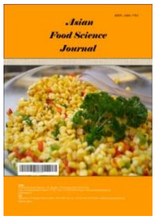

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Moisture Sorption Characteristics as Influenced by Application of Hurdles in *Dambu-nama* Production

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Authors' contributions

This research work was carried out in collaboration among all authors. Author MOE designed the study, performed the statistical analysis. Author CCA wrote the protocol, wrote the first draft of the manuscript and managed the analyses of the study. Author MAI managed the literature searches. Author SMG assisted in the compilation of the manuscript. All Authors read and approved the final manuscript.

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ABSTRACT

Moisture transfer characteristics of *dambu-nama* (DN) as influenced by addition of citric acid, salt and sugar as hurdles were investigated. Four products comprising DN with 0.1% citric acid + 2% salt and 2% sugar ($DNC_{0.1}$); DN+ 0.2% citric + 2% salt + 2% sugar ($DNC_{0.2}$); DN + 0.3% citric acid +2% salt + 2% sugar ($DNC_{0.3}$) and a control DN without citric acid , salt or sugar (DNC_0) were produced and subjected to moisture sorption studies at different temperatures (30, 40, 50 and 60^{o}C) and relative humidities of(10-96%). Incorporation of the hurdles into *dambu-nama* resulted in increase in monolayer moisture contents, surface areas, net isoteric heats and entropy of sorption. The isotherms obtained were best described by the Henderson model followed by the GAB and Oswin models. The hurdles also resulted to a J-shaped isotherm instead of the sigmoidal isotherm shape.

Keywords: Moisture sorption; hurdles; dambu-nama; influenced and hystersis.

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1. INTRODUCTION

Dambu-nama is a Nigerian traditionally dried meat product. It is commonly obtained from beef, goat meat, mutton or camel meat and is popularly consumed in the Northern parts of Nigeria. Proximate analysis by [1] indicated that the product contains 46.51% protein, 15.65% fat and 5.76% ash. The common ingredients used for the production of *dambu-nama* are spices and seasoning such as garlic, ginger, onions, chillies, salt and neutralized hydrolysed vegetable proteins. According to [2] and also [3], *dambu-nama* has good nutritive values but poor keeping quality at room temperature. It can serve as a snack or combined with other foods as part of daily diet for the consumers. It also provides a convenient dry pack for travellersand campers because of its light weight, easy- to pack, tender and ready- to- eat qualities [1].

Hurdle technology is the application of combinations of microbial and enzyme inhibitory agents (such as organic acids, salt and sugar) in food preservation. The technology is based on synergistic effects of the hurdles which are employed at very low concentrations. This trend emerged as a result of consumer demand for minimally processed and yet acceptable and safe food products. Meat deterioration begins immediately following slaughter due to chemical, enzymatic and microbiological (bacteria, yeast and moulds) activities resulting in oxidative changes, discolouration, mouldiness, off-flavour, slimness etc. The major deteriorative agents are microorganisms which render the meat unacceptable and unfit for human consumption [4]. Chemical preservatives are used to create hurdles to these agents thereby assuring safety, stability and enhanced sensory and nutritional qualities of food products [5]. From an understanding of the hurdle effects, the concept of hurdle technology was developed [6]. This means that hurdles are deliberately combined to improve microbial stability, sensory attributes, nutritional and economic properties of foods. Thus, hurdle technology aims to improve the total quality of foods by judicious application of combination of hurdles. According to [7], the most important hurdles used in food preservation are temperature (high or low), water activity (a_w), acidity (pH), redox potential (Eh), preservatives (e.g. sugar, salt, citric acid nitrites, sorbate, sulphite) and competitive microorganisms (e.g. Lactic acid bacteria).

In developing countries the application of hurdle technology is important for stability, safety and acceptability of meat products that do not require refrigeration. This is because of poor handling and processing methods which tend to lower safety and storage stability of traditional meat based products.

According to [8] and also [9], citric acid is used in meat marinates as a chelator of pro-oxidants and to improve the water holding capacity and tenderness of beef muscle. Studies by [10] indicated that the use of hurdles such as organic acids and salt solutions promoted shelf-life extension and inhibition of growth of microorganisms [11]. Also reported that the use of sodium lactate and lactic acid enhanced the chemical, microbiological and sensory characteristics of marinated chicken.

The application of hurdles such as citric acid, salt, sugar and spices would further enhance the storage stability, sensory, nutritional and microbiological stability of *dambu-nama* [12,13]. According to [14], there is increasing consumer demand for meat products that are low in salt, fat, cholesterol, nitrites and calories with higher health promoting bioactive components such as carotenoids, unsaturated fatty acids, sterols and fibres.

Therefore, there is the need for the application of hurdle technology to the processing and production of *dambu-nama*. Information about the influence of hurdles on the moisture transfer characteristics of *dambu-nama* are lacking.

2. MATERIALS AND METHODS

2.1 Source of Raw Materials

Sixteen (16 kg) of pre-rigor hind quarters beef were purchased from the abattoir of Makurdi Modern Market within 2 h of slaughter of cow. The sample was placed in plastic buckets with ice blocks and transported promptly to the laboratory where they were trimmed of fats, washed with chilled deionised water and packed in black polyethylene bags. The packs were kept in a household deep freezer (model: HR 98; manufacturers: Thermacool) and used for *Dambu-nama* production within 24hours.

