Hygroscopic behaviour of cassava flour from dry and water groups

Comportamento higroscópico das farinhas de mandioca tipos seca e d'água

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ABSTRACT

Cassava flour from dry and water groups are important starch products widely consumed in the Amazonian region of Brazil. In this study, the moisture sorption isotherms of cassava flour from dry and water groups were obtained in the water activity (a_w) range from 0.09 to 0.94 (dry group) and 0.07 to 0.93 (water group) at 25°C and the applicability of eight mathematical models in data prediction was evaluated. Both cassava flour from dry and water groups exhibited type II isotherms (sigmoid) and the hysteresis effect between adsorption and desorption isotherms was shown almost over the entire range of a_w. The moisture of cassava flour from dry and water groups should not exceed 11.3g H_2O $100g^{-1}$ (a_w=0.6) to ensure the theoretical microbiological stability and the monolayer moisture value (9.0 and 7.9 g H_2O $100g^{-1}$, respectively) indicated the level of moisture content to be reached during the drying process to avoid unnecessary power consumption. Finally, the data fit showed that the mathematical model of GAB were able to predict efficiently the sorption isotherms of both cassava flour from dry and water group at the entire a_w range.

Key words: **Manihot esculenta**, sorption isotherms, mathematical models, starchy product, water activity.

RESUMO

As farinhas de mandioca do grupo seca e do grupo d'água são produtos amiláceos amplamente consumidos na região amazônica do Brasil. Neste estudo, as isotermas de adsorção e dessorção de umidade foram obtidas para ambos os produtos a 25°C, na faixa de atividade de água (a_w) de 0,09 a 0,94 (tipo seca) e 0,07 a 0,93 (tipo d'água). Foi avaliada a aplicabilidade de oito modelos matemáticos na predição das isotermas de sorção. Tanto a farinha tipo seca quanto a farinha tipo d'água apresentaram isotermas tipo II (sigmoide) e um efeito de histerese entre as isotermas de adsorção e dessorção, que se estendeu em toda a faixa de a_w. Para que a estabilidade microbiológica teórica seja assegurada, a umidade em ambas as farinhas de mandioca não pode alcançar valores superiores a 11,3 g H_2O $100g^{-1}$ (a_w=0,6). O valor da monocamada das farinhas seca e d'água (9,0 e 7,9 g H_2O $100g^{-1}$, respectivamente) indicou que esses níveis de umidade não devem ser atingidos durante a secagem dos produtos, para evitar gasto desnecessário de energia. Finalmente, o modelo de GAB mostrou-se altamente eficiente na predição das isotermas de sorção de ambos os produtos, em toda a faixa de a_w estudada.

Palavras-chave: **Manihot esculenta**, isotermas de sorção, modelos matemáticos, produto amiláceo, atividade de água.

INTRODUCTION

Cassava is the main carbohydrate source for a significant portion of the population in Brazilian Amazonian region, especially in Pará State, with high economic importance to producers and small cities mainly due to the production of cassava flour. Cassava flour from dry and water groups are starch products widely consumed in Amazonian region, which is obtained after drying cassava roots (**Manihot esculenta** Crantz). The difference between the production of cassava flour from dry group and cassava flour from water group is related to the fermentation process of cassava roots for producing the water group flour (MAPA; 2011). The

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fermentation step of roots is performed during four days in water tanks, providing a peculiar acid taste to the cassava flour from water group and this sensory characteristic was reported as its main attractive (CHISTÉ & COHEN, 2011).

The data prediction of sorption isotherms are extremely important, since drying is the main process to obtain different kind of flours and is the most widely used method for the food preservation due to the reduction of water activity (a_w). This approach leads to the efficient modelling of drying processes, the design and optimization of drying equipment, the prediction of shelf-life, the moisture changes, which may occur during storage, and also to select appropriate packaging material (KULCHAN et al., 2010).

The water sorption data for cassava starch (MISHRA & RAI, 2006; PERDOMO et al., 2009; COVA et al., 2010; SOUZA et al., 2013) and cassava-flour-based baked product (KULCHAN et al., 2010) were described in the literature. In addition, this research also reported the hygroscopic behaviour of tapioca flour (CHISTÉ et al., 2012), another different kind of cassava product, which is produced with starch of high purity degree extracted from cassava roots.

