

# Moisture sorption isotherm proximate composition, lipid oxidative stability and sensory characteristics of sun dried kilishi in storage

## ABSTRACT

The study investigated the relationship between moisture sorption isotherm and some quality parameters of sun-dried kilishi in storage using various methods of analysis and experimentation. The Guggenheim, Anderson, de Boer (GAB) model was used to determine sorption isotherm characteristics, thiobarbituric acid reactive substances (TBARS) assay was used to measure malondialdehyde (MDA) levels while procedures of Association of Official Analytical Chemists and 9- point hedonic scale were used to evaluate proximate composition and sensory quality respectively. The results of sorption indicated that the product has water activity and monolayer moisture of 0.39 and 15.82 (g H<sub>2</sub>O/g Solid) respectively while proximate composition of sundried kilishi before and after storage at 45 °C showed that the protein content of this product after 35 days of storage was significantly higher ( $p < 0.05$ ) than before storage. The study also revealed a decrease in fat content in storage, as malondialdehyde level increases from 0.223 to 0.341. Sensory characteristics were affected in storage. Aroma, flavour, juiciness and overall acceptability significantly decrease ( $p < 0.05$ ) after storage at 45 °C for 35 days as compared to the sensory status of this product before storage. In conclusion, moisture sorption and environmental conditions need proper control to keep sun dried kilishi stable on shelf.

**Keywords:** Adsorption, Kilishi, Packaging, Product, Shelf-life and Storage

## INTRODUCTION

Kilishi is a popular Nigerian dried meat snack that is rich in flavor and has a unique texture. The term "kilishi" is derived from the Hausa language, where "kilishi" means a thin sheet of dried meat that is seasoned with a mixture of spices and peppers [1]. Typically, kilishi is made from lean cuts of beef or goat meat that are marinated in a blend of spices such as ginger, garlic, cayenne pepper, and salt before being air-dried or sun-dried to preserve the meat [2].

Moisture sorption plays a critical role in the preservation of food products by influencing their shelf life [3], texture, and overall quality. When food is exposed to ambient moisture levels, it can lead to microbial

growth [4], enzymatic reactions [5] and chemical degradation, all of which can compromise the safety and sensory attributes of the product. By controlling the moisture content through sorption processes, the growth of microorganisms can be inhibited, enzymatic activities can be slowed down, and chemical reactions can be minimized [6]. This can result in extended shelf life, improved texture, and enhanced flavor retention of the food product. Therefore, understanding the importance of moisture sorption in food preservation is crucial for maintaining the quality and safety of food products, particularly in the case of sun-dried kilishi. The study of sorption isotherm in food products holds significant importance in understanding the behavior of moisture content over a range of relative humidity levels. This understanding is crucial for determining the equilibrium moisture content, which directly impacts the shelf life and overall quality of food products. By studying the sorption isotherm of sun-dried kilishi during storage, we can gain insights into the moisture adsorption and desorption characteristics of this traditional Nigerian meat product, aiding in optimal storage conditions and preservation methods [7]. Additionally, the sorption isotherm data can provide valuable information on the proximate composition changes in kilishi over time, helping to assess nutritional quality and safety aspects [8]. Therefore, delving into the sorption isotherm of food products like kilishi is essential for enhancing storage practices and ensuring product quality and safety in the food industry.

The importance of proximate composition analysis in food storage cannot be overstated. Understanding the chemical and physical properties of a food product is crucial for determining its shelf life, quality, and safety over time [12]. Proximate composition analysis provides valuable information about the moisture, fat, protein, ash, and carbohydrate content of a food item, which directly impacts its stability and susceptibility to spoilage. By conducting proximate composition analysis on sun-dried kilishi during storage, we can track changes in nutrient content and moisture levels, allowing us to make informed decisions about the best storage conditions to maintain its quality and extend its shelf life [13]. This analysis serves as a vital tool in the food industry to ensure that products remain safe for consumption while preserving their nutritional value and flavor profile. Ultimately, proximate composition analysis plays a critical role in optimizing food storage practices to enhance food quality and safety [14].

