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Influence of Moisture Sorption Isotherm on Selected Thermodynamic Characteristics of Moringa (Moringa oleifera Lam.) Seeds and Grits

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Abstract

Moringa seeds is a crop that is eaten raw or converted into grits for various uses. Knowledge of sorption isotherms is necessary to correctly carry out drying and storage operations, which will have some effect on the thermodynamic characteristics of the seeds. This study was therefore designed to investigate the influence of the moisture sorption isotherm on some thermodynamic characteristics of moringa seeds and grits. The adsorption and desorption isotherm data of stored moringa seeds and grit were obtained by determining the Equilibrium Moisture Content (EMC) at temperatures ranging from 20℃ to 40℃ and water activity (a_w) levels from 0.09 to 0.92, using thermostatic water bath and concentrated H_2SO_4. The isotherm data were used to estimate sorption's isosteric heat (ΔH) and entropy (ΔS). The approach was based on the Clausius-Clapeyron equation, which has been frequently used in the literature to assess how temperature impacts sorption isotherms. The ΔH decreased as the EMC increased and it was within the ranges of 9.90-40.86 $kJmol^{-1}$ and 4.75-38.71 $kJmol^{-1}$ for seeds and grits, respectively, in adsorption and within the ranges of 10.85-45.09 $kJmol^{-1}$ and 4.82-41.66 $kJmol^{-1}$ for seeds and grits, respectively, in desorption. The ΔS ranged from 0.03 to 0.124 $kJmol^{-1}K^{-1}$ for seeds and 0.014 to 0.113 $kJmol^{-1}K^{-1}$ for grits in adsorption; 0.033-0.137 $kJmol^{-1}K^{-1}$ for seeds and 0.015-0.124 $kJmol^{-1}K^{-1}$ for grits in desorption. The ΔH and ΔS increased exponentially as the EMC decreased. Higher energy was expended to break the forces in desorption than in adsorption. Hence, the reason for the higher net isosteric heat and entropy in desorption than in adsorption. These findings can serve as a guide in predicting and maintaining the shelf life of moringa seeds and grits in storage.

Categories: Food and Pharmaceutical Engineering, Bioprocess Engineering, Chemical Thermodynamics and Separation Processes
Keywords: moringa, sorption isotherm, equilibrium moisture content, isosteric heat, enthalpy

Introduction

Moringa (*Moringa oleifera* Lam.) is a fast-growing, drought-resistant tree of the family, Moringaceae. All the parts of the tree including the root, bark, flower, leaves, seeds, and their extracts have several health benefits. Moringa seeds contain a range of vital phytochemicals; hence, they are highly nutritious containing high levels of protein, oil, vitamins, and minerals, making them a promising functional food and an essential crop for food security in many developing countries [1]. This explains why the moringa tree was named a miracle tree.

In spite of its nutritive value, postharvest stability of moringa seeds deteriorates due to moisture changes during storage. The moisture exerts strong impacts on microbial organism growth, enzymatic activities, lipolysis, and nutrient loss [2]. One highly efficacious method of comprehending and regulating moisture-related alteration through moisture sorption isotherm modeling defines water activity (a_w) and equilibrium moisture content (EMC) in relation to one another at a specified temperature. Moisture sorption isotherms prove excellent assets in devising plans to dry, package, and store as well as forecasting product stability in the long term [3].

Numerous research works have investigated moisture absorption and desorption in seeds and seed products. Alimi et al. [4] investigated *Moringa oleifera* seed absorption and desorption characteristics at two temperatures (25°C and 60°C) over a broad range of water activity, revealing Type II (sigmoidal) characteristics. They also found out that absorption data were best described by Fraction Linear and modified Oswin models. Their results substantiated the fact that EMC decreases as temperature increases at a specific water activity level, an aspect critical in storage under hot climates.

Similar findings have also been reported in other oil-rich seeds. For example, Arslan-Tontul [5] examined chia seeds and reached similar Type II isotherms, and in these, isosteric heat (ΔH) was at its maximum at

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lower EMC and reflected strong moisture interactions between water and dry matrix. Martinez-Monteagudo and Salais-Fierro [6] used Clausius-Clapeyron modeling to study cheese and showed that enthalpy (ΔH) and entropy (ΔS) of sorption decline as EMC increases and it becomes easier to desorb water as moisture wetness increases in matrix.