Thus, considering the importance of cassava products to people who lives in Brazilian Amazonian region, the hygroscopic behaviour of cassava flour from both dry and water groups at storage temperature (25°C) will be reported here for the first time. Furthermore, the sorption isotherms of cassava flour were obtained and the applicability of mathematical models in data prediction for adsorption and desorption moisture was also evaluated.

MATERIAL AND METHODS

Samples

The cassava flour from dry and water groups (1kg each) was acquired in bulk at the local open-air markets in Belém, Pará, Brazil (Latitude 01°27'21''S and Longitude 48°30'16''W). The samples were ground in a food processor and homogenized. A sample of each cassava flour was evaluated.

Proximate composition

The recommended methods of the Association of Official Analytical Chemists (AOAC, 1997) were adopted to determine the moisture, ashes, total lipids, total proteins (conversion factor of 5.75 from total nitrogen to total protein), reducing sugars, total sugars and starch contents. All the experiments of proximate composition were performed in triplicate and expressed in g $100g^{-1}$.

Sorption isotherms and determination of monolayer moisture content

The hygroscopic properties of cassava flour from dry and water groups were obtained from the adsorption and desorption isotherms at 25°C, according to the procedure described by SOUZA et al. (2013). The sorption isotherms were graphically plotted considering the moisture content *versus* a_w of each measurement using Microsoft Office Excel 2003 and Statistica 7.1 softwares. The monolayer moisture content (m_o) was calculated using the BET linear equation (Equation 1), which was applied in the linear region of the isotherms range from 0.09 to 0.40 (dry group) and 0.07 to 0.35 (water group) of a_w.

$$\frac{a_w}{(1-a_w)\,m} = \frac{1}{m_o.C} + \frac{(C-1)}{m_o.C}.a_w \qquad (1)$$

where, m = moisture content (g $100g^{-1}$ dry basis - d.b.); a_w = water activity; m_o = monolayer moisture content (g $100g^{-1}$ d.b.); and C = constant related to the sorption heat.

Mathematical modelling of sorption isotherms

Eight mathematical models presented in table 1 were fitted to the isotherms by the non-linear regression using the Statistica 7.1 software. Mean relative deviation modulus (P) and coefficient of determination (R^2) were used to compare the fit precision of the sorption models and the Levenberg-Marquardt method was used in the non-linear regression procedure (P<0.05). The P values lower than 10% were adopted as an indicative of a good fit for practical purposes (PENG et al., 2007).

RESULTS AND DISCUSSION

Proximate composition of cassava flour from dry and water groups

The cassava flour from dry and water groups used to obtain the sorption isotherms presented starch as the major component (76.57±1.72 and 68.32±1.68g $100g^{-1}$, respectively, wet basis – w.b.) (Table 2). These values were in the same range or slightly superior than that reported for cassava flour from dry (67.67-79.59g $100g^{-1}$) and water (61.31-66.53g $100g^{-1}$) groups traded in ten different markets in Belém (Brazil) (CHISTÉ et al., 2006; CHISTÉ & COHEN, 2010). In addition, the other chemical constituents of these kinds of cassava flour (Table 2) were determined to obtain an overview

Table 1 - Mathematical models used to fit the sorption isotherms of cassava flour from dry and water groups.

Models	Mathematical equations	Reference
Halsey	$m = \left[\dfrac{-a}{\ln a_w}\right]^{\frac{1}{b}}$	(CHIRIFE & IGLESIAS, 1978)
Handerson	$m = \left[\dfrac{-\ln(1 - a_w)}{a}\right]^{\frac{1}{b}}$	(CHIRIFE & IGLESIAS, 1978)
Kuhn	$m = -\dfrac{a}{\ln a_w} + b$	(CHIRIFE & IGLESIAS, 1978)
Mizrahi	$a_w = \dfrac{a + m}{b + m}$	(CHIRIFE & IGLESIAS, 1978)
Oswin	$m = a\left[\dfrac{a_w}{1 - a_w}\right]^b$	(CHIRIFE & IGLESIAS, 1978)
Smith	$m = a - b \cdot \ln(1 - a_w)$	(CHIRIFE & IGLESIAS, 1978)
BET	$m = \dfrac{m_o \cdot c \cdot a_w}{1 - a_w} \cdot \left(\dfrac{1 - (n+1) \cdot a_w^n + n \cdot a_w^{n+1}}{1 - (1-c) \cdot a_w - c \cdot a_w^{n+1}}\right)$	(FIGUEIRA et al., 2004)
GAB	$m = \dfrac{m_o \cdot c \cdot k \cdot a_w}{[(1 - k \cdot a_w) \cdot (1 + (c-1) \cdot k \cdot a_w)]}$	(MAROULIS et al., 1988)

m = moisture content (g 100g^{-1} d.b.); a_w = water activity; a, b, m_o, k and c terms are the parameters to be estimated by fitting.

