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MATHEMATICAL MODELING AND HYSTERESIS OF SORPTION ISOTHERMS FOR PADDY RICE GRAINS

Juliana S. Zeymer[1*], Paulo C. Corrêa[2], Gabriel H. H. Oliveira[3], Fernanda M. Baptestini[4], Renata C. Campos[2]

[1*]Corresponding author. Federal University of Viçosa/ Viçosa - MG, Brazil.
E-mail: jujuszeymer@gmail.com | ORCID ID: http://orcid.org/0000-0003-1553-3494

KEYWORDS

adsorption, desorption, hygroscopic equilibrium, relative humidity, temperature.

ABSTRACT

Knowledge on sorption isotherms is important for predicting drying and storage processes of a product. A sorption isotherm can be generated from two processes: desorption and adsorption. The lag between these two curves is called hysteresis. Static method was used to obtain the equilibrium moisture content of paddy rice grains, in different temperature (10, 20, 30, 40 and 50 ± 1°C) and relative humidity (between 11 and 76% ± 2%) conditions. Equilibrium moisture content data were correlated with eight mathematical models and. The Chung Pfost model had the best fit to the experimental data. Desorption and adsorption isotherms, represented by the Chung Pfost model, showed a sigmoidal shape, characteristic of type II curve. Equilibrium moisture content values obtained by desorption were higher than those obtained by adsorption, evidencing the hysteresis phenomenon.

INTRODUCTION

Rice (*Oryza sativa* L.) is a cereal of elevated economic and social value, being a basic food for more than half of the world´s population (Utami et al., 2017). It is one of the three most produced and consumed cereals worldwide, behind wheat and corn (Ziegler et al., 2017).

Rice is harvested according to the seasons over the year, and post-harvest procedures are determinant for rice quality maintenance throughout the year (Ziegler et al., 2017). A series of physical-chemical, biochemical and metabolic reactions happen during rice grain storage. These reactions are directly influenced by the storage system, initial grain quality, grain moisture content, temperature and air relative humidity in the storage environment (Chen et al., 2015).

Equilibrium moisture content is given by the equilibrium between moisture content of the grains, and the psychrometric conditions of the surrounding air. This condition is reached when the partial pressure of water vapor in the product is equal to the partial pressure of water vapor in the surrounding air (Corrêa et al., 2014). Equilibrium moisture content is useful for determining water loss or gain under certain conditions of temperature and relative humidity, and is directly related to drying, aeration and storage (Corrêa et al., 2010).

For constant air relative humidity and water activity, there are two equilibrium moisture contents, depending on the experimental conditions (adsorption or desorption), since the product may have a lower or higher moisture content than the equilibrium for the current environmental conditions (Corrêa et al., 2014). For a given water activity, the equilibrium moisture content values, in a given water activity, is higher in desorption isotherm than in adsorption isotherm.

According to Brooker et al. (1992), this phenomenon occurs because grains are porous materials formed by narrow capillaries of small diameters and tubes of bigger diameters. In addition to that, pores of small diameters control capillary emptying during the desorption process, resulting in the reduction of relative humidity in the grain porous media. When grain gains moisture during adsorption process, capillaries are not completely filled as they were prior to desorption, leading to a lower moisture content value than before, which is the hysteresis effect (Wolf et al., 1972). Hysteresis is important to determine the required protection against moisture gain and to estimate deterioration possibility due to chemical reactions and microorganisms (Fellows, 2006).

[2] Federal University of Viçosa/ Viçosa - MG, Brazil.
[3] Federal Institute of Minas Gerais Southeast/ Manhuaçu - MG, Brazil.
[4] Federal University of Espírito Santo/ Alegre - ES, Brazil.
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Relationship between a product´s moisture content and relative humidity at equilibrium (water activity) may be expressed by mathematical models. These models are important, because they report the product´s moisture content at a given environmental condition, without the need to accomplish expensive and time-consuming tests. Furthermore, in addition to weather forecast, mathematical models may be used in order to predict what may happen to stored grains (Corrêa et al., 2016).