Food grade common salt (iodized NaCL), sugar, citric acid (BDH), vegetable oil, Spices (powdered mixture of dehydrated onions, ginger, garlic and chillies) and neutralized hydrolysed vegetable protein (“maggi cubes”, Food Specialities Limited, Nigeria) were purchased from a supermarket in Makurdi. The spices and ground maggi cubes were mixed in a ratio 1:1 (w/w) to obtain the spices used for this study.

The research outlay was a 4×4×4×4 randomized experimental design comprising 4 levels each of citric acid (0.2,0.4,0.6,0.8%), salt (1,2,3,4%), sugar (1,2,3,4%) and spices (2,3,4,6%) was employed to obtain a possible 256 combinations. Because of this large sample size, sensory evaluation based on evolutionary operations (EVOP) using table of random numbers was employed for eliminations. The preliminary sensory tests suggested that citric acid was the most critical hurdle affecting acceptability of the *dambu-nama*. At concentrations of 0.4% and above (citric acid), > 2% for salt and sugar, the products were either too harsh or sweet for acceptance. With sugar and salt at 2% level each, a 4 × 4 design (0.1, 0.2, 0.3, 0.4% citrate; 1, 2, 3, 4, 5% spices) that gave 16 possible combinations was then used for further eliminations to obtain the three (3) most preferred product samples.

$DNC_{0.1}$ = *Dambu-nama* + 0.1% citric acid + 2% salt + 2% sugar + 4% spices
$DNC_{0.2}$ = *Dambu-nama* + 0.2% citric acid + 2% salt + 2% sugar + 4% spices
$DNC_{0.3}$ = *Dambu-nama* + 0.3% citric acid + 2% salt + 2% sugar + 4% spices

These 3 products, together with a control sample (DNC_0) containing only 4% spices (as in traditional products) which served as a control were used for moisture transfer study.

Essentially for each product, 4 kg of beef were manually cut into thin slices (approximately 2×2×2 mm, length × width × thickness) using sharp stainless steel knives. The meat slices were mixed with appropriate ratios of citric acid, salt, sugar and spices followed by simmering in stainless steel pressure cookers for about 10mins. The cooked meats together with resultant stock were pounded using previously washed mortar and pestle into a mash. After separation of the meat strands, they were dried in an air draft electric oven (Model: TT-9053, Techmel and Techmel USA) at 60°C for 30 min. This enhanced products handling and moisture reduction.

The strands were then deep fried in vegetable oil over an electric heater for 5min. After frying, the strands were drained in a plastic basket, dried in the oven at 60°C for 3 h followed by cooling, packaging and storage in plastic containers with tight lids.

2.2 Moisture Sorption Studies

Sorption studies were performed as described by [15]. Six concentrations of sulphuric acid (10,20, 30,40,50 and 60%) were used to provide constant relative humidity's ranging from 0.1677 to 0.9570 at 30,40,50 and 60℃, respectively. About 200ml of each acid solution were introduced into 500 ml airtight plastic containers. Wire gauzes were force-fitted into the plastic containers over the saturated solution=ns to hold samples. When required for desorption studies, ground samples (DNC_0, $DNC_{0.1}$, $DNC_{0.2}$ and $DNC_{0.3}$) were rewetted by sprinkling with de-ionized water and mixing with a spatula. The rehydrated samples were allowed to equilibrate in household refrigerator for 24 h before use.

In order to avoid possible contamination caused by the development of microorganisms, especially at high water activities, 5ml of toluene was measured into small glass bottles and placed in the plastic container. Duplicate samples, each 0.5 g of dried or rehydrated samples were weighed in crown cork and placed on the wire gauze above the solutions for adsorption or desorption isotherm studies respectively. The containers were covered tightly with lids and allowed to equilibrate in thermostatically controlled incubators (Model Biochemical Incubator LRB-70f, England) set at selected temperatures (30,40,50 and 60℃). The samples were removed and weighed every 2 days until differences between three (3) consecutive readings were < 0.5% of sample weight. The total time for removing and putting back in the airtight container was about 2-5 minutes as recommended by the cooperative project, COST 90 [16]. This minimized atmospheric moisture sorption or desorption during weighing. The equilibrium moisture contents (EMCs) were determined by material balance from the initial moisture content for both dried and rewetted samples [17].

2.3 Moisture Sorption Data Analysis

From sorption data generated, the EMC versus a_w for each temperature were plotted to obtain moisture sorption isotherms. The EMC vs a_w data were fitted with BET, GAB, Henderson and Oswin models. The BET monolayer moisture contents were calculated from the BET plots using data up to 0.46 a_w since the BET model is usually valid only for water activities below 0.5 [15]. The GAB monolayer moisture values were calculated from data in the a_w range 0.11- 0.85.

The monolayer moisture values were used to determine the apparent surface areas of the sorbent (S_o) since

$$S_{o=} (1/M_s)\ N_o\ A\ M_o\ (m^2/g\ \text{solid}) \quad (1)$$

where
M_s is the relative molecular mass of water, N_o is Avogadro's number (6.023×10^{23} molecules/ mol) and A is apparent surfaces area of one water molecules ($1.05\times10^{-19} m^2$).

