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DESORPTION ISOTHERMS OF GRAIN SORGHUM (*Sorghum bicolor* [L.] Moench) FLOUR

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KEYWORDS

water activity, cereal,
mathematical models.

ABSTRACT

This study aimed to obtain the sorption isotherms of grain sorghum flour, fit mathematical models to experimental data, and recommend safe water levels for preserving the material at different temperatures. Sorghum grains with an initial moisture content of 34% dry basis (db) were subjected to drying at a temperature of 60 °C, reaching moisture contents ranging from 32.20 to 9.8% db. Water activity was obtained using Hygropalm Aw1 equipment placed inside a BOD chamber at temperatures of 10, 20, 30, and 40 °C. The experimental data were fitted to mathematical models frequently used to predict the isotherms of plant products. The modified Oswin model presented the best fit to estimate the sorption isotherms of sorghum flour. Safe moisture content limits for storing sorghum flour are 18.45, 17.3, 16.08, and 15.2% db for temperatures of 10, 20, 30, and 40 °C, respectively. The isosteric heat for the range of equilibrium moisture contents from 9.80 to 32.20% db varied from 2779.49 to 2498.46 kJ kg⁻¹.

INTRODUCTION

Sorghum (*Sorghum bicolor* (L.) Moench) is native to Africa and ranks 5th among the world's most cultivated cereals, trailing corn, rice, wheat, and barley (FAO, 2020). Brazil is seventh in the ranking of annual sorghum production (Conab, 2023). Sorghum is an excellent cereal due to its nutritional quality and can be included in human nutrition in Brazil, with great importance for people sensitive to gluten, as it is a gluten-free cereal low in lipids and rich in protein, fiber, vitamin B, and minerals (Food Data Central (2019). The amount of nutrients and phytochemicals in sorghum products can vary depending on the type of grain and processing (Indriarningsih et al., 2023).

Sorghum can be included in the diet in the form of flour and is used in the manufacture of cakes, breakfast cereals, breads, tortillas, biscuits, and pasta (Queiroz et al., 2017). The introduction of sorghum flour into human nutrition is still recent in Brazil, as a large part of sorghum production is used as a cover crop and animal feed. However, there is no specific legislation for sorghum flour in Brazil, which makes it necessary to study the stability of sorghum flour to maintain its quality throughout storage time.

Changes in color, flavor, texture, and nutritional and functional quality may occur during shelf life if the flour is not in adequate storage conditions such as packaging, temperature, moisture content, and lack of controlled conditions (Gusmão et al., 2018). In this sense, sorption isotherms allow the determination of the maximum water sorption capacity, improving food preparation processes, increasing storage time, and maintaining nutritional quality (Aksil et al. 2019).

The sorption isotherm describes the amount of water sorbed (adsorption or desorption) at equilibrium by a material with known water activity (Aw), constant temperature, and constant pressure (Galdeano et al., 2018).

Hygroscopicity allows for predicting the stability of dehydrated foods during storage and makes it possible to analyze the material's capacity to absorb or lose water to the environment, which is related to its physical, chemical, and microbiological stability throughout storage (Araújo et al., 2020).

Knowledge of water sorption characteristics is related to optimal storage regime conditions and can contribute to the preservation of nutritional qualities and technological properties (Berk, 2018). Knowing the

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sorption isotherms at different temperatures and the most appropriate mathematical equations enables the calculation of thermodynamic functions such as the isosteric heat of sorption (Li et al., 2021).

Thus, this study aimed to obtain the sorption isotherms of grain sorghum (*Sorghum bicolor* (L) Moench) flour, fit mathematical models to experimental data, and recommend safe moisture contents for the preservation of sorghum flour at different temperatures and obtain the isosteric heat of desorption.