about the chemical composition, all in accordance with the previous values reported by our research group (CHISTÉ et al., 2006; CHISTÉ et al., 2007; CHISTÉ & COHEN, 2010; CHISTÉ & COHEN, 2011). However, only the starch content of cassava flour from dry group (84±2%, calculated in dry basis - d.b.) was in agreement with the Brazilian law (MAPA, 2011), that requires at least 80% of starch content and was classified as type 2. The starch content of the cassava flour from water group (74±2% d.b.) did not follow the same requirements.

Table 2 - Proximate composition and water activity of cassava flour from dry and water groups.
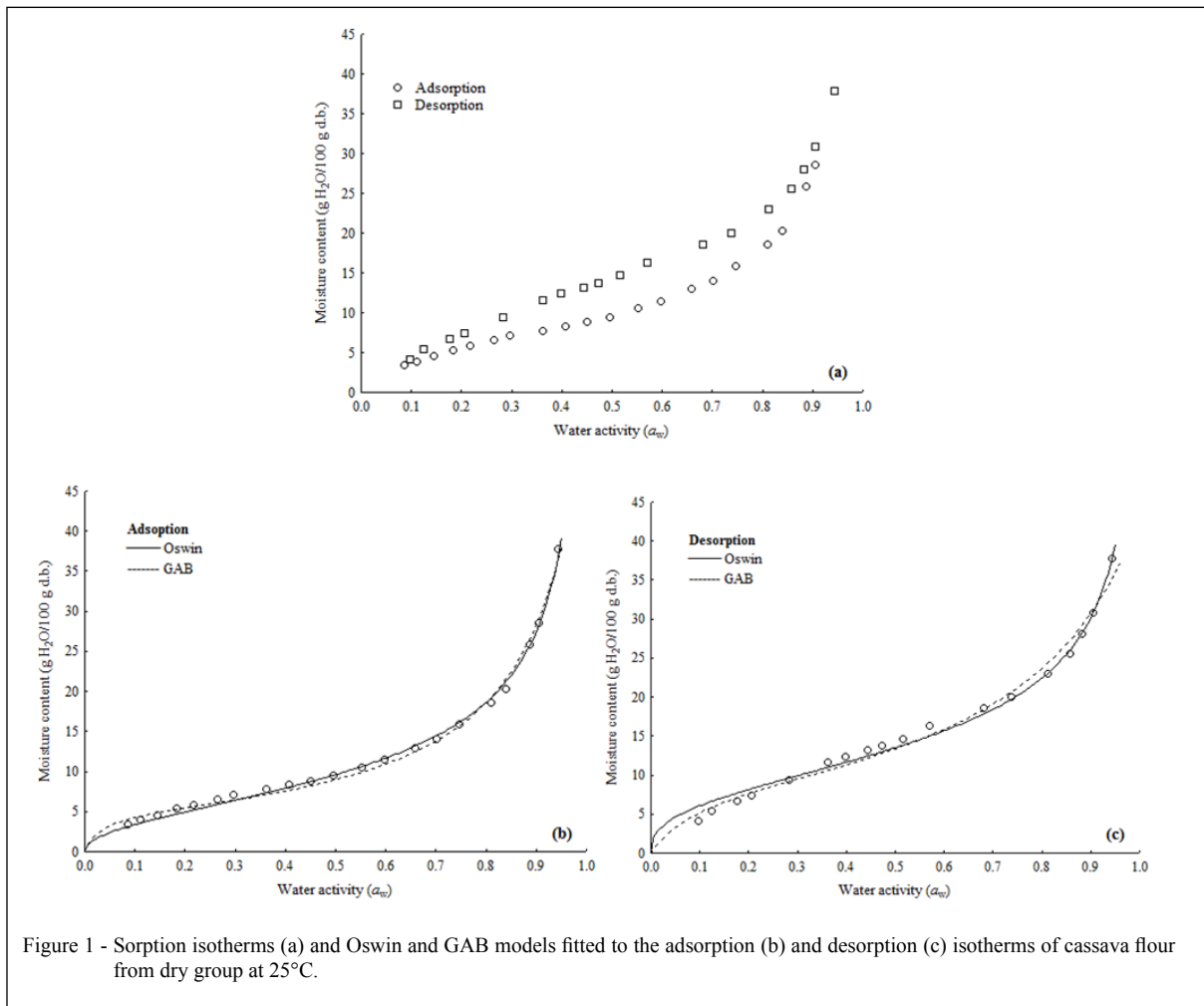
	------------Cassava flour----------	
	Dry group	Water group
Moisture (g 100g^{-1})	9.17 ± 0.02	8.28 ± 0.07
Ashes (g 100g^{-1})	0.83 ± 0.01	0.75 ± 0.01
Total lipids (g 100g^{-1})	0.26 ± 0.06	1.04 ± 0.05
Total proteins (g 100g^{-1})	0.52 ± < 0.01	1.10 ± < 0.01
Reducing sugars (g 100g^{-1})	0.51 ± < 0.01	0.34 ± 0.02
Total sugars (g 100g^{-1})	1.10 ± 0.02	0.42 ± 0.01
Starch (g 100g^{-1})	76.57 ± 1.72	68.32 ± 1.68
Water activity (a_w)	0.53 ± < 0.01	0.45 ± 0.01