Kachru & Matthes (1976) studied the sorption pattern of rough rice, at temperatures between 19 ºC and 42 ºC, demonstrating the hysteresis effect. Durakova & Menkov (2004) reported the moisture sorption characteristics of rice flour at three storage temperatures (10, 20 and 30°C), using the Brunauer-Emmett-Teller (BET) equation. Investigation on sorption isotherms of rice oil powder was done using the Guggenheim-Anderson-de Boer (GAB) model (Ratchanee et al., 2015). Mousa et al. (2014) researched the sorption isotherms of rough rice MR219 variety at temperatures of 20, 30, 40 and 50 ºC. However, researches aiming to analyze the sorption process of paddy rice grain, cv. Urucuia are scarce or absent in scientific literature. Studies on hygroscopic equilibrium for different agricultural products and cultivars are essential, even when products are physically and chemically similar. Absence of hygroscopicity data for paddy rice grain led the industry to use data from paddy red rice, resulting in significant errors regarding equilibrium moisture content values.

Due to the lack of researches regarding paddy rice grain hygroscopy and the need to properly store these grains, this work aimed to determine the sorption isotherms of paddy rice grain, obtained by desorption and adsorption, at different temperature and air relative humidity conditions and analyzing the hysteresis phenomenon.

MATERIAL AND METHODS

The study was conducted in the Agricultural Products Physical Properties and Quality Laboratory belonging to the National Storage Training Center (CENTREINAR), located at the Federal University of Viçosa, Viçosa, MG, Brazil.

Irrigated paddy rice grains cv. Urucuia, from the EPAMIG Experimental Farm, in southern Minas Gerais, were used. Grains were hand harvested with an initial moisture content of approximately 0.28 (d.b.) and used in the desorption process. For the adsorption process, grains were dehydrated in an oven with forced air circulation (model 400-3ND/Gehaka brand) at 40 ºC, until reaching a final moisture content of 0.17 (d.b.). Later, these grains were submitted to different air conditions in order to gain moisture (adsorption).

The static method (Brasil, 2009), with five different temperatures (10, 20, 30, 40 and 50 ± 1 °C) and four relative humidities (10, 30, 50, 70% ± 2%) (Table 1), was employed in order to acquire the equilibrium moisture content of paddy rice grains, for both desorption and adsorption processes. Thus, leading to a total of 20 different applied combinations.

TABLE 1. Relative humidity values related to saturated salt solutions at temperatures of 10, 20, 30, 40 and 50 °C.

Saline solution	Temperature (°C)				
	10	20	30	40	50
LiCl	11.3	11.3	11.3	11.2	11.1
$MgCl_2$	33.5	33.1	32.4	31.6	30.5
$Mg(NO_3)_2$	57.4	54.4	51.4	49.9	45.4
NaCl	75.7	75.5	75.1	74.7	74.4

Each saturated salt solution was placed inside hermetic desiccators. The sorption recipients were placed above the solutions, with three repetitions, containing approximately 20 g of sample. Desiccators containing the samples were placed in BOD chambers (model 347 CD/Fanem brand) in order to control the internal temperature required for the experiment.

During the process, sorption recipients were periodically (intervals of 24 h) weighted in an analytical scale (model AY220/ Marte brand). Hygroscopic equilibrium was reached when mass variation remained invariable or lower than 0.01 g for three consecutive weighings. After hygroscopic equilibrium was reached, moisture content of each sample was determined using the gravimetric method, using an oven with forced air circulation at 105 ± 1°C for 24 h in three repetitions, according to Brasil (2009).

Sorption isotherms of paddy rice grain was determined using a mathematical model that best represented experimental data. This mathematical model was selected among the ones showed in Table 2, which are frequently used to represent the hygroscopicity of agricultural products.

