishi* were generally affected by the packaging material and duration of storage. The initial fat content of 6.15 % in fresh sample increased significantly ($p<0.05$) to 11.33% in carton packaging, 8.01% in high density polyethylene packaging and 9.50 % in low density polyethylene packaging. This trend can be explained by the characteristic porosity of the packaging material. Carton packs are generally absorptive as such they can allow passage of fat through permeation (Garba, 2023) which is not desirable as fat are well known to be a dissolving medium for many important vitamins in meat. The fat content of the sample (6.15-11.33 %) was generally within the moderate fat content of *kilishi* which lies between 6.40 % and 13.33 % reported by Falowo, (2023) and were within the range reported by Mgbemere *et al*., (2011) of 13.4%., but fall below 16.21 to 27.31% reported by Ndife *et al*., (2022) and 17.07 to 22.42% by Jones *et al*., (2001). This high-fat content of the *kilishi* samples could be principally contributed by the groundnut cake powder which probably contains residual fat and represents a considerable amount of the product. Jones *et al*., (2001) and Seydou *et al*. (2019) noted that *kilishi* is very high in lipid content on a dry matter basis (about 25.30%), consisting mostly of triglycerides while the level of fat in fresh meat was less than 10.0%. position of the animal meat.

Protein is a major indicator of a good quality meat and meat product. Falowo, (2023) reported that, in comparison to other meat products, *kilishi* meat contained higher protein content with its protein content ranging from 16-23%. This increase in protein is because of the reduce moisture content of *kilishi,* meat which was significantly dehydrated by drying thereby improving and concentrating the percentage protein content and other nutrients in the meat. Similar reports have shown that cooking or heat processing usually decreases moisture content and concentrates other nutrients in meat products (Iheagwara and Okonkwo, 2016). In addition, the type and amount of slurry ingredients used may also influence the protein content of *kilishi* meat (Idowu *et al*., 2010). Idowu *et al*., (2010) has reported that the addition of groundnut cake paste tend to increase or contribute to higher protein content of *kilishi* meat when compared to other meat products. The protein content of *kilishi* as affected by storage period and packaging material in this research has showed a variability based on the type of packaging film used. The protein content of the oven dried *Kilishi* in a fresh form was 46.49 % which falls below the reported 49-71 % (Mgbemere *et al*., 2011; Olusola *et al*., 2012). This value increased significantly ($p<0.05$) to 55.24 % in carton film, 49.77% in high density polyethylene film and 52.50 % in low density polyethylene packaging after 3 month of storage. The ability of the *kilishi*

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sample to gain protein upon storage can only be justify by the inability of the packaging films to provide a total barrier to external environment which is not desirable in all meat products (Garba, 2023). A source of protein is an essential element of a healthy diet, allowing both growth and maintenance. There has been debates and published works on protein intake and health. Issues that arise from the potential for protein intakes are predicated on protein intakes to be in excess of the recommended intake (Emmanuel *et al*., 2020). These potential deleterious issues were based mostly on renal function, bone health, kidney stones, cardiovascular disease and cancer. For bone health, protein as part of a well-balanced diet may be beneficial for bone, possibly at dietary levels in excess of the recommended intake; in order to minimize the risk of kidney stones in patients who are at risk, the protein safe level of 0.83 g/kg per day is recommended but not in excessive amounts (i.e. less than 1.4 g/kg per day, preferably from vegetable sources; inverse relationships between protein intake and blood pressure have been reported by Emmanuel *et al*., (2020); for cancer, it has been reported that high dietary protein results in better survival in women with breast cancer (Emmanuel *et al*., 2020). From our results, a *kilishi* consumption of 64.4 g/100g per day would yield greater than 58.1 kg per day for a 70kg adult by an excess of 6.3 g/100g or 9.78%.