MATERIAL AND METHODS

The experiment was carried out in the Laboratory of Postharvest of Plant Products of the Federal Institute of Education, Science and Technology Goiano, located in the municipality of Rio Verde, GO, Brazil. The sorghum grains were harvested manually in the municipality of Rio Verde, GO, at the geographic location 17°44'20.88" S and 50°57'55.79" W. The grains were threshed manually and impurities were removed using 2.2-mm opening circular sieves and 3 x 22-mm oblong opening sieves and, subsequently, homogenization was conducted using Boerner equipment.

Sorghum grains of the Jade genotype were dried in a forced-air ventilation oven at a temperature of 60 °C using the gravimetric method to obtain the five moisture contents. Knowing the initial moisture content of the grains allowed obtaining different moisture contents. After drying, the sorghum grains were crushed in an industrial blender and then subjected to the grinding process in a Fortinox STAR FT-80/1 electric knife mill, obtaining flour moisture contents ranging from 32.1 to 9.80% db. The flours were packaged in polyethylene plastic packaging and stored in a BOD chamber until analysis was conducted.

Flour sorption isotherms were determined using the static-indirect method, with the water activity (A_w) determined using the Hygropalm Model aW1 equipment. Triplicate samples were used for each moisture content, with approximately 35 g of flour inserted inside the equipment. The device was placed in a BOD chamber regulated at temperatures of 10, 20, 30, and 40 °C. The water activity in the equipment was obtained after stabilizing the temperature, and the moisture content of the samples in the oven was subsequently determined following the methodology by Brasil (2009). Mathematical models frequently used to represent the hygroscopicity of plant products were tested using the experimental data. The expressions are shown in Table 1.

TABLE 1. Mathematical models used to predict the sorption phenomenon of grain sorghum flour (*Sorghum bicolor* (L) Moench).

Model designation	Model	
$X_e = a - b \cdot \ln[-(T+c) \cdot \ln(a_w)]$	Chung-Pfost	(1)
$X_e = [-1/(c \cdot T^d)] \cdot \ln[\ln(a_w)/(-a \cdot T^b)]$	Chen Clayton	(2)
$X_e = \exp[a - (bT) + (c \cdot a_w)]$	Copace	(3)
$X_e = \exp[a - (b \cdot T) / (c - \ln(a_w))]$	Harkins	(4)
$X_e = [\ln(1 - a_w) / -a(T+b)]^{1/c}$	Modified Henderson	(5)
$X_e = (a + b \cdot T) / [(1 - a_w) / a_w]^{1/c}$	Modified Oswin	(6)
$X_e = a \cdot [a_w \cdot (b/T^c)]$	Sabbah	(7)
$X_e = \exp\{a - (b \cdot T) + [c \cdot \exp(a_w)]\}$	Sigma Copace	(8)
$X_e = [\ln(1 - a_w) / (a \cdot (T^b))]^{1/c}$	Cavalcanti Mata	(9)

X_e – equilibrium moisture content, % db; a_w – water activity, decimal; T – temperature, °C; a, b, c – coefficients dependent on the product.

The mathematical models were fitted using non-linear regression analysis by the Gauss-Newton method. The best model was selected considering the significance of the model coefficients using the t-test, the magnitude of the coefficient of determination (R^2), the estimated mean error (SE), the values of the mean relative error (P), the chi-square test (χ^2) at a significance level of 0.01, and the confidence interval at 0.99 ($p < 0.01$). The estimated mean and relative error and the chi-square test for each of the models were calculated according to eqs (10), (11), and (12), respectively.

$$SE = \sqrt{\frac{\sum (Y - \hat{Y})^2}{RDF}} \quad (10)$$

$$P = \frac{100}{N} \sum \frac{|Y - \hat{Y}|}{Y} \quad (11)$$

$$\chi^2 = \frac{\sum_{i=1}^n (y - \hat{y})^2}{RDF} \quad (12)$$

Where:

Y is the experimental value;

\hat{Y} is the value estimated by the model;

N is the number of experimental observations, and

RDF is the residual degree of freedom of the model (number of observations minus the number of model parameters).

