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MOISTURE SORPTION STUDIES USING THE GAB MODEL OF A BEEF BASED DAMBUN NAMA (A GROUND MEAT PRODUCT) STORED OVER A PERIOD OF SIX MONTHS

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ABSTRACT: Water adsorption isotherms of Dambun Nama, a ground beef product, were determined over a storage period of six months, at an average ambient temperature of $33.8^{\circ}C$ and an accelerated temperature of 50°C, using the static gravimetric *method. The study was aimed at establishing the moisture sorption* profile of a traditional meat product Dambun Nama common in Northern Nigeria, produced from beef and stored over a period of six months. The sample was produced using a standardized method; it was sterilized, packaged in six airtight glass containers and stored at ambient temperature as the sample stock for the analyses over a period of six months. Data was generated from the sorption studies based on the isopiestic transfer method using concentrated sulphuric acid solutions. The isotherms are generally shaped like the type 4 isotherms for food products. Between 33.8°C and 50°C adsorption, the monolayer moisture content ranged from 0.036 to 0.048 gH_2O/g solids, and between 0.037 to 0.049 gH₂O/g solids for desorption. There was a general increase in the GAB constants K and C. The correlation coefficient (R) values obtained for adsorption ranged from 0.941 to 0.959, and for desorption, the values ranged from 0.940 to 0.957, indicating that the GAB model was suitable in describing the moisture sorption profile of Dambun Nama within the prescribed water activity ranges and storage period.

KEYWORDS: Dambun Nama, Sorption isotherms, monolayer moisture content, Adsorption, Desorption.



INTRODUCTION

Dambun Nama is a traditional low moisture ground meat product common to Northern Nigeria. It is a locally processed product of reduced bulk that serves as a convenient source of animal protein. However, poor facilities for bulk storage, and the high potential for water reabsorption when exposed to increasing relative humidity, make its dispensing and retailing difficult even at the local level. Preliminary storage studies of process standardization of Dambun Nama using sensory evaluation had been carried out (Yusuf & Gambo, 2021). However, to establish standard and acceptable processing, packaging and storage methods, the basic scientific data of the product must be generated and utilized to that effect. One of the most important of these data for dehydrated products is the moisture sorption profile at a chosen storage temperature. This is because water profoundly influences stored product attributes, especially their quality and safety (Van den Berg, 1985). Moisture sorption studies have been carried out on numerous agricultural produce and products; however, the information on the sorption isotherms of Dambun Nama during a storage period of six months is unavailable in literature. The study was aimed at determining the moisture sorption characteristics of Dambun Nama between the temperatures of 33.8°C to 50°C as influenced by a six months storage period. The objectives of the research include assessing the influence of temperature and time on the sorption behaviour of the product, and analyzing the data generated using the GAB sorption model equation.

THEORETICAL UNDERPINNING

Moisture available for microbial and chemical activities plays a vital role on the storage stability of foods. Water availability (Leake, 2006; Yusuf et al., 2020; Roos et al., 2018) is the most important single factor governing food deterioration, of which microbial spoilage is usually the most rapid. As far as food spoilage is concerned, moisture availability has been identified as the most crucial factor because it forms the central coordinating parameter for all other spoilage agents, biological or chemical (Labuza, 2008; Mor-Mur & Yuste, 2010). To completely understand water relations in a product requires an understanding of the amount of water (moisture content) that can be held at a given energy state or water activity (Yusuf et al., 2020). Water in moist materials has an activity, which can reach the highest value of 1.0. This water in equilibrium with the surrounding water vapour determines the sorption isotherms of the materials (Zhen-shen et al., 2022), that is, the dependence between the moisture content (usually on dry basis) and water activity at constant temperature. The nature of this relationship depends on the interaction between water and other ingredients. The amount of water vapor that can be absorbed by a product depends on its chemical composition and physical structure. The sorption isotherms of biological and food materials are mostly of sigmoid shape (Brunauer, 1943; Blahovec & Yanniotis, 2009), and they had been classified according to their shape and processes (Bell & Labuza, 2000). The isotherms most frequently found in food products are the sigmoidal types 2, 3 and 4 (Blahovec & Yanniotis, 2009; Basu et al., 2006; Mathlouthi & Roge, 2003). The water sorption isotherms usually describe the relationship between the equilibrium moisture content and water activity at a certain temperature. These isotherms are important in optimising drying parameters, in calculating different thermodynamic properties such as isosteric heat of sorption, sorption entropy and Gibbs free energy, which provide information about food structure, energy requirements and molecular state of water within the products (Sánchez-Torres et al., 2021; Tunç & Duman, 2007).