Net isosteric heats and entropies of sorption were deduced from Clausius-Clapeyron equation (13).

2.4 Statistical Analysis

Parameters for the BET, Henderson and Oswin models were analyzed by least square linear regression and the GAB quadratic functions were analyzed using SPSS package. Accuracies of fit of the models were verified by calculating the root mean square of error (%RMS) according to [18] as follows:

$$\%\ RMS = \sqrt{\frac{\Sigma\left(\frac{M_{obs}-M_{est}}{M_{obs}}\right)^2}{n}} \times 100 \quad (2)$$

Where M_{obs} and M_{est} are experimental and predicted moisture values respectively and n is number of experimental data.

3. RESULTS AND DISCUSION

3.1 Moisture Sorption Characteristics

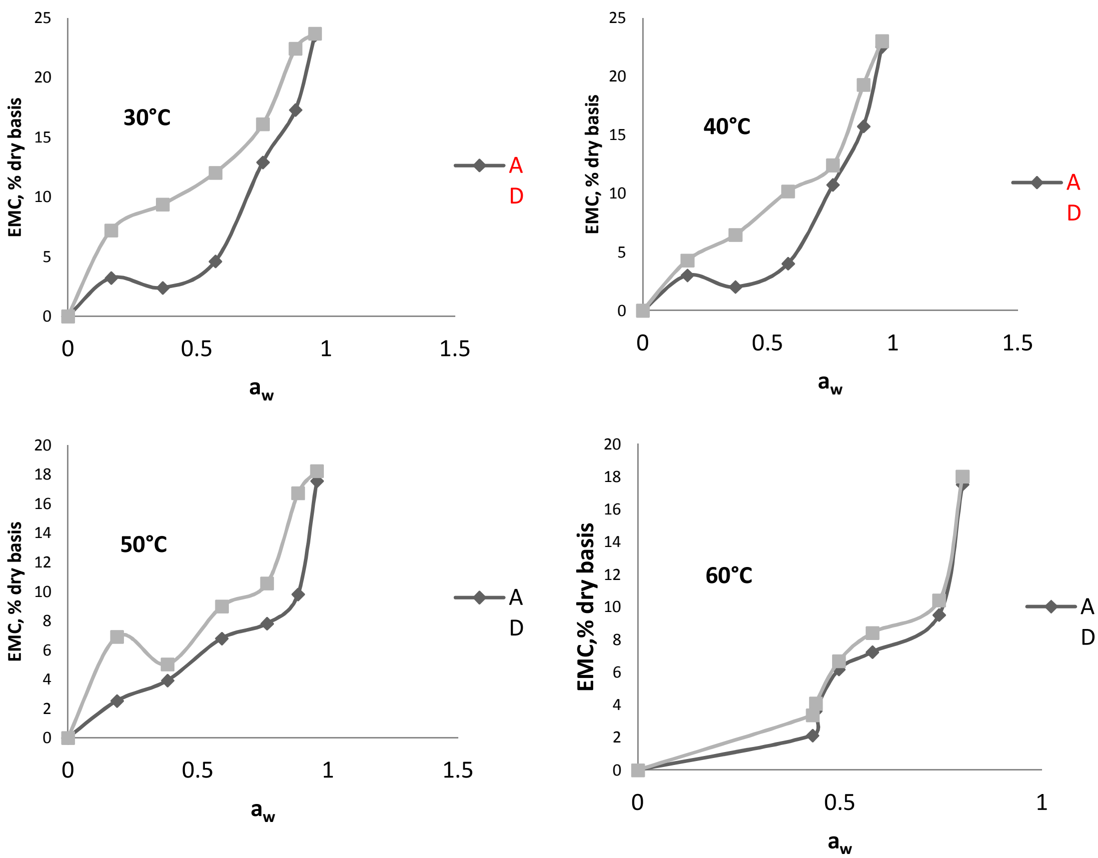
The moisture adsorption and desorption Isotherms of hurdled *dambu-nama* samples as influenced by the application of hurdles are as shown in Figs. 1 and 2, respectively. *Dambu-nama* without the hurdles exhibited sigmoidal-shaped curves which are typical of most food materials while products containing the hurdles tended to be J-shaped. Equilibrium moisture content (EMC) for both adsorption and desorption decreased with increase in temperature at constant water activity. The addition of hurdles (citric acid, salt, sugar and spices significantly ($p > 0.05$) affected the EMC values. The EMC values were lower for untreated sample (DNC_0) than those with hurdles. The EMC of *dambu-nama* products treated with hurdles decreased with increasing level of citric acid. Data from Figs. 1 and 2 were reploted for each *dambu-nama* product and presented in Figs. 3 to 6. These plots indicated differences in adsorption and desorption isotherms patterns of *dambu-nama*. The hysteresis loop observed became more pronounced as temperature increased.