This research also extends these findings by characterizing moisture sorption isotherms for moringa seeds and grits in an extended temperature range (20-40°C) and predicting some characteristic thermodynamic parameters such as ΔH, ΔS, and Gibbs free energy (ΔG). These findings can be used to develop optimized moringa drying and storage systems to retain their functional and nutritional characteristics.

Materials And Methods

Experimental design

The current study was an experimental laboratory work undertaken with the objective of determining the isotherms of adsorption and desorption of moringa seeds and grits as well as estimating the thermodynamic parameters, specifically net isosteric heat (ΔH) and entropy of sorption (ΔS) using the Clausius-Clapeyron equation. A completely randomized design (CRD) with three replications was adopted for the experiments. The independent variables were the levels of temperature (20, 25, 30, 35, and 40°C) and water activity (aw; 0.09-0.92). The dependent variables consisted of EMC and ΔH and ΔS for both adsorption and desorption.

Materials

Recent moringa seeds bought from a certified agricultural center in Oyo State, Nigeria, were thoroughly manually cleaned in an attempt to remove extraneous matters and broken seeds. Grits were made through dehulling and coarse grinding of the seeds using a laboratory burr mill (Model: Retsch ZM 200, Germany) fitted with a 1 mm sieve. All samples were conditioned before embarking on the work so that an initial moisture content of approximately 10% (wet basis) was achieved. Moisture content was measured using the conventional oven drying technique at 105°C for 24 hours [7].

Experimental set up

Moisture sorption isotherms were determined with the static gravimetric method outlined by [8]. To find out water activities ranging between 0.09 and 0.92, nine H_2SO_4 saturated solutions were pre-created as shown in Table *1*. Pre-weighed Petri dishes with 30 grams of either grit samples or seed samples were then put inside glass desiccators with the acid solutions of 350 mL. Each of the desiccators was then put inside a thermostatically controlled water bath (Julabo SW23, Germany) at the pre-set temperature.

Each sample was weighed every 6 h with an analytical balance (±0.001 g) until three consecutive readings are constant, indicating equilibrium [9]. The EMC was calculated on a dry weight basis.

H_2SO_4 (%)	Temperature (°C)				
	20	25	30	35	40
15	0.9237	0.9241	0.9245	0.9253	0.9261
25	0.8218	0.8218	0.8252	0.8285	0.8317
35	0.6607	0.6651	0.6693	0.6773	0.6846
40	0.5599	0.5656	0.5711	0.5816	0.5914
45	0.4524	0.4589	0.4653	0.4775	0.4891
50	0.3442	0.3509	0.3574	0.3702	0.3827
55	0.2440	0.2505	0.2563	0.2685	0.2807
60	0.1573	0.1625	0.1677	0.1781	0.1887
65	0.0895	0.0933	0.0972	0.1052	0.1135

TABLE 1: Concentrated H2SO4 and corresponding water activity values

Determination of isosteric heat and entropy of sorption

The sorption isotherm data were used to estimate the isosteric heat of sorption. The approach was based on the Clausius-Clapeyron equation, which has been frequently used to assess the effect of temperature on sorption isotherms. The technique has been reported to work for a large range of temperature levels and is characterized by its simplicity and reliability [10]. Clausius-Clapeyron equation is as given in Equation (1) [11].

$$d(lna_w)/d(1/T) = -(q_st/R) \quad (1)$$

where:

a_w is the water activity (decimal)

q_{st} is the net isosteric heat of sorption (kJ mol^{-1})

R is the universal gas constant (kJ mol^{-1} K^{-1})

T is absolute temperature (K).

The net isosteric heat of sorption was calculated by plotting the natural log of the water activity (ln a_w) against the inverse of the absolute temperature (1/T) at five different EMCs by determining the slope (-q_{st}/R). This procedure was based on the fact that the net isosteric heat of sorption remain unchanged at different temperatures. Differential entropy of sorption (ΔS, kJ mol^{-1} K^{-1}) was calculated from Gibbs-Helmholtz equation given in Equation (2).

$$\Delta S = ((q_st - \Delta G)/T) \quad (2)$$

while, (ΔG, kJ mol^{-1}) which is Gibb's free energy was calculated using the Van't Hoff expression given in Equation (3) [12].

$$\Delta G = -RTlna_w \quad (3)$$

Equations (2) and (3) were combined and rearranged to give Equation (4) [13].

$$lna_w = (-q_st/RT + \Delta S/R) \quad (4)$$

Entropy of sorption (ΔS) was determined from the plot of (ln a_w) against (1/T) at a particular EMC as the intercept value of (ΔS/R).