A product's isotherm can also be used to determine its packaging requirements depending on the product's sensitivity to moisture and the type of conditions it may be exposed to. Generally, extended shelf-life of food can be achieved by reducing the moisture content to levels below those required by microorganisms (Andrade et al., 2011). Therefore, it is necessary to assess the relationship between moisture content of product and relative humidity of the atmosphere at a certain temperature, and this can be described by moisture sorption isotherms (Zhen-Shan et al., 2022). The sigmoidal sorption isotherms can be described by many sorption models, including the 'GAB' (Guggenheim, Anderson, and de Boer) model, which is a kinetic model and considered one of the most useful and versatile. It can be used for the assessment of the experimental data at water activity of between 0.1 to 0.9 (Van den Berg, 1984; Blahovec & Yanniotis, 2010), and it provides a good description of the sorption behaviour of almost every food product (Goula et al., 2008). GAB model equations have been adopted by the American Society of Agricultural Engineers for describing sorption isotherms (Jantawat & Siripatrawan, 2006).

METHODOLOGY

I. Materials

Fresh beef from skeletal muscle, seasonings, vegetable oil, water.

II. Sample Preparation

The sample used in this study was the *Dambun Nama* prepared based on the procedure established by Yusuf and Garba (2021), where the preparation and processing methods were standardized through sensory evaluation and particle size control using standard screens. Cubed fresh beef from skeletal muscle was seasoned, cooked until very tender, ground and aired. Sieves of mesh sizes 3, 4, 5 and 6 were used in the size sorting of the meat shreds. The retained shreds from mesh number 4, with an average shred size of 0.195 inches were adopted as the sample because their sizes compared well with the average shred size of the locally hand sorted sample, which had an average shred size range of between 0.191 to 0.221 inches. The sample was slowly and gradually stir-fried, with the average meat temperature not exceeding 110°C, until the shreds turned to a uniform golden brown colour, with no evidence of steam coming out of the pan. The product was then spread on a stainless-steel tray to aid its cooling and the escape of any residual moisture.

The percent proximate moisture, ash, fat, protein and carbohydrate contents of the product were 6.37%, 7.89, 14.20, 71.23 and 0.31 respectively, as determined by the standard procedures of Nielson (2010).

III. Experimental Procedure and Design

Both adsorption and desorption experiments of the sorption studies were carried out on the sample after every four weeks. The equilibrium moisture content was determined using the static gravimetric method (Arslan-Tontul, 2021; Yusuf et al., 2020; Stepien et al., 2020; Jowait et al., 1983) by exposing the *Dambun Nama* samples to different environments of constant relative humidity inside airtight containers. The environments were created by preparing different concentrations of sulphuric acid (H₂SO₄) that corresponded to the water activities for adsorption and desorption, from a_w 0.981 to 0.167, and 0.981 to 0.188, respectively, for the temperature range between 33.8°C and 50°C. The method had been found to be more flexible



and versatile in terms of food materials to be tested (Yusuf et al., 2020). It is accurate, simple, inexpensive and suitable for routine laboratory measurements that depend on the equilibrium of the water activities in two materials in a closed system. Each set of experiments was done in duplicates. The samples for 50°C were kept inside an incubator to maintain the constant temperature. The other set of samples were kept at ambient temperature during the study period, which was an average of 33.8°C. The weight of the samples in each water activity condition was taken at 24-hour intervals until the change in sample weight between two successive readings was not more than 0.009 g (Yusuf & Abubakar, 2011). This was recorded as the equilibrium point, and it was reached between an average of 6 days. The final moisture content for each duplicate was determined by the oven drying method at 105°C, and mean values were recorded.