Models with the best fitting were subjected to the Akaike information criterion (AIC) and Bayesian-Schwarz information criterion (BIC) to select a single model and describe the isotherm process in each condition, according to the following expressions:

$$AIC = -2\log\text{like} + 2p \quad (13)$$

$$BIC = -2\log\text{like} + p\log(n) \quad (14)$$

In which:

p is the number of parameters, and

$\log\text{like}$ is the value of the logarithm of the likelihood function considering the parameter estimates.

Integral isosteric heat

The values of the net isosteric heat of sorption (or differential enthalpy) for each equilibrium moisture content were obtained using the Clausius-Clapeyron equation (Iglesias & Chirife, 1976), as shown below:

$$\frac{\ln a_\omega}{T} = \frac{h_{st}}{RTa^2} \quad (15)$$

Where:

a_ω is the water activity;

Ta is the absolute temperature (K);

Δh_{st} is the net isosteric heat of sorption (kJ kg^{-1}), and

R is the universal gas constant ($8.314 \text{ kJ kmol}^{-1} \text{ K}^{-1}$) with water vapor equal to $0.4619 \text{ kJ kg}^{-1} \text{ K}^{-1}$.

The net isosteric heat of sorption is obtained for each equilibrium moisture content by integrating [eq. (15)] and assuming that the net isosteric heat of sorption is independent of temperature, according to [eq. (16)] (Wang & Brennan, 1991).

$$\ln(a_\omega) = \left(\frac{\Delta h_{st}}{R}\right) \frac{1}{Ta} + c \quad (16)$$

In which:

a_ω is the water activity;

Ta is the absolute temperature (K);

Δh_{st} is the net isosteric heat of sorption (kJ kg^{-1});

R is the universal gas constant ($8.314 \text{ kJ kmol}^{-1} \text{ K}^{-1}$) with water vapor equal to $0.4619 \text{ kJ kg}^{-1} \text{ K}^{-1}$, and

C is the model coefficient.

The values of water activity, temperature, and equilibrium moisture content were obtained from the sorghum flour sorption isotherms, using the model that best fitted the experimental data. The integral isosteric heat of sorption was obtained by adding the value of the latent heat of vaporization of free water to the values of net isosteric heat of sorption, according to [eq. (17)].

$$Q_{st} = \Delta h_{st} + L = a \cdot \exp(-b \cdot X_e) + L \quad (17)$$

in which:

Q_{st} is the integral isosteric heat of sorption (kJ kg^{-1});

a, b, and c are the model coefficients, and

L is the latent heat of vaporization of free water (kJ kg^{-1}).

RESULTS AND DISCUSSION

Table 2 shows the coefficients of the sorption isotherm models fitted to the grain sorghum flour data, as well as the statistical parameters. Only the modified Oswin model out of the nine tested models presented all significant coefficients according to the t-test. The other models presented at least one of the coefficients that were not significant by the t-test, indicating a limitation in the coefficient estimate, which may promote an error in fitting the model to the experimental data.

TABLE 2. Coefficients of the models fitted to the experimental data of grain sorghum flour, coefficient of determination (R^2), estimated mean error (SE), chi-square (χ^2), relative mean error (P, %), and criteria of selection of Akaike information (AIC) and Bayesian-Schwarz information (BIC).