3.2 Adsorption and Desorption Isotherms

3.2.1 The effects of water activity (a_w) and temperature on the EMC of *dambu-nama*

Adsorption and desorption isotherms of the *dambu-nama* products treated with the hurdles were J-shaped and conformed to the type 3 classification [19,20,15]. The control product exhibited sigmodial S-shape typical of most food materials [21]. According to [22], a typical sigmoid sorption isotherm is divided into three different parts. The first region is the monolayer region (a_w 0 to 0.22) in which the water is bounded by the hydrophilic water-ion or water dipoles interactions. The water in this phase is the most strongly adsorbed, immobile and depends on the chemical composition of the products and is also affected by pre-treatments. The second region corresponds to the linear portion of the isotherm (aw 0.22 to 0.73) which represents the water held in the matrix. The third region (aw 0.73 to 1.00) corresponds to the last concave region which represents the least strongly bound water and the most mobile which is designated as bulk water phase [23]. It can be observed from the isotherms (Figs. 3 to 4) that at constant water activity, EMC increased with decreasing temperature. The EMC of *dambu-nama* increased with increase in values of water activity at all temperatures. This trend is consistent with the findings of earlier workers [24,15,25,26]. For both desorption and adsorption, the equilibrium moisture content, increased with increasing water activity, the increase being more profound within water activity range of 0.6 to 0.9. This implies that at a water activity > 0.5, microbial growth, enzymatic reactions and lipid oxidation will occur much faster leading to rapid spoilage of *Dambu-nama* samples especially the hurdleless samples.

According to Caurie [27], changes in vapour pressure in foods with atmospheric humidity result in the characteristics sigmoid shape of water sorption isotherms. The isotherms crossing observed for both adsorption and desorption modes can be attributed to conformational changes in state of water and the adsorbent. Similarly, isotherm crossing behaviours were also reported for whey proteins, pure and blended carbohydrate polymers [28]. The J-sahped isotherms are not uncommon in food isotherms. Earlier workers [29] reported J-shaped isotherms for salted catfish. The characteristic increase in EMC with increase in a_w can be attributed to high affinity for moisture by the salt and sugar which dissolves causing swelling of the *dambu-nama* thereby exposing more active binding sites. The EMCs decreased with increase in temperature hence the isotherms were lower as temperature increased. At any given moisture there was a consistent shift of equilibrium relative to higher values with increase in temperature. The *dambu-nama* products became less hygroscopic with increase in temperature. Increase in temperature caused lowering of isotherm curves which increases thea_w at a constant moisture content thereby making the product more susceptible to microbial spoilage. Similar trends were also observed for fish flour by [30] and fresh water crayfish by Ariahu et al. [15]. As the temperature increased the excitation of molecules as well as the distance and thus attraction between molecules varies [31]. This observation implies that an increase in temperature increases the a_w at constant moisture content, making the product more susceptible to microbial, nutritional and aesthetic degradation [32]. Higher adsorption isotherms imply higher ease of moisture uptake. Therefore effective packaging in moisture barrier packaging materials is suggested for storage of *dambu-nama* treated with the hurdles. Higher desorption isotherms have dehydration implications in that it indicates that the *dambu-nama* treated with the citric acid, salt and sugar will be more difficult to dry. This can be attributed to high affinity for water and hygroscopicity of the citrates, salt and sugar resulting in tendency to hold on tightly to bound water.



Figs. 1 and 2. Moisture sorption isotherms (adsorption and desorption) of $DNC_{0.3}$ at 30-60°C
$DNC_{0.3}$ = Dambu-nama + 0.3% citric acid + 2% salt +2% sugar + 4% spices