The experiment was therefore laid in a $1 \ge 1 \ge 1 \ge 6$ factorial arrangement of a completely randomized design, that is, 1 model equation 1 sorption situation to obtain the sorption information on 1 product during 6 months of storage. The model for the factorial experiment is:

 $y_{ijgk} = \mu + A_i + B_j + D_g + C_k (AB)_{ij} + (AC)_{ik} + (BC)_{jk} + (DC)_{gk} + (ABDC)_{ijgk} \varepsilon_{ijgk}$

where:

 y_{ijgk} = observation in a beef product (*A*), 1 model (*B*), 2 sorption situations (*D*), and 6 months storage period (C); μ = the overall mean; A_i = the effect on 1 product; B_j = the effect of 1 model;

 D_g = the effect of the sorption situations; C_k = 6 months storage period; $(AB)_{ij}$ = the effect of the interaction of 1 product with 1 model; $(AC)_{ik}$ = the effect of the interaction of 1 product with 6 months storage period; $(DC)_{gk}$ = the effect of the interaction of 1 product with 2 sorption situations;

 $(BC)_{jk}$ = the effect of the interaction of 1 model with 6 months storage period;

 $(ABDC)_{ijgk}$ = the effect of the interaction of 1 product with 1 model, 2 sorption situations, and 6 months storage period; ε_{ijgk} = random error with mean 0 and variance σ^2

IV. Fitting of EMC Data to the GAB Sorption Equation

From the experimental data, the equilibrium moisture content (EMC) of the sample for both adsorption and desorption was calculated at each water activity using:

$$EMC = \frac{Total\ moisture}{Solid\ content}\ (\frac{g\ water}{g\ solids})$$

The experimental EMC values at each water activity for adsorption and desorption were fitted into the equation of a moisture sorption isotherm model, the Guggenheim-Anderson-De Boer (GAB) model. The equations used are as shown below:

$$\frac{a_w}{\left[(1-Ka_w)M\right]} = \frac{1}{M_o CK} + \left[\frac{C-1}{M_o C}\right]a_w$$

The polynomial form of the GAB equation is represented as:

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$$\frac{a_w}{M} = \propto a_w^2 + \beta a_w + \gamma$$

The constant K accounts for the value of the multilayer, which has different properties with the monolayer and with liquid water. K can be calculated from the quadratic equation formed from the GAB formula and from the equation of a second-degree polynomial trendline.

$$K = \left(\sqrt{\beta^2} - 4\alpha\gamma\right) - \frac{\beta}{2}\alpha\gamma$$

The constant C which represents the total heat of the first layer was calculated using:

$$C = 2 + \frac{\beta}{\alpha} K$$

The GAB monolayer moisture content was calculated by the equation:

$$M_o = \frac{1}{\sqrt{\beta^2 - 4 \propto \gamma}}$$

where:

 a_w represents water activity, M is the moisture content in % (dry basis), M_o is the Monolayer moisture content. α , β , and γ are values obtained from the trendline equation representing the different groupings in the GAB formula. (Labuza, 2008; Yusuf *et al.*, 2020).