Lipid oxidation in food products, such as sun-dried kilishi, is a critical concern as it can lead to the development of rancidity, undesirable flavors, sensory degradation, consumer rejection, and the formation of toxic substances, highlighting the importance of controlling oxidative processes in the meat industry [15.] This oxidation process is primarily influenced by factors like temperature, oxygen exposure, and the presence of pro-oxidants [16, 17, 18]. Lipid oxidation in foods can result in the formation of free radicals, which not only deteriorate the lipids themselves but also affect proteins, causing oxidation and aggregation that can alter their physicochemical characteristics and biological functions, thereby reducing food quality [19]. Also, the evaluation of the sensory characteristics of kilishi stored under various conditions will provide insights into its sensory changes during storage.

However, there is a noticeable gap in the research pertaining to the sorption isotherm behavior of sundried kilishi and nutritional stability of this product stored above the temperature experimented to determine its water activity. This gap in knowledge is significant as it hinders the optimization of storage conditions and shelf life of kilishi products. Therefore, this research aims to fill this gap by investigating the sorption isotherm characteristics of sundried kilishi using analytical techniques and mathematical models. Understanding the sorption behavior of kilishi will contribute to the development of efficient storage and packaging strategies in the food industry.

## **2. MATERIALS AND METHODS**

### **2.1 Preparation procedures of sun-dried kilishi**

2000g of boneless beef was procured from a reputable beef seller in Ado Ekiti, Nigeria. All excess fats and adhering connective tissues were trimmed off from the beef and sliced into flat sheets of 3mm by thickness to facilitate absorption of slurry and drying process. All dried non-meat ingredients were mixed and oil was added to constitute a homogenous mixture. Flat thin sheets of semitendinosus muscle of beef were sun dried for at least 6 hours thereafter soaked it in the kilishi ingredient slurry for 1 hr 30 minutes. The soaked muscles were spread out on the oven rack for proper draining. Oven trays were slightly lined with oil, baking sheets were spread on the oiled tray, the drained raw kilishi were separately placed side by side on the flat trays and cooked in already preheated oven at 63° for 40 minutes. Mixture of tsire (suya spice), pepper and olive oil (slurry) were used to coat the already oven-dried muscles. These were

returned to the oven for 10 minutes for final drying. Processed kilishi product was cooled to room temperature prior to further analysis. The preparation procedure for kilishi was repeated three times to obtain three set of kilishi product for experiment to be carried out in triplicate.

**Table1. Ingredients composition of kilishi**

Ingredients	Quantity (gms)
1000g semitendinosus muscle of beef cattle	
Garlic paste	2.5
Ginger paste	2.5
Cameroun pepper	5.0
Chili pepper	5.0
Black pepper	5.0
Tsire (suya spice)	50.0
olive oil	10.0
Salt	2.5.
Thyme	2.5
Curry	2.5
Paprika	5.0
Turmeric powder	7.5
Total	100

## 2.2 Procedure of Adsorption

Using the static gravimetric method [20], the equilibrium moisture content of sun dried kilishi was determined at 30 °C. Five (5g) grams of sun dried kilishi in triplicate were placed in a mesh bag, which was dropped above the saturated salt solutions contained in a glass jar. The jar was tightly closed with the aid of petroleum jelly and placed in a temperature-controlled cabinet ( $\pm 0.2$  °C). The samples were equilibrated with set humidity conditions until a stable weight was achieved. The sample was weighed daily with digital mettler balance until the mass difference was less than 0.001g. The determination of the equilibrium moisture content was carried out by oven dry method, the sample was dried at 105 °C for 24 h. All the experiments were carried out in triplicate. Ten saturated salt solutions prepared ranged

between 0.030 to 0.970. The water activities and the salt solutions used were as reported [21] and this is as given in Table 2.