TABLE 2. Mathematical models to represent hygroscopic equilibrium curves.

Model name	Model	Equation number
Modified Henderson	$U_e = \left[\frac{\ln(1-a_w)}{-a(T+b)}\right]^{\frac{1}{c}}$	(1)
Modified Halsey	$U_e = \left[\frac{\exp(a-bT)}{-\ln(a_w)}\right]^{\frac{1}{c}}$	(2)
Modified Oswin	$U_e = (a+bT)\left[\frac{a_w}{1-a_w}\right]^{\frac{1}{c}}$	(3)
Copace	$U_e = \exp[a-(bT)+(ca_w)]$	(4)
Sigma-Copace	$U_e = \exp[a-(bT)+c\exp(a_w)]$	(5)
Chung Pfost	$U_e = a - b\ln[-(T+c)\ln(a_w)]$	(6)
Smith	$U_e = a-(bT)-c\ln(1-a_w)$	(7)
Harkins Jura	$U_e = \frac{\exp(a-bT)}{c-\ln(a_w)}$	(8)

In which:

U_e - equilibrium moisture content, % d.b.;

a_w - water activity, (decimal);

T – temperature, (ºC), and

a, b, c - coefficients that depends upon the product.

Model was adjusted to experimental data by non-linear regression analysis, throughout the Gauss-Newton method, using Statistica software. In order to select the model that best predicted the equilibrium moisture content of paddy rice grain, the following statistical parameters were considered: determination coefficient (R^2), mean relative error (MRE), estimated standard error (SEE), and randomness of residual values. The MRE and SEE values of each model were calculated using eqs (9) and (10), respectively:

$$MRE = \frac{100}{n}\sum\frac{|Y-\hat{Y}|}{Y} \quad (9)$$

$$SEE = \sqrt{\frac{\Sigma(Y-\hat{Y})^2}{DF}} \quad (10)$$

In which:

MRE - mean relative error, %;

SEE- estimate standard error, % d.b.;

Y - observed value;

$\hat{Y}$ - estimated value by the model;

n - number of observed data, and

DF - residual degrees of freedom (number of observed data minus number of model parameters).

Folowing model selection, analysis of the hysteresis phenomena was made using the sorption isotherms, which were obtained through the difference between the equilibrium moisture content values encountered for desorption and adsorption.

RESULTS AND DISCUSSION

Tables 3 and 4 show the parameters of the mathematical models fitted to the hygroscopic equilibrium experimental data, obtained for desorption and adsorption, respectively, under different air temperatures and relative humidities. Furthermore, values of mean relative error (MRE), estimated standard error (SEE), coefficient of determination (R^2) and residual values analysis are also presented.

TABLE 3. Model parameters fitted to equilibrium moisture content of paddy rice grains, with coefficient of determination (R^2), estimated standard error (SEE) and mean relative error (MRE), obtained by desorption.

Model	Parameters*	R^2 (%)	SEE (decimal)	MRE (%)	Residual plot
Modified Henderson	a = 0.00005 b = 59.29579 c = 2.01760	99.08	0.74	8.07	Random
Modified Halsey	a = 4.978758 b = 0.011060 c = 2.044422	97.43	1.19	13.65	Biased
Modified Oswin	a = 14.25468 b = -0.06960 c = 2.84464	98.80	0.84	8.89	Biased
Copace	a = 1.839119 b = 0.005876 c =1.639578	98.14	1.04	11.45	Biased
Sigma-Copace	a = 1.032387 b = 0.005735 c = 0.959289	96.86	1.35	15.65	Biased
Chung Pfost	a = 37.41332 b = 6.23300 c = 49.64200	99.60	0.49	5.16	Random
Smith	a = 7.669984 b = 0.080895 c = 9.388660	98.34	0.99	11,45	Biased
Harkins-Jura	a = 2.965899 b = 0.005945 c = 0.620576	98.92	0.80	8.69	Random

* Significance level of 1% for “t” test

TABLE 4. Model parameters fitted to equilibrium moisture content of paddy rice grains, with coefficient of determination (R^2), estimated standard error (SEE) and mean relative error (MRE), obtained by adsorption.