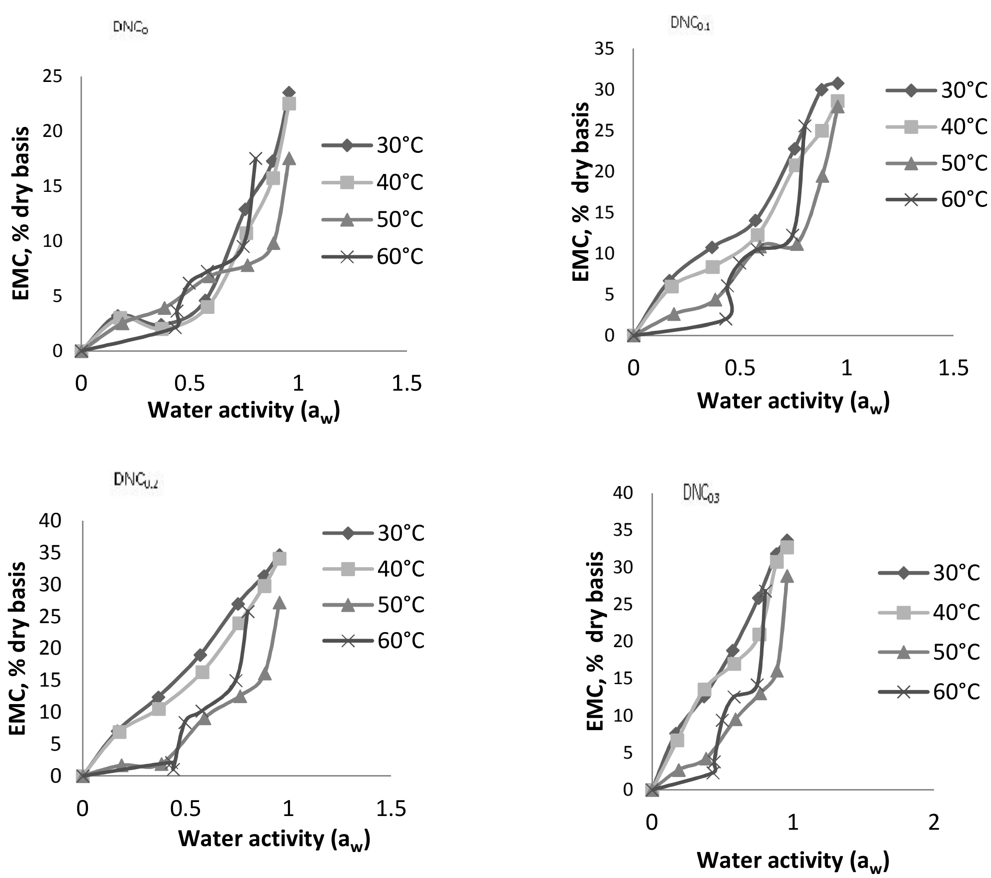


Fig. 3. Moisture Adsorption Isotherms of *Dambu-nama* as Influenced by Hurdle

DNC_0 = Dambu-nama + 4% spices (control),
$DNC_{0.1}$ = Dambu-nama+ citric acid 0.1% + salt (2.0%) + sugar 2.0%)+ spices (4.0%),
$DNC_{0.2}$ = Dambu-nama + citric acid (0.2%) + Salt (2.0%) + Sugar (2.0%) + Spices (4.0%),
$DNC_{0.3}$ = Dambu-nama + citric acid (0.3%)+ salt (2.0%) + sugar(2.0%) + spices(4.0%)

3.3 The Effect of Hysteresis

The effect of the mode of sorption as shown in Fig. 4 through 22 indicated that treated *dambu-nama* samples at any given a_w had a higher moisture content in the desorption and adsorption modes than the untreated (control) *dambu-nama* samples. The difference in moisture content indicates the presence of hysteresis and agreed with findings of [33] for potatoes and [15] for crayfish. Hysteresis in this product appears to be more marked at higher temperature. The difference in equilibrium moisture content between adsorption and desorption processes of *dambu-nama* followed the general trend i.e. the difference was pronounced in the middle range and less in lower and higher ranges of water activity. The hysteresis curves for *dambu-nama* indicates that at constant equilibrium moisture content, the corresponding water activity is higher for adsorption.

According to [34], hysteresis is the phenomenon by which at a given level of water activity and temperature, an adsorbent holds a smaller amount of moisture during adsorption process than during the corresponding desorption process. Earlier reports [35,36] indicated that the desorption isotherm of the hysteresis loop is always at a higher energy level than the adsorption isotherm. This energy difference is said to arise from physical changes in the adsorbent matrix which expose new energetic sites which absorb moisture on return to lower activity rather than desorb moisture and this is argued to be responsible for the occurrence of hysteresis phenomenon. Similarly, Yan et al. [37]

stated that the extent of hysteresis is related to the nature of the state of the components in food and reflects their potentials for structural and conformational rearrangements which alter accessibility of energetically favourable polar sites. Also, [15] reported that the greater the amount of water held by a product on desorption, the more it would promote chemical reactivity and increase the viability of microorganisms.

Several other explanations [38], in the original wet condition, the polar sites in the molecular structure of the materials are almost entirely satisfied by adsorbed water. Upon drying and shrinkage, the molecules and their water holding sites are drawn closely enough together to satisfy each other. This reduces the water holding capacity of the materials upon subsequent adsorption. The hysteresis exhibited by the *dambu-nama* products can be attributed to the process of irreversibility and those structural and physico-chemical changes that the materials undergo during dehydration. In consonant with earlier reports by [39] for dehydrated products, the principal factors affecting hysteresis in *dambu-nama* include temperature, storage time, drying temperature and the number of successive adsorption and desorption circles. The irreversibility of the adsorption and desorption curves play very significant roles in quality characteristics of the *dambu-nama* products. Conversely, at constant water activity the EMC is higher for desorption which may imply that for storage purposes, the adsorption curve is more appropriate but the reverse is the case for drying.