**Table 2: Names of saturated salt solution used for sorption experiment**

Salt	Aw (30°C)
Cesium Flouride	0.030
Lithium Chloride	0.113
Potassium Acetate	0.216
Magnesium Chloride	0.324
Potassium Carbonate	0.432
Sodium Bromide	0.560
Potassium Iodide	0.679
Sodium Chloride	0.751
Potassium Chloride	0.836
Potassium Sulfate	0.970

### 2.3 Determination of equilibrium moisture content

This was done as stated below:

$$EMC = \frac{W_e}{W_i} (M_i + 1) - 1 \quad (\text{Tantala et al., 2019) [22] \dots\dots\dots \text{Equation 1}$$

where  $W_e$  is the equilibrium weight of the sample (g),  $W_i$  is the initial weight of the sample (g), and  $M_i$  is the initial moisture content of the sample (g)

The suitability of GAB equation for  $a_w$  of 0 - 0.95 [23], was employed to analyze the water activity and monolayer moisture value of sun dried kilishi.

GAB Equation =  $\frac{M}{Mm} = \frac{ABaw}{(1-Baw)(1-Baw+ABaw)}$  [24] .....Equation 2

[25]

## 2.4 Stability test

Ten gramme (10g) of sun dried kilishi was stored in polyethylene bag of 20 micron thickness and 12cmX 8cm size for 35 days at 45 °C and relative humidity (RH) of 85% using Dynamic Thermal Humidity chamber. The set up was done in triplicate. The kilishi samples were sealed in the polyethylene bags. Nutritional composition such as protein, fat, ash, moisture content and carbohydrate were evaluated and compared with its nutritional composition before storage [27].

## 2.5 Sensory evaluation sun-dried kilishi

The sensory evaluation was conducted by the methods of Kumari et al. [26]. Semi-trained taste panelist comprising ten members of both sexes participated in the assessment of sensory properties of the samples. Individual unit cell housed the assessors and each person was given unsalted biscuits and table water to cleanse palate after each taste of the sample. Samples were labelled and independently evaluated using a 9- point hedonic scale ranked as follows; like extremely to very much (8–9 scores), like moderately to like slightly (5–7 scores), neither like nor dislike to dislike slightly or dislike moderately (2–4 scores) and dislike extremely to dislike very much (0–1 score) for aroma, flavor, tenderness, texture and over all- acceptability.

## 2.6 Proximate composition determination of sun-dried kilishi

Sun-dried kilishi samples were analyzed for proximate composition according to the methods outlined by the Association of Official Analytical Chemists (AOAC). In order to ensure correctness and reliability of the results, all analyses were conducted in triplicate [27].

## 2.7 Lipid oxidation

Thiobarbituric acid reactive substances (TBARS) assay was used to measure malondialdehyde (MDA) levels as a marker of lipid oxidation [28]. This was done in triplicate to ensure correctness of data.

## 2.8 Statistical analysis

Statistical analysis was carried out using IBM SPSS Statistics 20, One-way ANOVA Post Hoc Multiple Comparisons of Ryan-Einot-Gabriel-Welsch F' test at 0.05 significance level [29] to analyse proximate composition, sensory properties and lipid oxidation.

## 3. RESULTS AND DISCUSSION

Figure 2 showed the sample image of the product after production. Results and interpretation of sorption isotherm behavior of sun-dried kilishi revealed interesting insights into the moisture sorption characteristics of this popular Nigerian food product. The sorption isotherm data indicated a typical type 4 shape, suggesting kilishi's ability to exhibit a steep rise in adsorption at low pressures. The interpretation of this behavior highlights the importance of understanding kilishi's water activity levels to maintain its shelf stability during storage. These findings contribute significantly to the knowledge base on the moisture sorption properties of sun-dried kilishi, aiding in the development of appropriate storage and packaging strategies to ensure product quality and safety over time. Overall, the results underscore the need for precise control of environmental conditions to prevent moisture-induced deterioration in kilishi quality.