Model	Parameters*	R^2 (%)	SEE (decimal)	MRE (%)	Residual plot
Modified Henderson	a = 0.00008 b = 62.29053 c = 1.87538	99.11	0.70	9.59	Random
Modified Halsey	a = 4.478713 b = 0.010657 c = 1.915281	97.81	1.11	14.78	Biased
Modified Oswin	a = 13.11786 b = -0.06562 c = 2.65584	98.96	0.76	9.88	Random
Copace	a = 1.700181 b = 0.006091 c =1.754145	98.45	0.93	12.22	Random
Sigma-Copace	a = 0.841173 b = 0.005930 c = 1.024176	97.35	1.21	16.84	Biased
Chung Pfost	a = 35.93673 b = 6.08728 c = 50.48329	99.64	0.45	5.99	Random
Smith	a = 6.747202 b = 0.077581 c = 9.217345	98.80	0.82	11.25	Biased
Harkins-Jura	a = 2.809354 b = 0.006131 c = 0.531248	99.02	0.74	9.82	Random

* Significant at 1% probability by “t” test

The analyzes of tables 3 and 4, show that all mathematical models had elevated coefficient of determination values, which according to Kashaninejad et al. (2007) indicates a satisfactory representation of this models to the phenomenon under investigation. However, the coefficient of determination must be used just as an aid index for nonlinear models (Baptestini et al., 2017a). For a more detailed analysis, remaining statistical parameters must be used.

Several authors argue that a model is adequate if the MRE value is lower than 10% (Rosa et al., 2010; Castell-Palou et al., 2012; Corrêa et al., 2014; Costa et al., 2015). According to Kashaninejad et al. (2007), MRE values indicates a deviation of the observed values, relating to the estimated curve by the model. Thus, it was verified that Modified Henderson, Modified Oswin, Chung Pfost and Harkins-Jura models had satisfactory MRE values (below 10%). However, among the listed models, the Chung Pfost model had the lower MRE values, in both sorption processes.

According to Draper & Smith (1998), the model´s ability to reliably describe a specific physical process is inversely proportional to the SEE value. Thus, lower values of this variable indicate a better fit of the model to the observed data. Chung Pfost model also had lower SEE values among the tested models.

To assure that the selected model is capable to describe the phenomenon, an analysis of the residual plot is recommended. The chosen model must have residual values close to the horizontal zone, around zero, and should not form geometric figures. If these trends occur, the model will have regions that overestimate or underestimate the real condition, thus the model is inadequate to represent the phenomenon under study (Goneli et al., 2011; Alves et al., 2013; Corrêa et al., 2014).

Residual distribution in the Chung Pfost model, for both desorption and adsorption, is random among the equilibrium moisture content range, with positive and negative values (Figure 1).