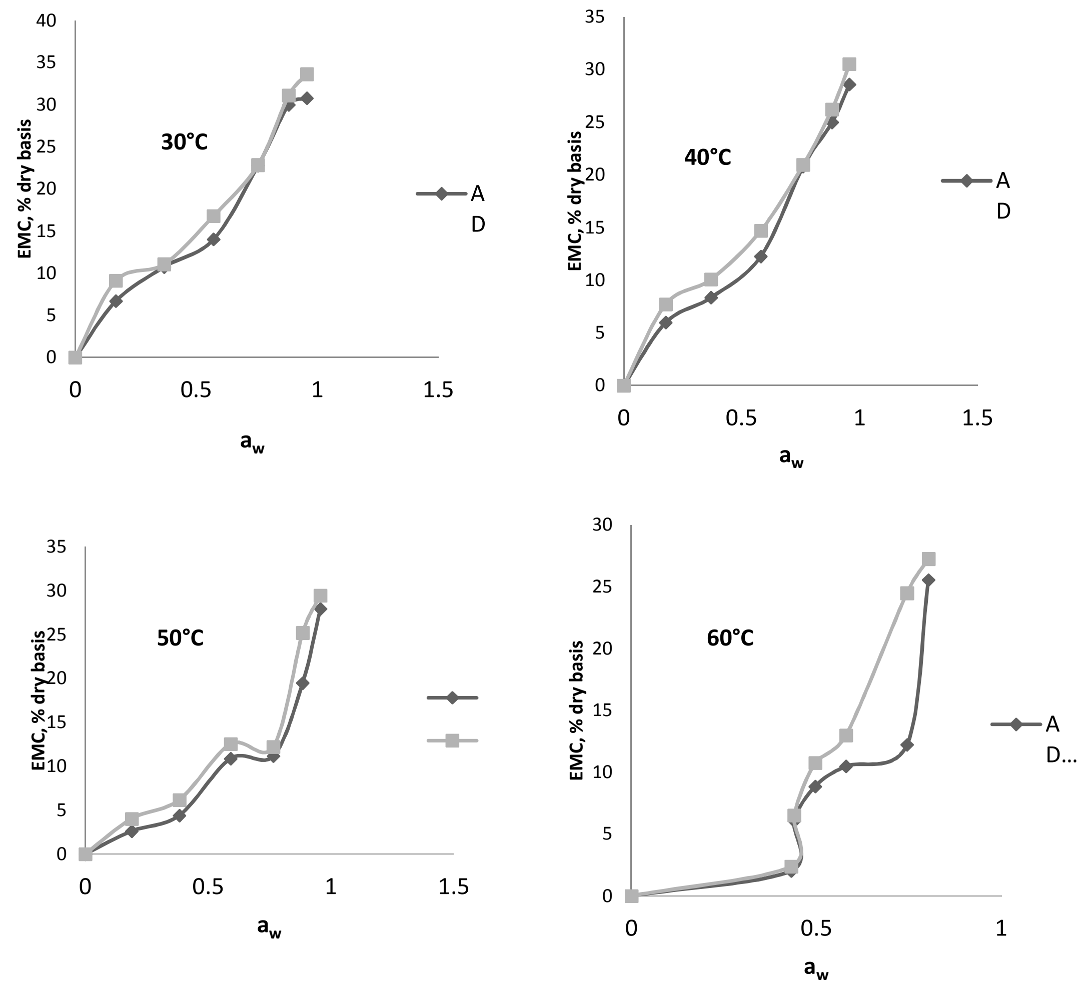


Fig. 4. Moisture sorption hysteresis by DNC_0 at 30-60°C

DNC_0 = Dambu-nama + 4% spices (control)