Nutritional composition analysis of kilishi in storage revealed that the moisture content of the product significantly increased in storage, leading to changes in its overall composition (Table 5). Specifically, the protein content was found to increase while the fat content decreased, resulting in alterations to the product's texture and flavor profile. Studies have shown that the moisture content of kilishi tends to decrease or increase post-storage, leading to potential changes in protein, fat, and carbohydrate concentrations [30]. The increase in moisture content of this product when stored at 45<sup>o</sup> C and 85% RH may be due to the influence of high relative humidity which enabled the product to gain moisture through the packaging material. Generally, the effects of storage conditions on the proximate composition of kilishi may be crucial considerations in maintaining the nutritional quality of this popular dried meat product

[31].The particle size of spices used in marination could affect the proximate composition, indicating the importance of spice particle size in meat composition [32].

The result of lipid oxidation showed that storage at 45<sup>o</sup> C and 85% RH led to increase in lipid oxidation. This might be connected with an increase in moisture content. As moisture levels increases, lipid oxidation showed a corresponding upward trend, suggesting a possible correlation between water moisture and oxidative processes. Research had revealed moisture content and temperature as significant factors in lipid oxidation, with higher levels of both leading to increased rates of oxidation [33, 34]. These findings emphasize the importance of controlling moisture content during storage to prevent lipid oxidation in kilishi. Understanding these outcomes is vital for enhancing the quality and shelf life of kilishi and similar products in the food industry. [35]. Also, exposure to high temperatures may lead to lipid rancidity, affecting the overall nutritional value of kilishi. Lipid oxidation under high temperatures can generate free radicals, which in turn can eventually lead to protein oxidation and aggregation, affecting the physicochemical characteristics and biological functions of proteins [19].

**Table 3. Sorption Isotherm of Sun Dried Kilishi**

aw (30 <sup>o</sup> C)	EMC	aw/M
0.030	5.78 ± 0.010 <sup>a</sup>	0.0052
0.113	6.10 ± 0.020 <sup>b</sup>	0.0185
0.216	10.45 ± 0.020 <sup>c</sup>	0.0207
0.324	11.20 ± 0.020 <sup>d</sup>	0.0289
0.432	16.13 ± 0.020 <sup>e</sup>	0.0267
0.560	22.52 ± 0.030 <sup>f</sup>	0.0249
0.679	24.25 ± 0.010 <sup>g</sup>	0.0279
0.751	25.15 ± 0.020 <sup>h</sup>	0.0299
0.836	27.31 ± 0.030 <sup>i</sup>	0.0306
0.970	29.22 ± 0.020 <sup>j</sup>	0.0332