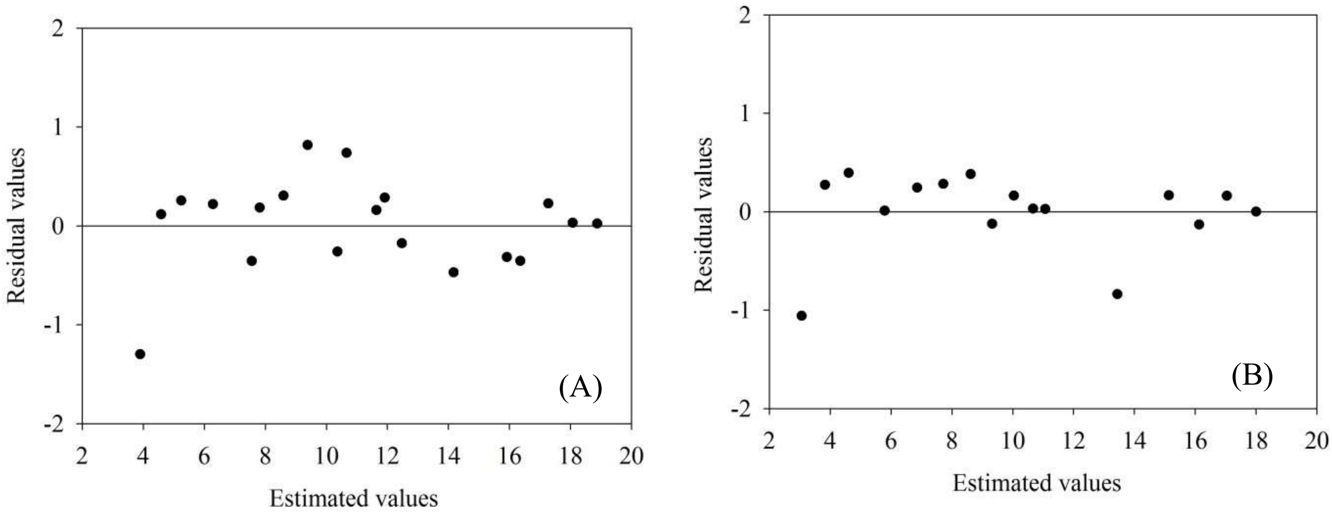

FIGURE 1. Chung Pfost model residual values for desorption (A) and adsorption (B) of paddy rice grains.

Through analyses of the statistical parameters used in this study, it be concluded that the Chung Pfost model was the most representative of hygroscopic equilibrium for the paddy rice grain experimental data , showing R^2 between 99.60 and 99.64%, SEE between 0.49 and 0.45 and MRE between 5.16 and 5.99 %, along with residual random distribution. Mousa et al. (2014) recommended the Oswin model to represent sorption of Malaysian paddy, indicating the importance of studying different varieties of the same product, due to differences in chemical composition.

According to Ullmann et al. (2016), the Chung Pfost model has been used for starchy products, being the model chosen to represent the hygroscopicity of sweet sorghum seeds. This model also fitted satisfactorily to hygroscopic equilibrium of okra (Goneli et al., 2010), wheat (Li, 2012), cotton (Oliveira et al., 2013), sugar beet (Corrêa et al., 2016), kalibu chickpea, black sesame and white sesame(Armstrong et al., 2017) and red kidney beans (Jian & Jayas, 2018) seeds.

Figure 2 shows the experimental results of equilibrium moisture content of paddy rice grain, obtained for desorption and adsorption, along with the isotherms determined by the Chung Pfost model.