Sorghum flour							
Model	Coefficient	R^2	SE	χ^2	P (%)	AIC	BIC
Chung-Pfost	a=53.6438*** b=9.7605*** c=73.3307 ^{ns}	94.04	2.47	6.09	12.55	97.65	101.63
Chen Clayton	a=5.7917 ^{ns} b=-0.2780 ^{ns} c=0.1153* d=-0.0418	93.86	2.58	6.67	12.57	100.25	105.22
Copace	a=1.3761*** b= 0.00446 ^{ns} c= 2.3922***	93.54	2.57	6.605	11.58	99.26	103.25
Harkins	a=2.4464*** b=0.0051· c=0.2165***	95.51	9.02	81.33	47.99	92.00	95.98
Modified Henderson	a=0.0002 ^{ns} b=137.2000 ^{ns} c=1.2510***	94.48	22.99	528.82	97.07	96.11	100.09
Modified Oswin	a=13.5296*** b=-0.0732** c=2.1724***	97.43	1.62	2.63	7.21	80.85	84.84
Sabbah	a=35.0419*** b=0.7909* c= -0.2091 ^{ns}	88.85	3.38	11.40	17.14	110.19	114.17
Sigma Copace	a=0.5097* b= 0.0047· c=1.2397***	95.75	2.08	4.34	8.48	90.89	94.87
CavalcanteMata	a=-0.2264* b=0.1134 ^{ns} c=1.2396***	92.15	19.77	390.80	84.75	69.69	72.25

*** Significant at 0.001 by the t-test; ** Significant at 0.01 by the t-test; · Significant at 0.1 by the t-test; * Significant at 0.05 by the t-test; ns: Not significant by the t-test.

Several parameters must be analyzed to choose the best mathematical model to describe the hygroscopicity phenomenon of sorghum flour. The coefficient of determination (R^2) must present a value higher than 90%, estimated mean error (SE) and chi-square (χ^2) must present the lowest values, and the mean relative error (P) must be lower than 10% (Siqueira et al., 2016; Hawa et al., 2020).

Only the Sabbah model did not present a coefficient of determination above 90% among the nine analyzed models. According to Madamba et al. (1996), values above 90% express a satisfactory representation of the process under study. However, the coefficient of determination cannot be analyzed in isolation to select non-linear models, as it is not a good representation parameter (Draper & Smith, 1998). The modified Oswin and Sigma Copace models presented the lowest values for the estimated mean error (SE), that is, 1.622 and 2.0845, respectively. According to Draper & Smith (1998), lower values for this parameter indicate a better fit of the model to the experimental data.

The modified Oswin and Sigma Copace models had the lowest chi-square values (2.6314 and 4.3450). The lower the chi-square value (χ^2) in a confidence interval, the

better the model fits in representing the studied phenomenon. The fitting of the models to the experimental data showed a magnitude higher than 92% for the coefficient of determination (R^2), except for the Sabbah mathematical model.

The mean relative error (P) values showed that only the modified Oswin and Sigma Copace presented magnitudes lower than 10% for the studied models. According to Mohapatra & Rao (2005), only models with a P value below 10% are suitable for describing the phenomenon. The modified Oswin and Sigma Copace models stood out for the parameters of mean relative error (P), mean estimated error (SE), and coefficient of determination (R). Therefore, AIC and BIC were tested to verify which mathematical model best describes the sorption isotherms of grain sorghum flour.

According to Akaike (1974) and Schwarz (1978), the lower the AIC and BIC value, the better the model will describe the phenomenon. Therefore, the modified Oswin mathematical model presented the lowest values among the two models analyzed using AIC and BIC (Table 2), being recommended for representing the sorption isotherms of

grain sorghum flour. These criteria have been used satisfactorily by different authors as an auxiliary criterion in choosing mathematical models to estimate the studied processes (Ferreira Junior et al., 2018; Gomes et al., 2018; Quequeto et al., 2019). Considering all the evaluated statistical parameters relative to the fitting of the

mathematical models to the experimental data, the modified Oswin model was used to represent the sorption isotherms of grain sorghum (*Sorghum bicolor* (L.) Moench) flour (Figure 1). The modified Oswin model was also used to represent the sorption isotherms of Bulgarian whole grape flour (Bogoeva & Durakova, 2020).