RESULTS AND DISCUSSION

Sorption Studies at 33.81°C and 50°C Using the GAB Model

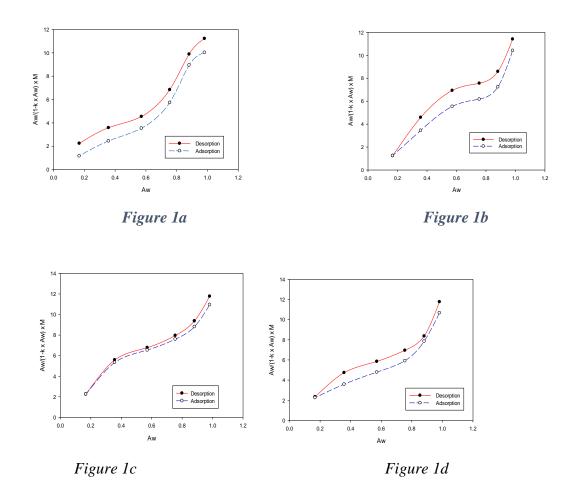
The GAB isotherms of *Dambun Nama* at 33.8°C and 50°C are generally shaped like the type 4 isotherms for food products. Type 4 isotherms indicate the behaviour of a swell-able hydrophilic solid (Basu et al., 2006). The two curves on each graph indicate the adsorption and desorption behaviour of the product at a particular temperature condition and a specific storage period. The different paths followed by each curve indicates the phenomenon of hysteresis that is often encountered in sorption isotherms. The inflection points that are generally observed on both the adsorption and desorption paths in all the months reflect transition points in the binding energy of water (Arun & Sakamon, 2000) indicating different types of water binding in the food product.

From most of the sorption plots of the product, it was observed that there was a distinct hysteresis effect, and in some cases the two plots are very close to each other, indicating similar positions at the same temperature and water activity. The shape and width of sorption hysteresis loops are dependent both on temperature and pore diameter of the sample. A wide hysteresis loop is expected if network and pore blocking effects are present, and the distribution of pore size and shape is not well defined (Yusuf et al., 2020; Thommas *et al.*, 2002; Rouquerol *et al.*, 1999). Therefore, the characteristic feature of any type of hysteresis loop is associated with the geometry and the texture of the sample or adsorbent. Absorption of significant amounts of



water into the internal structure of a solid had been shown to influence the properties of the solid (Yusuf et al., 2020). This becomes obvious in the width of the hysteresis path when plotted, as shown especially in the plots of months two and four. The phenomena is more in materials that are amorphous, and Labuza (1984) also reported that hysteresis is related to the nature and state of the components in a food, reflecting their structural and conformational arrangements, which alter the accessibility of suitable polar sites, under different conditions of temperature and water activities. This may hinder the movement of moisture in or out of the food.

Sorption Isotherms of Dambun Nama at 33.8°C





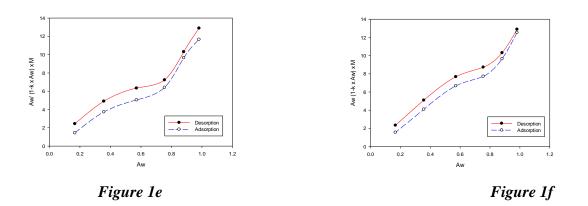


Fig 1a to 1f: Isotherms at 33.8°C in Months 1 to 6

It was also reported that hysteresis is generally attributed to the condensation of some of the water in the capillaries (Kapsalis, 1987). During the months of study, there was a consistent decrease in monolayer moisture content as the temperature rose from 33.8°C to 50°C, with values for adsorption ranging between 0.035 to 0.048 g H₂O/g solids equivalent to 3.5 to 4.8 g $H_2O / 100$ g solids. For desorption, the values ranged from 0.037 to 0.049 g H_2O/g solids, equivalent to 3.7 to 4.9 g H₂O / 100 g solids. A decrease in GAB monolayer moisture content (M_{0}) with an increase in temperature is an indication that the absorbed molecules gained kinetic energy (Arslan-Tontul, 2020), making the attractive forces to loosen, and thus allowing some water molecules to break away from their sorption sites, thereby decreasing the equilibrium moisture values. McMinn and Magee (2003) and Brennan et. al; (1990) reported that the monolayer values for most foods lie in the range of 0.05 to $0.11g H_2O/g$ solids, or 5.0 to 11 g H₂O/ 100 g solids. However, the structural changes induced by temperature (Barreiro et al., 2003; Kim et. al., 1998; Tunc & Duma, 2007), or the process of moisture removal, can cause a decrease or increase in the monolayer moisture content. This is as a result of the changes in the number of active polar sites due to chemical and physical changes (Machalkova et. al., 2014; Rosa et al., 2021).