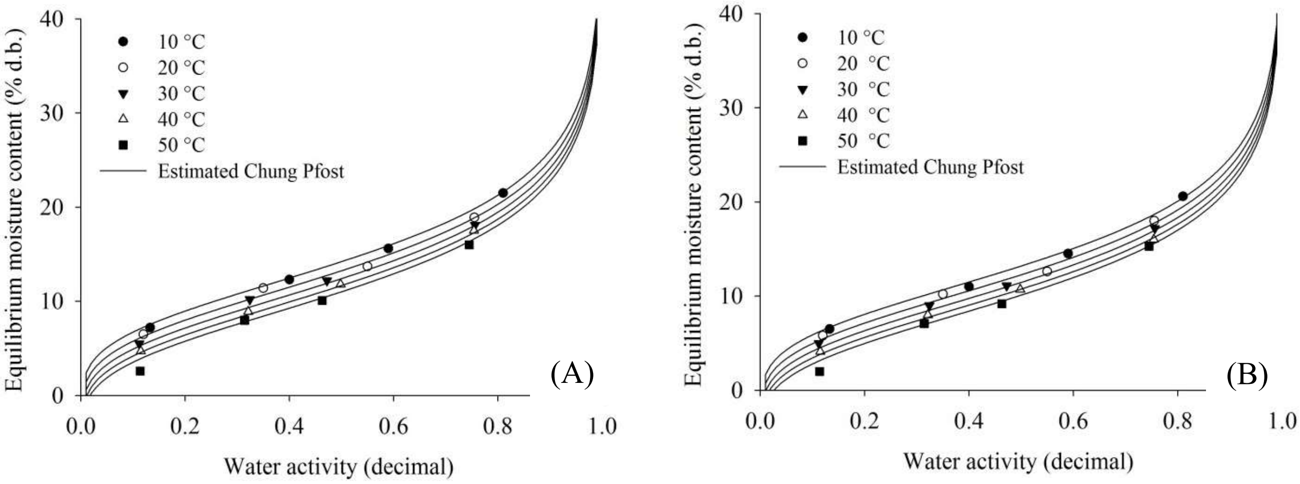

FIGURE 2. Observed and estimated values, by the Chung Pfost model, of equilibrium moisture content of paddy rice grain, obtained for desorption (A) and adsorption (B).

Through Figure 2, it can be verified temperature influence over the sorption isotherms of paddy rice grain, since it requires an increment of water activity in order to attain the same equilibrium moisture content for increasing temperature. Recent studies reported the same behavior for different agricultural products (Oliveira et al., 2017a; Oliveira et al., 2017b; Zeymer et al., 2017; Arufe et al., 2018; Espinosa-Andrews & Rodríguez-Rodríguez, 2018).

García-Pérez et al. (2008) argue that as temperature rises, there is an increase of the molecular disorder degree of water present at the adsorbent surface, leading to instability and facilitating the breakage of intermolecular connections between water molecules and sorption sites, thus allowing water vaporization and, consequently, decreasing equilibrium moisture content.

Desorption and adsorption isotherms of paddy rice grain (Figure 2) exhibited a sigmoidal shape type, characteristic of a type II curve, according to the IUPAC classification (1985). As stated by Labuza & Altunakar (2007), the type II isotherms shape is caused by synergistic effects of the Raoult's law, capillaries effects and moisture interactions at the material surface.. These authors also report that this isotherm shape describes two regions, one between 0.2 and 0.4 of equilibrium moisture content and another between 0.6 and 0.7. These regions are the result of physicochemical processes, such as, the multilayers creation and the pores filling (0.2-0.4 region), followed by larger pores filling and solutes dissolution (0.6-0.7 region). Several researchers verified the same pattern in starchy products, including rice (Brett et al., 2009; Mousa et al., 2014; Chen et al., 2017; Purohit & Rao, 2017; Zhao et al., 2017).

Table 5 shows temperature and relative humidity values used in the experiment, along with the equilibrium moisture content for paddy rice grains experimental data obtained for desorption and adsorption, and hysteresis magnitudes.

TABLE 5. Equilibrium moisture content experimental values for desorption, adsorption and hysteresis of paddy rice grains.

Temperature (°C)	Relative humidity (%)	U_e desorption (% d.b.)	U_e adsorption (% d.b.)	Hysteresis (% d.b.)
10	13.30	7.20	6.50	0.700
	40.00	12.30	11.00	1.300
	59.00	15.60	14.50	1.100
	81.00	21.50	20.60	0.900
20	12.00	6.50	5.80	0.700
	35.00	11.40	10.20	1.200
	55.00	13.70	12.60	1.100
	75.50	18.90	18.00	0.900
30	11.20	5.50	5.00	0.500
	32.40	10.20	9.00	1.200
	47.20	12.20	11.10	1.100
	75.60	18.10	17.20	0.900
40	11.50	4.70	4.10	0.600
	32.10	8.90	8.00	0.900
	49.80	11.80	10.70	1.100
	75.40	17.50	16.60	0.900
50	11.40	2.60	2.00	0.600
	31.40	8.00	7.10	0.900
	46.30	10.10	9.20	0.900
	74.50	16.00	15.30	0.700

in which,

U_e desorption - equilibrium moisture content of desorption, % d.b.,

U_e adsorption - equilibrium moisture content of adsorption, % d.b.

