



# Water adsorption isotherms and thermodynamic properties of cassava bagasse



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## ABSTRACT

Losses of food industry are generally wet products that must be dried to posterior use and storage. In order to optimize drying processes, the study of isotherms and thermodynamic properties become essential to understand the water sorption mechanisms of cassava bagasse. For this, cassava bagasse was chemically analyzed and had its adsorption isotherms determined in the range of 293.15–353.15 K through the static gravimetric method. The models of GAB, Halsey, Henderson, Oswin and Peleg were fitted, and best adjustments were found for GAB model with  $R^2 > 0.998$  and no pattern distribution of residual plots. Isotheric heat of adsorption and thermodynamic parameters could be determined as a function of moisture content. Compensation theory was confirmed, with linear relationship between enthalpy and entropy and higher values of isokinetic temperature ( $T_B = 395.62$  K) than harmonic temperature. Water adsorption was considered driven by enthalpy, clarifying the mechanisms of water vapor sorption in cassava bagasse.

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## 1. Introduction

Worldwide, several studies have been developed to encourage more efficient applications of byproducts and/or residues from agroindustry. They intend to satisfy environmental and economic concerns by exploring valuable properties in food wastes. The big availability of these raw materials is a result from the large food processing in countries with economy based on agriculture, such as Brazil. It implies in the generation of many kinds of residues, including orange seeds/skin, sugarcane bagasse, coffee pulp/husks, corn stover, peanut shells, grape pomace and cassava bagasse.

Cassava bagasse, specifically, is a residue obtained from the starch extraction process from cassava. It is a fibrous material that still presents approximately 50% of residual starch in the dry matter. In addition to that, the relative low contents in proteins and ashes reinforce advantages to the application of bioconversion processes in comparison to other residues [1]. The poor conditions of starch extraction plants cause a low efficiency of 25% based on wet material and consequently an annual amount of almost 2 million tons of wet cassava bagasse, solely in Brazil [2].

The main problem found during residues management is linked to their handling. The majority of resources should pass through a moisture removal procedure to reduce mass and volume. The dehydration process is one capable way to minimize the storage and transportation costs, besides extending the product shelf life and maintaining the initial characteristics until its destination [3].

The knowledge of water sorption behavior becomes a necessary study to correctly design equipment and drying processes [4]. The use of more accurate methods on the product drying can avoid undesirable effects that may occur on the composition [5,6]. Therefore, sorption isotherms appear to provide information about equilibrium moisture content ( $X_{eq}$ ) and water activity ( $a_w$ ) by simulating different relative humidity in equilibrium with the product. Sorption properties are useful in drying kinetics studies involving the application of Fick's law for thin layers of wet residues (Eq. (1)), for example:

$$\frac{\bar{X} - X_{eq}}{X_0 - X_{eq}} = 2 \sum_{n=0}^{\infty} \frac{1}{\gamma_n^2} \exp\left(\frac{-\gamma_n^2 Dt}{l^2}\right) \quad (1)$$

Where  $\bar{X}$  is the average moisture content in the product,  $X_0$  is the initial moisture content,  $D$  is the diffusion coefficient,  $l$  is the characteristic dimension,  $t$  is time and  $\gamma_n^2 = (2n + 1) \frac{\pi}{2}$ .

The determination and modeling of sorption isotherms in different temperatures gives information about the product conditions

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during storage, since the material stability can be predicted by the availability of water molecules in front of variations in temperature and relative humidity [7]. Moreover, the degree of interactions between water molecules and the residue matrix is as important as the amount of water molecules. It can be measured by the isosteric heat of sorption and many other thermodynamic functions capable of being calculated by isotherms data [8,9].

Thermodynamic parameters are variables responsible by providing insights about the material microstructure, as well as theoretical interpretations for matrix–water interactions [10]. Among them, it could be found the differential enthalpy, differential entropy and free Gibbs energy. Differential enthalpy is valuable for designing equipment involved in water removal processes, once it represents the binding strength of water to the food compounds. On the other hand, differential entropy represents the amount of available sorption sites in a specific energy level [11]. Free Gibbs energy intend to correlate these two functions, corresponding qualitatively to the difference between the total energy available (enthalpy) and the unavailable energy (entropy) [10]. The presence of a linear correlation states that the isokinetic, or enthalpy–entropy, compensation theory exists due to changes in the sorbent–sorbate interaction. The corresponding slope is the isokinetic temperature, representing the temperature at which all reactions in series proceed at the same rate.

Thus, this work intended to characterize the composition of cassava bagasse and present the water sorption properties of cassava bagasse. Among the water sorption properties, adsorption isotherms of powdered cassava bagasse were acquired and modeled in different moisture and temperature conditions. In addition, the application of experimental data of isotherms was used to evaluate thermodynamic properties and the enthalpy–entropy compensation theory, clarifying the water adsorption mechanisms.

## 2. Material and methods

### 2.1. Raw material

The cassava bagasse was obtained directly from a starch extraction industry in northwest of São Paulo state, Brazil. The industrial waste was collected at the entrance of the storage silo and transported in coolers until the Laboratory of Physical Measurements of UNESP (Campus of São José do Rio Preto), where it was stored in plastic bags at 258.15 K in a freezer. The bagasse was naturally thawed to proceed to the analyses.