There is also a significant decrease in the monolayer moisture content values with an increase in temperature (Labuza *et. al.*, 1985; Van den Berg, 1985, Kaymak & Sultanoglu, 2001). The monolayer moisture contents were less than 0.1 gH₂O/g solids (db) in all the months, which was the maximum value reported for food materials (Labuza *et. al.*, 1985). There was a general increase in the constants K and C_G as the storage time increased. An increase in K was an indication that at higher temperature, the multilayer molecules became more entropic, while a decrease of C was an indication of more enthalpy and a gain in kinetic energy resulting in a significant loss of moisture at higher temperatures (Seth et al., 2018; Diosady *et al.*, 1996). At certain temperatures, water vapour is absorbed by amorphous solids, and not simply adsorbed (Labuza, 2008). This can change the bulk properties of that solid, as reflected by the increase of K values with a corresponding decrease of the monolayer moisture contents. According to Rahman (1995), macromolecules such as starch, protein, and agar usually have higher monolayers, while high fat foods such as avocado, peanuts, and whole milk showed lower monolayers. This is reflected in the relatively high monolayer moisture contents obtained with *Dambun Nama* being a high protein food. He reported that a range of 0.01 to 0.14g H₂O/g



solids of monolayer moisture contents had been recorded for foods and food components. According to Barreiro *et al.* (2003), and Kim *et al.* (1998), structural changes induced by temperature, or the process of moisture removal, can physically and/or chemically affect the active polar sites causing changes in the monolayer moisture content (Machalkova *et al.*, 2014). This was evident in the decrease of the monolayer moisture content of the sample as the storage period progressed at the experimental temperatures.

According to Labuza et al. (1985) and Van den Berg et al. (1985), there is a significant decrease in the monolayer moisture content values with an increase in temperature. Park et al. (2001) also reported that when globules of fat surround proteins and/or starch granules of a food material, it means that there is an increase of hydrophobic constituents like fat, which binds very little water, thereby decreasing the monolayer value. It was observed that as the storage life progressed, there was a leaching out or separation of the oil used during the processing of the product, forming a layer on the surface of the product. Ali et al. (2001) reported that the storage of fat containing foods over temperatures above 30°C causes fat migration. This they reported as a result of the leakage of liquid glycerides from the centre of the product to the surface. The correlation coefficient (R) ranged from 0.998 to 0.999 for adsorption data, while the coefficient of determination (R²) ranged from 0.997 to 0.999. For desorption, R ranged from 0.997 to 0.999, and R² ranged from 0.994 to 0.999. The correlation coefficient is used to assess the degree of association between two variables, factors, or data sets. It is a statistical measure of the strength of a linear relationship between two variables. Its values can range from -1 to 1. A correlation coefficient of -1 describes a perfect negative or inverse correlation, with values in one series rising as those in the other decline, and vice versa. A coefficient of 1 shows a perfect positive correlation or a direct relationship. A correlation coefficient of 0 means there is no linear relationship (Fernando, 2023). The results showed very strong positive linear correlation, indicating a linear relationship between Aw and EMC for both adsorption and desorption.

The coefficient of determination values (\mathbb{R}^2) represents the percent of the data that is the closest to the line of best fit, or the total variation in EMC that can be explained by the linear relationship between Aw and EMC. \mathbb{R}^2 is a measure that allows us to determine how certain one can be in making predictions from a certain model or graph (Mathbits, 2016). While the correlation coefficient quantifies the direction and strength of a linear relationship between two variables, the coefficient of determination represents the variance proportion in the dependent variable explained by the independent variable. It captures how well predictions match observations, or how much of the variation in observed data is explained by the predictions. \mathbb{R}^2 values closer to 1 indicate strong model explanatory power (Learn Statistics easily, 2023). From the \mathbb{R}^2 results, it shows that a high percentage of the data is close to the line of best fit, meaning excellent predictions can be made using the GAB model on the product. Table 1 shows summaries of the monolayer moisture contents and the constants obtained for *Dambun Nama*.