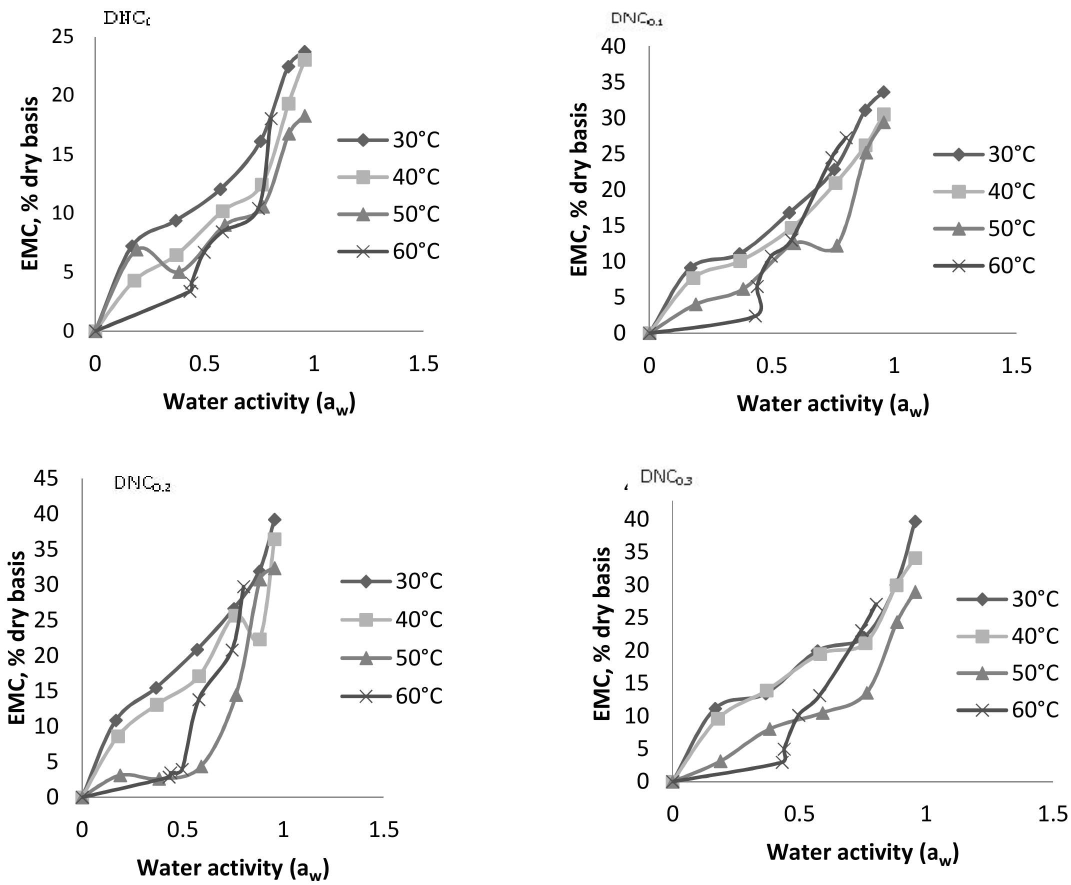


Fig. 5. Moisture Desorption Isotherms of *Dambu-nama* as Influenced by Hurdles

DNC_0 = Dambu-nama + 4% spices (control),
$DNC_{0.1}$ = Dambu-nama+ citric acid 0.1% + salt (2.0%) + sugar 2.0%)+ spices (4.0%),
$DNC_{0.2}$ = Dambu-nama + citric acid (0.2%) + Salt (2.0%) + Sugar (2.0%) + Spices (4.0%),
$DNC_{0.3}$ = Dambu-nama + citric acid (0.3%)+ salt (2.0%) + sugar(2.0%) + spices(4.0%)

3.4 Assessment of Sorption Models

The goodness of fit of the models examined were evaluated using percentage root mean square of error (%RMS). According to [35], %RMS ≤ 10 indicates a reasonable fit for practical purposes. The lower the value, the better the fit.The Henderson's model with overall mean %RMS ≤ 10 best described the moisture sorption isotherms of *dambu-nama* followed by the GAB and Oswin models in that order. The BET plot fitted poorly to the entire isotherms of *dambu-nama why?*. The Henderson model has the additional advantage of incorporating temperature, thereby making the model more versatile for describing the EMCs of *dambu-nama* products.

The BET isotherm is usually valid for a_w ≤ 0.5 [40,15]. However, the BET equation provides values of monolayer moisture content which is an important parameter in food deterioration studies. The low range of a_w application of the BET isotherm is considered a disadvantage when compared with the GAB model which predicts EMC values up to a_w level of 0.90 [19].

The GAB equation is a significant improvement over the BET model and it is based on theoretically sound principles. Its parameters have physical significance and it is considered to be the best equation available for representing multilayer isotherms for many food materials [41,30]. The model provides M_o values and other useful information related to heat of sorption of monolayer and multilayers of food samples.

The Oswin model usually has a high coefficient of regression and has the advantage of its simplicity of use in describing moisture sorption isotherms. It is found to be a good fit for sorption in various food products [42,18] especially for non-proteinous materials and best for starchy related foods.

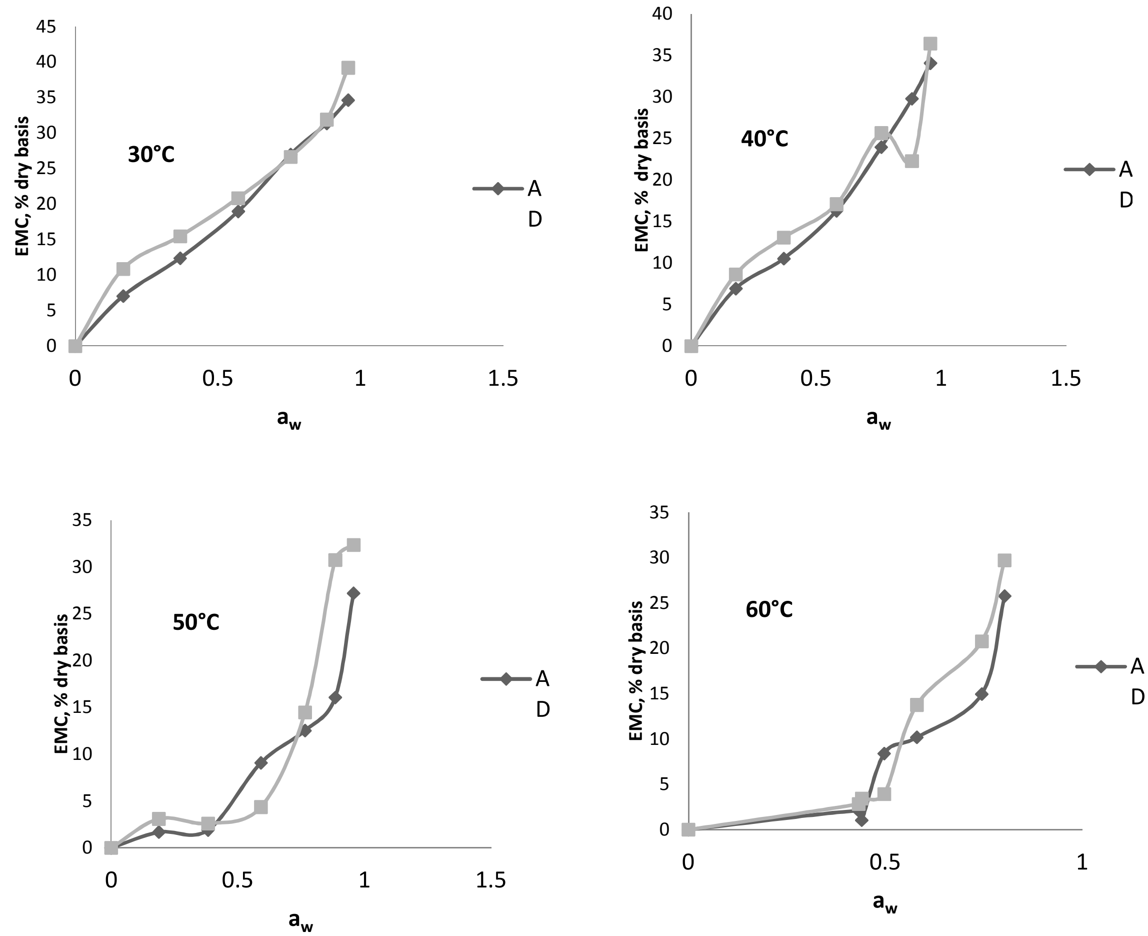
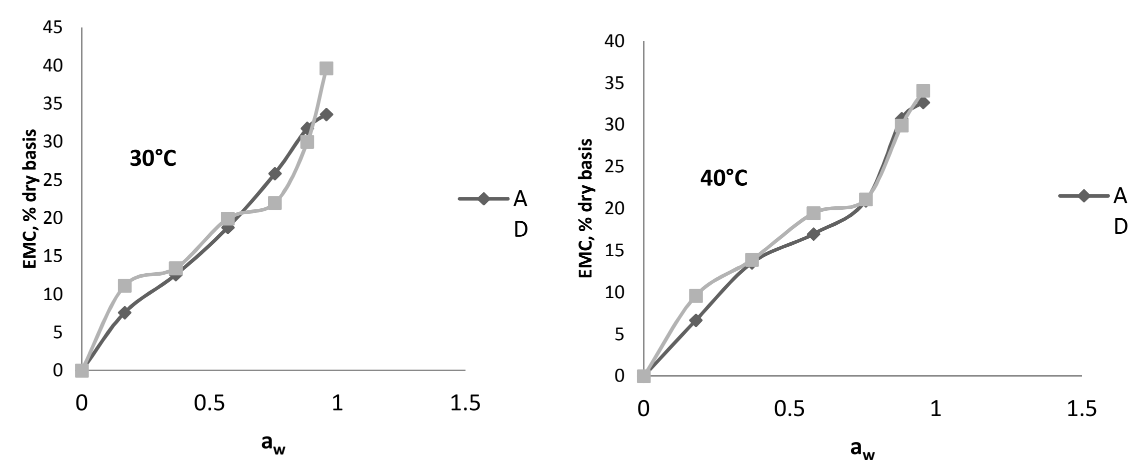