In order to carry out the isotherms analyses, cassava waste was dried in a convective tray dryer as reported by Rosa et al. [4] at 323.15 K by approximately 24 h. The dried waste was then milled using a rotor mill (model MA340, Marconi, Piracicaba, São Paulo, Brazil) equipped with a 30 mesh sieve to obtain a powdered cassava bagasse with particle size less than 595  $\mu\text{m}$ .

Saturated salt solutions were prepared to obtain an  $a_w$  range from 0.046 to 0.873 using lithium bromide (LiBr), lithium chloride (LiCl), lithium iodide (LiI), potassium acetate ( $\text{CH}_3\text{OOK}$ ), magnesium chloride ( $\text{MgCl}_2$ ), magnesium nitrate ( $\text{Mg}(\text{NO}_3)_2$ ), sodium bromide (NaBr), sodium nitrate ( $\text{NaNO}_3$ ), sodium chloride (NaCl), potassium bromide (KBr) and potassium chloride (KCl), obtained from Sigma–Aldrich (St. Louis, MO, USA). Table 1 contains the specifications for each salt.

### 2.2. Cassava bagasse composition

Eleven samples of wet fibrous mass of cassava of different production days were acquired from the starch extraction industry. They were characterized with respect to moisture and dry matter in an overnight oven at 378.15 K, ashes in a muffle at 823.15 K by

**Table 1**  
Sample table with salt specifications.

Salt	Supplier	Purity (%)
LiBr	Sigma–Aldrich	$\geq 99.0$
LiCl	Sigma–Aldrich	$\geq 99.0$
LiI	Aldrich (Sigma–Aldrich)	99.0
$\text{CH}_3\text{OOK}$	Sigma–Aldrich	$\geq 99.0$
$\text{MgCl}_2$	Sigma (Sigma–Aldrich)	$\geq 98.0$
$\text{Mg}(\text{NO}_3)_2$	Vetec (Sigma–Aldrich)	98.0
NaBr	Sigma–Aldrich	$\geq 99.0$
$\text{NaNO}_3$	Sigma–Aldrich	$\geq 99.0$
NaCl	Sigma–Aldrich	$\geq 99.0$
KBr	Sigma–Aldrich	$\geq 99.0$
KCl	Sigma–Aldrich	$\geq 99.0$

4 h, fats by Soxhlet extraction, proteins by micro–Kjeldahl method and carbohydrates content by difference. All methods were applied as recommended by AOAC [12].

### 2.3. Experimental analysis

Static gravimetric method was used to determine the isotherms of water sorption along the temperature range [13]. Different equilibrium relative humidities ( $RH = a_w \times 100$ ) were reached by using glass desiccators with saturated salt solutions of LiBr, LiCl, LiI,  $\text{CH}_3\text{OOK}$ ,  $\text{MgCl}_2$ ,  $\text{Mg}(\text{NO}_3)_2$ , NaBr,  $\text{NaNO}_3$ , NaCl, KBr and KCl dissolved into de-ionized water. The temperature of desiccators were set in a controlled temperature chambers type BOD (MA415, Marconi, Piracicaba, Brazil) to maintain temperatures of 293.15, 303.15, 313.15 K and an oven (MA030, Marconi, Piracicaba, Brazil) for higher temperatures of 323.15, 328.15, 338.15, 343.15, 348.15 and 353.15 K. All experiments were carried out at atmospheric pressure  $P_{\text{atm}} = 95.47$  kPa, measured by a barometer. Table 2 contains all values of  $a_w$  obtained from the study reported by Labuza [14] for each salt solution versus experimental data of  $X_{\text{eq}}$  at each specific temperature.

Approximately 3.0 g of powdered cassava bagasse in triplicate were weighed in small containers and placed into the desiccator. Samples were previously weighted and had the initial moisture content determined. The weights of the samples were measured every 4 days using an analytical balance (AUW220D, Shimadzu, Japan) until reach constant weight (about 4 weeks). Throughout the weight difference from the initial moisture, the equilibrium moisture content ( $X_{\text{eq}}$ ) of each condition was determined.

### 2.4. Isotherms modeling

The mean equilibrium moisture content was plotted against water activities to represent the water adsorption by the nine isotherms obtained. Although isotherms provide information about equilibrium moisture, their modeling also gives water activity values for specific equilibrium moisture content. Using the software OriginPro 8.0 (OriginLab Corporation, Northampton, MA, USA), non-linear regressions were performed to adjust the mathematical models commonly found in literature, such as:

Peleg's model [15]:

$$X_{\text{eq}} = k_1 a_w^{n_1} + k_2 a_w^{n_2} \quad (2)$$

Halsey's model [16]:

$$X_{\text{eq}} = (-h_1 \ln(a_w))^{-\frac{1}{h_2}} \quad (3)$$

Henderson's model [17]:

$$X_{\text{eq}} = \left( -\frac{1}{H_1} \ln(1 - a_w) \right)^{\frac{1}{H_2}} \quad (4)$$

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