Comparing equilibrium moisture content values for desorption and adsorption, for all air conditions used, leads to the conclusion that values acquired for desorption are always higher than those obtained for adsorption (Table 5).

One of the most accepted theories to explain the hysteresis phenomenon suggests that, during adsorption, the grain porous region formed by capillaries begins to swell up, due to the increase in relative humidity. When partial pressure of the the air´s water vapor becomes higher than the capillary´s vapor pressure, water moves towards the pore interior. During desorption, the pore is saturated at the beginning of the process. Water diffusion, from the grain´s periphery to surface, occurs when the partial pressure of the surrounding air´s water vapor is lower than the vapor pressure inside the capillary (Lahsasni et al., 2004).

It is possible to observe the temperature and water activity effect at the hysteresis (equilibrium moisture content of desorption minus the equilibrium moisture content of adsorption) of paddy rice grain in Figure 3.

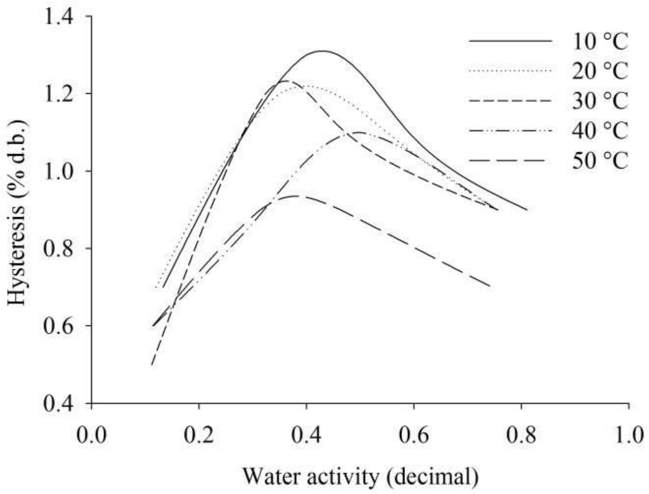

FIGURE 3. Temperature effect and water activity on hysteresis variation of paddy rice grains.

Hysteresis effect occurs during the entire water activity range, being more pronounced between 0.32 and 0.59, for all temperatures evaluated (Figure 3).

Analyzes of Figure 3, shows that hysteresis magnitudes decrease as temperature increases, as verified by different authors (Bejar et al., 2012; Corrêa et al., 2014; Souza et al., 2015; Goneli et al., 2016; Baptestini et al., 2017b; Torres et al., 2018). However, hysteresis values at 30 ºC were higher than hysteresis at 20 ºC, at the water activity interval between 0.3 and 0.4, probably due to the elasticity increase of the capillary walls. Similar results were encountered by Dalgiç et al., (2012), studying sorption isotherms and thermodynamic properties of mint leaves at temperatures of 15, 25 and 35 ºC. These authors verified that the hysteresis value at 25 ºC was lower when compared to the hysteresis at 35 ºC.

CONCLUSIONS

Equilibrium moisture content of paddy rice grain cv. Urucuia, is directly proportional to relative humidity and decreases as temperature increases, at a constant value of water activity.

Based on statistical parameters, the Chung Pfost model was the one that best fitted to experimental data, when compared to the other tested models, thus being chosen to represent the hygroscopicity of paddy rice grain.

Desorption and adsorption isotherms of paddy rice grain cv. Urucuia, represented by Chung Pfost model, exhibited a sigmoidal shape, characteristic of a type II curve.

Equilibrium moisture content values were higher for desorption than for adsorption, indicating the hysteresis phenomenon.

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