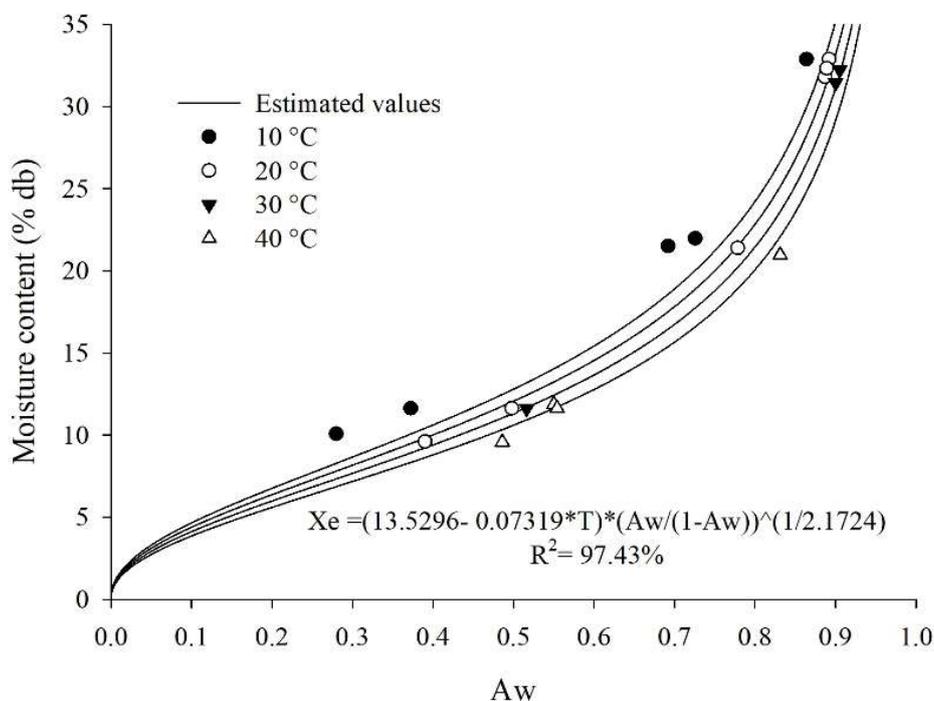


FIGURE 1. Sorption isotherms of grain sorghum (*Sorghum bicolor* (L.) Moench) flour estimated by the modified Oswin model.

The isotherms had a characteristic type II sigmoidal shape, according to the IUPAC classification (1985), which is typical of foods with a high starch content (Ribeiro et al., 2021). In this sense, Arslan-Tontul (2020) carried out the sorption isotherms of chia seeds, Rosa et al. (2021) determined the water sorption properties of papaya seeds, and Bogoeva & Durakova (2020) carried out the sorption isotherms of grape seed flour and found isotherms with similar characteristics (Type II isotherm model).

A water activity of 0.7 led to equilibrium moisture contents ranging from 18.45 to 15.20% db for the temperature range 10 to 40 °C. The equilibrium moisture content will be higher when the temperature decreases for the same water activity. Thus, the maximum equilibrium moisture contents for the safe storage of sorghum flour with a water activity of 0.7 at temperatures of 10, 20, 30, and 40 °C are 18.45, 17.3, 16.08, and 15.20% db or 15.58, 14.74, 13.85, and 13.20% db, respectively. Pumacahua-Ramos et al. (2017) found similar results when studying the sorption isotherms of quinoa flour at temperatures of 10, 20, 30, and

40 °C for safe storage with a water activity of 0.66 with values ranging from 20.13 to 14.10 % db.

The results show that the increase in water activity represents an increase in the moisture content of grain sorghum (*Sorghum bicolor* (L.) Moench) flour, which is commonly found for sorption isotherms of several plant products reported in the literature, such as yellow mombin pulp (Cavalcante et al., 2018).

Isotherms are important for the safe storage of sorghum flour. The safe limit of water activity so that stored products do not become susceptible to attack by microorganisms is around 0.7 (decimal) (Oliveira et al., 2005). Li et al. (2021) studied the equilibrium moisture contents of sorption and isosteric heat of Chinese wheat bran and recommended a moisture content of up to 14.21% db for safe storage, with water activity up to 0.7 Aw at 25 °C. Figure 2 shows the values of the integral isosteric heat of sorption (Q_{st}), in kJ kg^{-1} , as a function of the equilibrium moisture content (% db).