The Nitrogen value for *kilishi* samples stored for 3 months is presented in Table 2. Total volatile nitrogen contains total amount of volatile nitrogen bases together with nitrogen which is synthesized by reaction from protein. The result ranged from 32.72 in fresh sample, 20.79 in carton, 30.79 in high density polyethylene, and 26.02 in low density polyethylene pack after storage indicating a significant ($p \leq 0.05$) reduction in nitrogen value hence low protein degradation, therefore active packaging in all the selected packaging materials. The nitrogen value was also below the legal limit of 50 mgN/100g muscle and were still under the standard limit (<35 mgN/100g) as reported by Siripongvutikorn *et al*. (2009) for spiced salmon flesh waste. The nitrogen for the fresh and stored *kilishi* in this study are higher than 13.73

%, 13.16 – 18.90%, 13.80 - 16.46 %, 18.9% obtained in *Tunkusa Kilishi* and 14.8 % in groundnut flour *kilishi* reported by (Olusola *et al*., 2012 and Mgbemere *et al*., 2011) respectively.and lower than 18.21-22.15 %, 20.35-24.19 % reported by (Ajibola *et al*., 2016; Akpinar *et al*., 2006) and within the range of 25.20-31.40 % and 20.90-29.10 % reported by (Dairo *et al*., 2018; and Adesogun *et al*., 2020). However, the values of Total Volatile Basic Nitrogen (TVBN) obtained in this study were low for carton and low density polyethylene *kilishi* samples and this suggests that the level of protein decomposition or breakdown in *kilishi* samples were low.

TABLE 3 Physicochemical properties of fresh and stored *kilishi*

Samples	pH	Protein Solubility (mg/ml)	Peroxide Value (mEq/kg)	Thioberbituric Acid (mg/kg)
AFTER 1 MONTH OF STORAGE				
A	6.55±0.02^{a}	19.69±0.05^{c}	8.00±0.02^{b}	0.40±0.01^{a}
B	6.20±0.00^{b}	27.81±0.06^{a}	9.87±0.03^{a}	0.42±0.02^{a}
C	6.55±0.02^{a}	24.06±0.02^{b}	5.60±0.02^{d}	0.30±0.01^{b}
D	6.40±0.01^{a}	25.94±0.03^{b}	7.18±0.03^{c}	0.28±0.01^{b}
AFTER 2 MONTHS OF STORAGE				
A	6.55±0.02^{a}	19.69±0.05^{c}	8.00±0.02^{b}	0.40±0.01^{a}
B	5.95±0.00^{c}	29.69±0.05^{a}	10.96±0.01^{a}	0.59±0.01^{a}
C	6.25±0.02^{b}	25.63±0.04^{b}	7.71±0.01^{b}	0.31±0.00^{b}
D	6.10±0.01^{b}	27.81±0.02^{b}	8.10±0.01^{b}	0.36±0.00^{b}
AFTER 3 MONTHS OF STORAGE				
A	6.55±0.02^{a}	19.69±0.05^{c}	8.00±0.02^{c}	0.40±0.01^{b}
B	5.90±0.00^{c}	25.00±0.02^{a}	11.09±0.01^{a}	0.66±0.02^{a}
C	6.20±0.01^{b}	22.19±0.01^{b}	8.46±0.01^{c}	0.48±0.00^{b}
D	6.00±0.01^{b}	23.75±0.01^{b}	9.47±0.01^{b}	0.54±0.01^{b}

Means within same column with different superscripts differed significantly ($p \leq 0.05$)

Keys: (A = Fresh *kilishi*, B = Carton Storage, C = High density polyethelene Storage, D = Low density polyethelene Storage

Physicochemical analysis of *kilishi*

The physicochemical characteristics of the produced *kilishi* is presented in Table 3. The physicochemical characteristics explain the factors that can affect the degree of spoilage and influenced the concentration and availability of nutrients and microbial load of the product being processed. (Adeyeye *et al*., 2020), they also explain the adversity of the production process including heat application and other biochemical reaction during storage. Although the pH value was generally high (5.2-6.55) but is still not high enough to cause much changes in the product. The pH value is within the maximum limit of 6.0 suggested by (Ramatou *et al*., 2019) which indicated that the *kilishi* may have been produced from stock that was not well nourished nor well-rested. All packaging materials used did not also cause substantial change in pH level, with cardboard packaging offer best protection and maintain the pH to a close to fresh sample pH. Lower pH is not desired in meat product due to enzymatic and water activity. The trend seems to contrast the pattern recorded for the organic acids (lactic and acetic acids), suggesting an inverse correlation between concentration of organic acids and pH values of the *Tsire* samples. In a research investigation on the use of biological agents in the extension of fresh been in Nigeria, (Olaoye and Onilude, 2010) attributed the reduction in pH in the beef *Tsire* during storage to possible production of organic acids. However, in the present study, there was no significant ($p>0.05$) difference in the pH of the *kilishi* (a like meat product with *Tsire)* samples stored in different packaging materials, probably because no starter culture was inoculated in any of the samples to stimulate production of organic acids that may bring about reduction in pH.