Data analysis

Data analysis was performed with Microsoft Excel 365 using the linear regression functions for the Clausius-Clapeyron graphs.

Results And Discussion

The influence of moisture sorption isotherm at five temperatures and nine water activity levels on some thermodynamic characteristics, namely the Isosteric heat and entropy of sorption, of moringa seeds and grits determined in this study is shown in Table *2* and Table *3*, respectively.

EMC (% db)	Isosteric heat of sorption (kJ mol^{-1})		Entropy of sorption (kJ mol^{-1} K^{-1})	
	Adsorption	Desorption	Adsorption	Desorption
3	40.86	45.09	0.124	0.137
4	24.75	25.10	0.074	0.075
5	16.41	17.23	0.049	0.051
6	10.85	10.97	0.032	0.032
7	9.90	10.85	0.030	0.033

TABLE 2: Isosteric heat and entropy of sorption of moringa seed

EMC, Equilibrium Moisture Content

Table 2 displays the isosteric heat of sorption and entropy of sorption of moringa seeds at different EMC. This behavior is in line with how hygroscopic materials generally behave, which is that as EMC increases, the isosteric heat of sorption decreases. At an EMC of 3% (dry basis), the maximal isosteric heat values of adsorption (40.86 kJ mol^{-1}) and desorption (45.09 kJ mol^{-1}) were calculated. This indicates that at lower moisture levels, when more energy is required for moisture removal, stronger water binding takes place.

When it comes to entropy of sorption, the tendencies are very similar, because the values decrease as EMC increases. The entropy is 0.124 kJ mol^{-1} K^{-1} in adsorption at 3% EMC and 0.137 kJ mol^{-1} K^{-1} in desorption at the same humidity, indicating an increased randomness at lower moisture content. These results align with previous studies regarding seed sorption properties, where increased moisture levels are directly related to stronger water-binding forces, since at higher moisture content, active sorption sites may not be available [14,15].

EMC (% db)	Isosteric heat of sorption (kJ mol^{-1})		Entropy of sorption (kJ mol^{-1} K^{-1})	
	Adsorption	Desorption	Adsorption	Desorption
3	38.71	41.66	0.113	0.124
4	29.44	32.07	0.088	0.094
5	15.95	17.74	0.047	0.053
6	12.60	12.84	0.038	0.039
7	4.75	4.82	0.014	0.015

TABLE 3: Isosteric heat and entropy of sorption of moringa seed grits

EMC, Equilibrium Moisture Content

The isosteric heat and entropy of sorption for moringa seed grits are shown in Table 3. The overall pattern, where isosteric heat and entropy decrease as EMC rises, is comparable to that of whole moringa seeds. At the same EMC levels, the isosteric heat values for moringa seed grits are somewhat lower than those for whole seeds. For example, at 3% EMC, the adsorption isosteric heat for grits is 38.71 kJ mol^{-1}, while for whole seeds it is 40.86 kJ mol^{-1}. Processing-induced structural changes that can reveal additional sorption sites or change the binding energy distribution could be the cause of this reduction [16].

Recent research on seeds and products obtained from seeds is consistent with the characteristics that have been observed. Peanut kernels, for instance, show the highest sorption enthalpy at low EMC, which is followed by a noticeable decrease when the moisture content rises [14]. By altering the content and surface properties of seed meals, processing and fractionation can change the sorption behavior [16]. Furthermore, in milled plant powders, sorption kinetics and equilibrium are influenced by particle size and resulting surface area; finer fractions typically exhibit higher hygroscopicity and a quicker approach to equilibrium [17]. These comparisons validate our hypothesis that the thermodynamic energy needed for water uptake and release in moringa products is modulated by both composition (seed vs. grits) and physical form (whole vs. milled).

The regression models to predict the net isosteric heat of sorption (q_{st}) and entropy of sorption (ΔS) of moringa seed and grits at a given EMC are presented in Table 4 and Table 5, respectively.