Fig. 6. Moisture sorption isotherms (adsorption and desorption) of $DNC_{0.1}$ at 30-60°C

$DNC_{0.1}$ = Dambu-nama + 0.1% citric acid + 2% salt +2% sugar + 4% spices



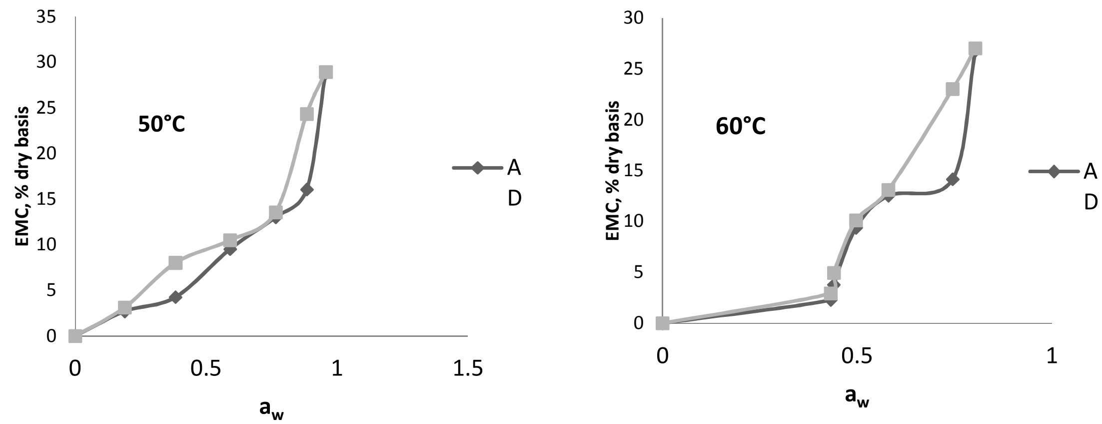


Fig. 7. Moisture sorption isotherms (adsorption and desorption) of DNC$_{0.2}$ at 30-60^{o}C

DNC$_{0.2}$ = Dambu-nama + 0.2% citric acid + 2% salt +2% sugar + 4% spices

4. CONCLUSIONS

The incorporation of citric acid (0.1 to 0.3%), 2% salt and 2% sugar as hurdles into *dambu-nama* resulted in the following findings.Treatment of *dambu-nama* with the hurdles transformed the moisture sorption isotherms of the product from typical sigmoid isotherms to J-shaped isotherms. Generally, the Henderson model gave the best fit isotherm equation for *dambu-nama* followed by the GAB and Oswin models in that order.

The best combination was 0.1% citric acid, 2.0 salt, 2.0 sugar and 4.0 splcies

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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