The protein solubility of *kilishi* indicates its ease for softening when in contact with saliva in the mouth. The higher the solubility, the faster the rate of softening (Rahman *et al*., 2005). The solubility of meat is directly related to its tenderness, juiciness and palatability. It influences the wetness and release of fluid from the meat and sustained the juiciness that develops during chewing. The solubility value of the fresh was lower than all the sample within the duration of storage. There was no significant ($p>0.05$) difference from the second to the fifth month of storage in all the packaging material. Within the length of the storage, the sample did not suffer much change in solubility as such the packaging can be said to have preserved the characteristics juiciness of the *kilishi*. Although the range is lower than 61.25 to 69.40 mg/ml reported by Ndife *et al*., (2022) and Omojola *et al*., (2017) with protein solubility of 50-70%. The Nigerian Industrial standard (NIS, 2008) for *kilishi* recommends a minimum protein solubity of 40% but it is still classified as good meat even after storage.

The peroxide value determine the degree of rancidity, it is also related to the innateness and protective quality of the packaging to prevent moisture and light passage. When light pass into a fatty food it causes photolytic rancidity and when moisture pass in the presence of oxygen it leads to oxidative rancidity which affect the organoleptic quality of the product. It is therefore required that packaging material should provide a total barrier to moisture and oxygen. The higher the peroxide value, the lower the quality of the food sample. Peroxides are the main primary oxidation products. High amounts of peroxides value leads to low oxidative stability (Marquez *et al*., 2009). The results of the first and third month of storage did not differ ($p>0.05$) significantly as well as the second, fourth and the fifth months of storage among all the packaging materials. Generally, the peroxide value is higher in carton packaging and increase with duration of storage in all other packaging, this indicate continuous spoilage which is not desired. The Nigerian Industrial Standand (NIS, 2008) recommend maximum peroxide value of 10 meq/kg while International Organization for Standardization (ISO, 2018) recommend maximum peroxide value of 12 meq/kg for *kilishi,* this indicate that the *kilishi* is healthy and fit for consumption.

Thiobarbituric acid (TBA) is a chemical reagent used to measure lipid oxidation and rancidity in foods, particularly in meat and meat product like *kilishi*. The result obtained for thiobarbituric acid (TBA) content are higher than 0.06-0.41 reported by Ndife *et al*., (2022) but fall within the acceptable limit of (1-2 mg MDA/kg lipid) TBA value for *kilishi,* which can be attributed to the processing method for *kilishi* particularly the removal of visible fat, since fat is a promoter of oxidation. More so, the result also indicates the antioxidant properties and potential of spices against lipid oxidation (Keefe and Wang, 2006). Thiobarbituric acid, (TBA) is the most widely used method for the measurement of secondary oxidation products. Olaoye, (2015) noted that TBA could be associated with rancidity development in food products through lipid oxidation, and increase in its value during storage may cause off flavour in meat products. The secondary stage of oxidation occurs when the hydroperoxides decompose to form carbonyls and other compounds, particularly aldehydes which gives the food product a rancid smell (Ogbonnaya and Imodiboh, 2009). Though there was sight change in thioberbituric acid among the packaging material within the length of the storage period, but these changes did not cause a serious increase/decrease that exceed minimum or maximum limit as such good packaging property, The lower value of TBA recorded in the samples stored in high density polyethylene pack and low density polyethylene film may be advantageous, as this may help prevent or reduce lipid oxidation in the meat product during storage, thereby reducing chances of spoilage. There was significant ($p \leq 0.05$) difference among the samples for TBA values from the first month to the last month of storage. TBA values higher than 0.5 (mg malonaldehyde/kg) have been noted to trigger spoilage in meat during storage as reported (Olusegun *et al*., 2018) where he noted high TBA in different *tsire* samples thus concluding high liability to spoilage during storage.

CONCLUSION

Significant differences existed in the proximate and physicochemicals properties of *kilishi* samples stored with different packaging materials. The fat, protein, ash, carbohydrate, protein solubility, peroxide value thiobarbituric acid increased significantly while moisture, nitrogen, and pH decreased significantly after 3 months' storage in all the packaging materials. High density polyethylene retained higher levels of moisture, ash, fat, protein, nitrogen, pH, peroxide value thiobarbituric acid more than low density polyethylene and carton. In comparison, low density polyethylene retained more moisture, ash, fat, protein, nitrogen, peroxide value and thiobarbituric acid than carton.

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