	Net isosteric heat of sorption (kJ mol^{-1})	Entropy of sorption (kJ mol^{-1} K^{-1})
Adsorption	q_{st} = -0.2633 EMC ^{3} + 6.3143EMC^2 - 50.08 EMC + 141.28	ΔS = -0.0008 EMC ^{3} + 0.0199 EMC ^{2} - 0.157 EMC + 0.4377
	R^2 = 0.9990	R^2 = 0.9986
Desorption	q_{st} = -0.4983 EMC ^{3} + 13.419 EMC ^{2} - 121.17 EMC + 377.56	ΔS = -0.0015 EMC ^{3} + 0.0409 EMC ^{2} - 0.3715 EMC + 1.1599
	R^2 = 0.9960	R^2 = 0.9957

TABLE 4: Predictive regression models for net isosteric heat of sorption and entropy of sorption of moringa seed

EMC, Equilibrium Moisture Content

	Net isosteric heat of sorption (q_{st}) (KJ/mol)	Entropy of sorption (ΔS) (KJ/mol K)
Adsorption	q_{st} = -0.0233 EMC ^{3} + 1.2771 EMC ^{2} - 19.418 EMC + 86.514	ΔS = 8E-05 EMC ^{3} + 0.0012 EMC ^{2} - 0.0431 EMC + 0.2309
	R^2 = 0.9838	R^2 = 0.9797
Desorption	q_{st} = 0.135 EMC ^{3} - 1.1271 EMC ^{2} - 8.6036 EMC + 74.352	ΔS = 8E-05 EMC ^{3} + 0.0015 EMC ^{2} - 0.0492 EMC + 0.2566
	R^2 = 0.9885	R^2 = 0.9895

TABLE 5: Predictive regression models for net isosteric heat of sorption and entropy of sorption of moringa seed grits

EMC, Equilibrium Moisture Content

Practical implication

The results obtained in this work can be considered useful for the enhancement of postharvest handling and storage of *Moringa oleifera* seeds and their products. The results can be employed as useful data for designing drying, storage, and packaging systems for moringa seeds. Knowledge of the influence of EMC on the isosteric heat and entropy of sorption can be used to determine the optimum drying temperature that requires less energy. The results also showed that milled products like the grits of moringa seed have low sorption energy. This means that milled or processed forms of products may require tighter control of moisture to prevent degradation of their quality during storage. In addition to this, the results can be employed to model the behavior of moisture in given environments and can help in designing storage materials with the optimum moisture-barrier resistance. Overall, the results obtained herein can contribute to the production of sustainable processing and storage technology for *Moringa oleifera* to thrive in hot climates like Nigeria.

Conclusions

The current study explored *Moringa oleifera* seed and grit sorption isotherms at five temperatures (20-40°C) and nine water activities (0.09-0.92) and examined their impact thereon on key thermodynamic properties.

Quantitatively, net isosteric heat of sorption (ΔH) of moringa seeds changed from 40.86 to 9.90 kJ mol^{-1} during adsorption and 45.09 to 10.85 kJ mol^{-1} during desorption with increasing EMC by 3 to 7% (dry basis). For grits, ΔH was relatively lower 38.71 to 4.75 kJ mol^{-1} during adsorption and 41.66 to 4.82 kJ mol^{-1} for desorption process.

Also, the entropy of sorption (ΔS) reduced with enhanced EMC. For seeds, ΔS varied from 0.124 to 0.030 kJ mol^{-1} K^{-1} during adsorption and 0.137 to 0.033 kJ mol^{-1} K^{-1} during desorption, whereas grits showed somewhat lesser values (0.113-0.014 kJ mol^{-1} K^{-1} during adsorption and 0.124-0.015 kJ mol^{-1} K^{-1} during desorption). These findings show that water molecules are generally more ordered at enhanced EMC and less energy is needed for sorption.

Specifically, desorption always showed a higher ΔH and ΔS than adsorption, confirming hysteresis and suggesting it takes more energy to desorb water from biological materials. Regression models built for all products yielded excellent fits ($R^2 \geqslant 0.98$), permitting reliable prediction of thermodynamic parameters based upon EMC.

The present work points out that moringa grits release and absorb moisture at a lesser rate compared to intact seeds, and hence has storage and process implications. The work can be adopted when it comes to designing efficient drying protocols, specifying packaging materials with suitable moisture barrier functionalities, and estimating shelf life during various storage situations, determining factors when it comes to preserving the nutritional and functional integrity of moringa.

Additional Information

Author Contributions

All authors have reviewed the final version to be published and agreed to be accountable for all aspects of the work.

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Critical review of the manuscript for important intellectual content: Akintunde Akintola, Babatunde O. Oyefeso, Rahman Akinoso

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Disclosures

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