Sorption isotherms of cassava flour from dry and water groups

The sorption isotherms at 25°C of cassava flour from both groups (Figure 1a and 2a) were classified as a typical sigmoid (type II). The same behaviour was reported for other cassava products (MISHRA & RAI, 2006; PERDOMO et al., 2009; CHISTÉ et al., 2012).

Considering that water molecules are strongly bound to hydrophilic biopolymers, such as proteins and polysaccharides, the number of sites that strongly bind water molecules must be less in the protein-rich substrate than in the carbohydrate rich substrate. As a result, starchy foods, such as cassava flour, show more Langmuir-like type II isotherms, while the protein-rich foods indicate more solution-like type II isotherms (YANNIOTIS & BLAHOVEC, 2009).

Hysteresis loop between adsorption and desorption isotherms exhibited consistent characteristics with the type-H3 hysteresis loop, according to IUPAC classification (SING, 1982) and was observed almost over the entire range of a_w for both cassava flour (Figure 1a and 2a); whereas, a more pronounced hysteresis effect was observed for cassava flour from dry group. According to CAURIE

Figure 1 - Sorption isotherms (a) and Oswin and GAB models fitted to the adsorption (b) and desorption (c) isotherms of cassava flour from dry group at 25°C.

(2007), a decrease in the hysteresis loop or its complete absence has been related to greater product stability during storage.
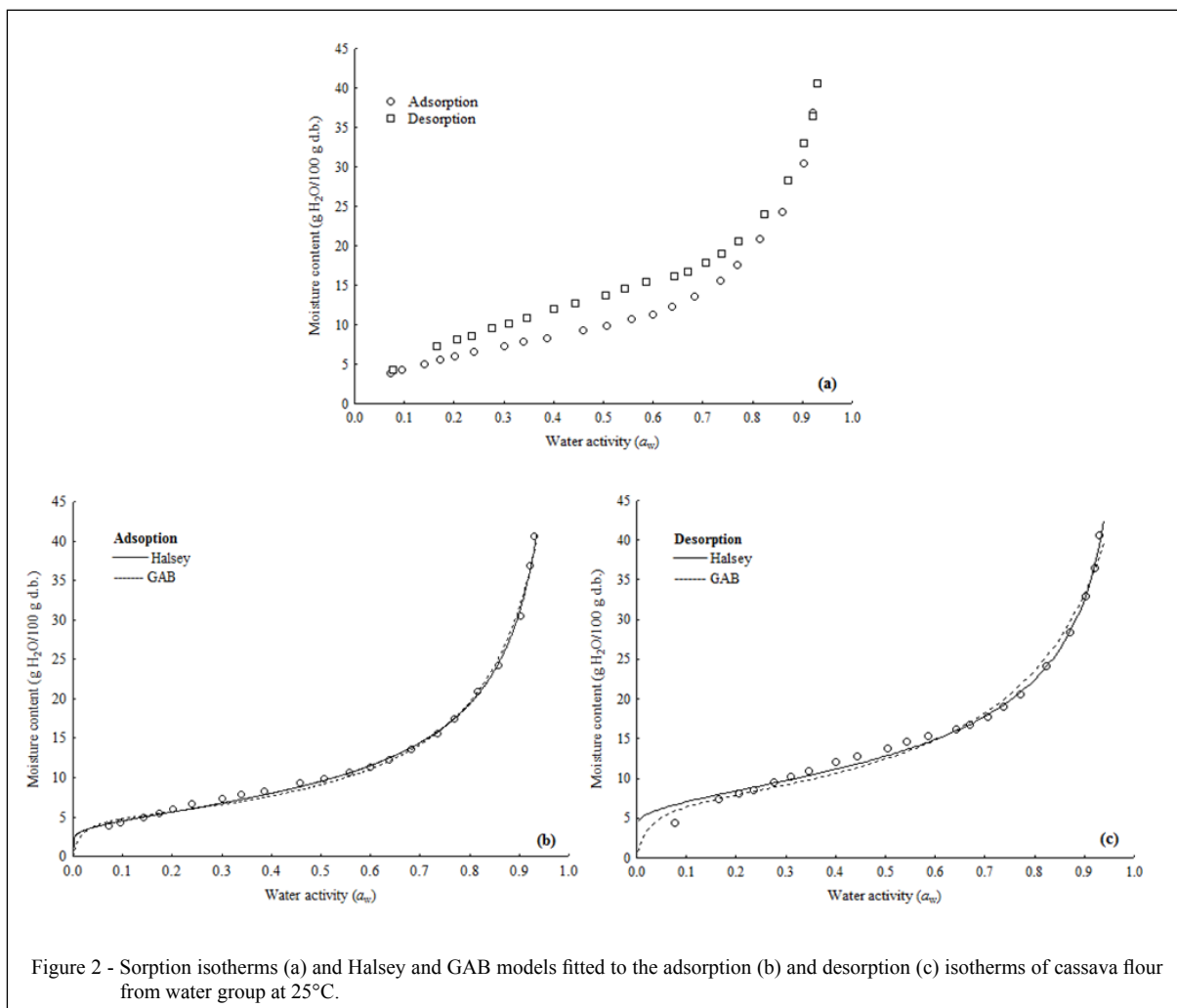
Considering that the theoretical microbiological stability can be ensured at $a_w < 0.6$ (SCOTT, 1957), the moisture content of cassava flour from dry group and from water group should not be higher than 11.3g H_2O $100g^{-1}$ d.b (for both products) during storage conditions at 25°C. Similar results were found for tapioca flour (10.1g H_2O $100g^{-1}$ d.b.) (CHISTÉ et al., 2012).