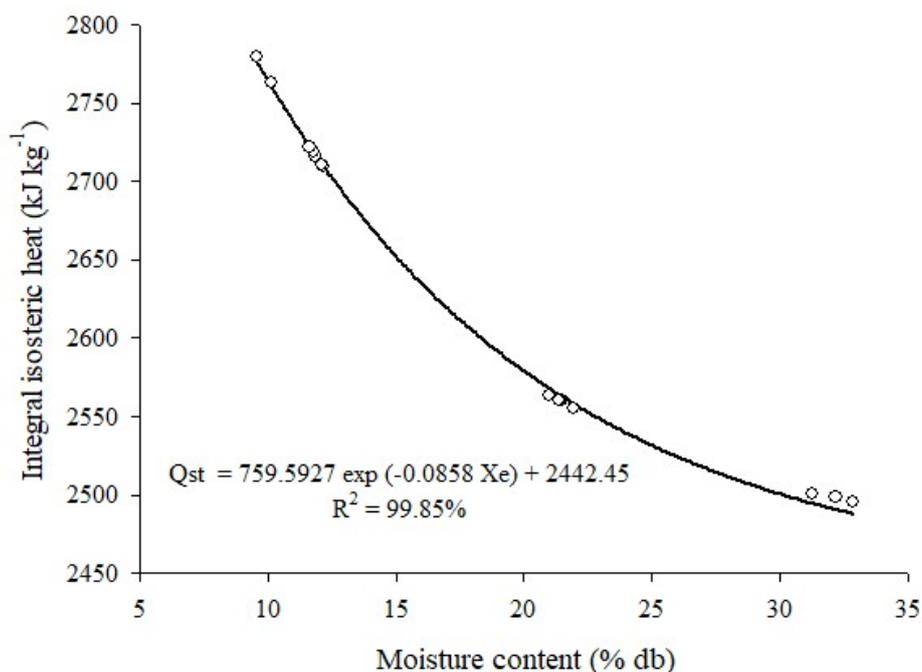


FIGURE 2. Observed and estimated values of the integral isosteric heat of sorption of grain sorghum (*Sorghum bicolor* (L.) Moench) flour as a function of equilibrium moisture content.

The reduction in moisture content led to an increase in the energy required to remove water from the product, represented by the values of the integral isosteric heat of sorption (Q_{st}).

The integral isosteric heat of sorption values for sorghum flour in the equilibrium moisture content range of 9.56 to 32.20% db varied from 2779.49 to 2498.46 kJ kg⁻¹. Li et al. (2021) observed a similar behavior and reported that the lower the moisture content, the higher the energy required to remove water molecules. Li et al. (2021) found isosteric heat values ranging from 3300 to 2400 kJ kg⁻¹ in the range of moisture contents from 5 to 28% db.

Isosteric heat of sorption values are related to physicochemical and processing factors, which influence the binding of water molecules, including the three-dimensional structure of food macromolecules and chemical compositions (Tadapaneni et al., 2018). Thus, the magnitude of the isosteric heat value varies for different food products, as observed in the present study. The used equation was satisfactory in describing the phenomenon, presenting a high significance of its parameters and coefficient of determination ($R^2 = 99.85\%$).

CONCLUSIONS

Sorghum flour can be stored safely against the development of fungi, yeasts, and bacteria as long as it has maximum equilibrium moisture contents of 18.45, 17.3, 16.08, and 15.2% db, respectively, for temperatures of 10, 20, 30, and 40 °C. The Oswin modified mathematical model fitted satisfactorily to the experimental data, and the integral isosteric heat of sorption values varied from 2779.49 to 2498.46 kJ kg⁻¹ in the range of equilibrium moisture contents from 9.56 to 32.20% db.

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