EMC = M = Equilibrium Moisture Content

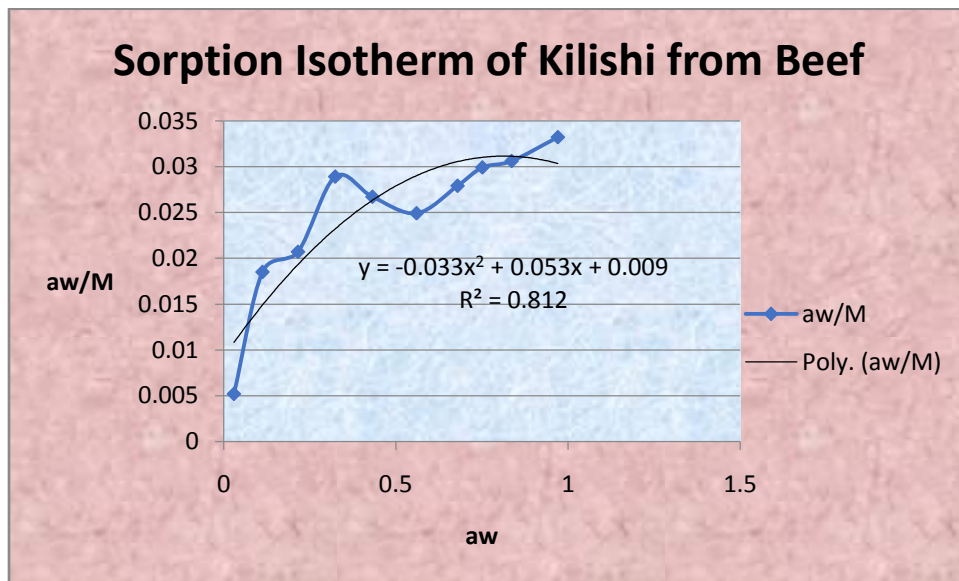


Figure 1. Adsorption Isotherm Curve of Sun Dried Kilishii

Table 4. Analysis of sorption data of Kilishii according to GAB Model

Product sample	Water activity ( $a_w$ )	Monolayer value ( $M_0$ ) (g H <sub>2</sub> O/g Solid)	$R^2$
Kilishi	0.39	15.82	0.812



Figure 2. Product sample of Sun Dried Kilishii

**Table 5. Proximate composition, Lipid oxidative stability and Sensory properties of Kilishi of sun dried kilishi before and after storage for 35 days at 45 °C and 85% RH.**

<b>Proximate composition</b>		
<b>Components</b>	<b>Composition (%)</b>	
	<b>Mean ±SD before storage</b>	<b>Mean ±SD after storage at 45 °C</b>
Crude protein	69.22±0.110 <sup>a</sup>	72.30±0.050 <sup>b</sup>
Moisture content	7.16±0.010 <sup>b</sup>	10.13±0.010 <sup>a</sup>
Ash	4.30±0.030 <sup>a</sup>	3.33±0.040 <sup>b</sup>
Fat	11.64±0.020 <sup>a</sup>	6.36±0.010 <sup>b</sup>
Carbohydrate	7.68±0.040 <sup>a</sup>	7.88±0.010 <sup>b</sup>
<b>Lipid oxidative stability</b>		
TBARS (MDA/Kg meat)	0.223±0.000 <sup>a</sup>	0.341±0.333 <sup>b</sup>
<b>Sensory properties of Kilishi</b>		
Aroma	7.17±0.010 <sup>b</sup>	5.33±0.293 <sup>a</sup>
Flavour	7.33±0.030 <sup>b</sup>	6.00±0.010 <sup>a</sup>
Juiciness	7.17±0.030 <sup>b</sup>	5.5±0.050 <sup>a</sup>
Texture	6.67±0.026 <sup>a</sup>	7.00±1.000 <sup>a</sup>

Tenderness	4.17±0.010 <sup>a</sup>	4.00±0.100 <sup>a</sup>
Overall acceptability	8.18±0.010 <sup>b</sup>	7.10±0.100 <sup>a</sup>

TBARS- Thiobarbituric Acid - Reactive Substance, SD- standard deviation

The results of sensory characteristics of sun-dried kilishi in storage revealed important insights into the behavior of the product over time. This information is crucial for determining the product's stability and shelf life. On the other hand, the sensory characteristics analysis in this study, provided valuable information on the changes in aroma, flavour and texture of kilishi during storage, offering a comprehensive understanding of its overall quality. The changes in sensory parameters of kilishi in storage might have been caused by decrease in fat during storage at relatively high temperature. Exposure to a temperature of 45<sup>o</sup> C and 85% RH may be responsible and these important factors of temperature humidity should be considered in order to prevent flavor alterations and rancidity in storage. By combining these results, we can make informed decisions about the storage conditions, and shelf-life recommendations for sun-dried Kilishi.

#### 4. CONCLUSION

The sorption isotherm analysis revealed that the moisture content of kilishi increased gradually over time, indicating the need for proper packaging and storage conditions to prevent moisture absorption and maintain product quality. Additionally, the lipid oxidation, proximate composition and sensory characteristics results demonstrated that the level of changes in nutritional status highlight the importance of maintaining storage below its water activity and good packaging materials maintain product quality and consumer satisfaction. Hence, kilishi should ideally be stored at a temperature much lower than 45<sup>o</sup> C and 85% RH to maintain its sensory characteristics and extend its shelf life. Also, regular monitoring of storage conditions such as temperature and humidity levels is essential to ensure the product's stability over time.

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