Sorption Isotherms of Dambun Nama Using the GAB Model at 50°C

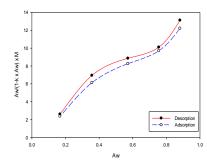


Figure 2a

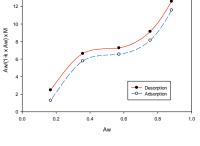


Figure 2b

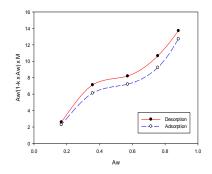
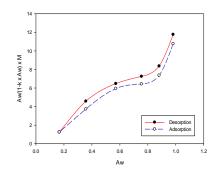


Figure 2c





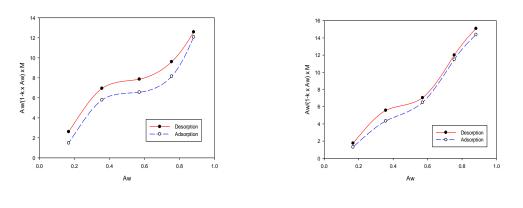


Figure 2e

Figure 2f

Figures 2a to 2f: Isotherms at 50°C in Months 1 to 6

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Model											
Μ	Тур	Sorption Values at 33.8°C					Sorption Values at 50°C				
	e	R	\mathbb{R}^2	Κ	CG	Mo	R	\mathbb{R}^2	Κ	CG	Mo
1	Ads	0.999	0.998	0.558	2.373	0.048	0.999	0.999	0.971	2.388	0.039
	Des	0.999	0.999	0.576	2.378	0.049	0.999	0.999	1.005	2.489	0.040
2	Ads	0.999	0.998	0.584	2.538	0.048	0.999	0.999	0.989	2.553	0.039
	Des	0.997	0.994	0.614	2.399	0.049	0.998	0.998	1.043	2.547	0.038
3	Ads	0.998	0.997	0.749	2.438	0.047	0.999	0.998	1.048	2.529	0.038
	Des	0.998	0.998	0.724	2.403	0.047	0.999	0.999	1.060	2.537	0.037
4	Ads	0.998	0.997	0.751	2.439	0.047	0.999	0.998	1.090	2.531	0.037

0.048

0.046

0.047

0.046

0.047

0.999

0.999

0.999

0.999

0.999

0.998

0.999

0.999

0.999

0.999

1.080

1.098

0.980

0.994

0.960

2.534

2.539

2.542

2.543

2.523

0.037

0.036

0.037

0.036

0.038

Table 4.13: Sorption Values for Dambun Nama at 33.81°C and 50°C Using the GAB Model

 $M = Months; C_G = Guggeinheim Constant; R^2 = Correlation Coefficient; M_O = Monolayer Moisture Content (g H₂O / g solids); R = Coefficient of Determination; Ads = Adsorption; De = desorption.$

CONCLUSION

Des

Ads

Des

Ads

Des

5

6

0.997

0.999

0.999

0.998

0.999

0.995

0.998

0.999

0.997

0.998

0.749

0.765

0.765

0.789

0.769

2.411

2.448

2.451

2.467

2.481

It can be concluded from the study that an increase in temperature from 33.8° C to 50° C caused a decrease in the monolayer moisture content (M_o) in all the months in the six months period of study, for both adsorption and desorption. The monolayer moisture contents (Mo) were less than 0.1 gH₂O/g solids (dry basis), which was the maximum value reported for food materials. Also, as the monolayer moisture content decreased, the constants K and C_G increased with an increase in temperature. From the values of the coefficients and the constants, and based on the range of monolayer moisture contents obtained, the GAB model gave a good fit to the experimental sorption data for the range of water activities used, and is suitable in describing the sorption characteristics of *Dambun Nama* over a storage period of six months. It was concluded that as the storage period of *Dambun Nama* increases, the level of safe moisture for its storage decreases, making it more vulnerable to biological and chemical activities that will negatively affect its attributes.

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