The desorption monolayer moisture contents of 9.0g H_2O $100g^{-1}$ d.b. (cassava flour from dry group) and 7.9g H_2O $100g^{-1}$ d.b. (cassava flour from water group) indicated the level of moisture contents to be reached during the drying process to avoid unnecessary power consumption. Since, the chemical reactions that depend on solvation are also expected to be slow in the

monolayer region (RAO et al., 2006), the a_w of both products is lower than 0.30 in this moisture levels and the microbiological stability will be highly ensured. These values were superior than that reported for tapioca flour (4.9g H_2O $100g^{-1}$ d.b.) (CHISTÉ et al., 2012).

Modelling of sorption isotherms

For adsorption, all the tested models displayed suitability for predicting the isotherm for both products (Table 3) with R^2 values ranging from 0.94 to 0.99. However, the lowest P values (lower than 10%) for cassava flour from dry group were observed for the models of Oswin (4.5%), Halsey (5.6%), GAB (5.7%) and Smith (8.4%), and for cassava flour from water group, only Halsey, GAB and BET exhibited P values lower than 10% (3.4%, 5.1% and 9.6%, respectively). These models were also reported to be suitable to fit the adsorption

Figure 2 - Sorption isotherms (a) and Halsey and GAB models fitted to the adsorption (b) and desorption (c) isotherms of cassava flour from water group at 25°C.

isotherm of ten hydrophobically modified cassava starches (COVA et al., 2010) and for tapioca flour (CHISTÉ et al., 2012).

For desorption, all tested models were also suitable for predicting the isotherm of both cassava flour with R^2 ranging from 0.85 to 0.99. For cassava flour from dry group, the lowest P values were observed for Handerson (4.8%), GAB (6.9%), Oswin (8.2%) and BET (9.9%) and only Oswin, Halsey, GAB and Smith models presented P values lower than 10 % (4.9%, 5.6%, 6.7% and 7.7%, respectively) for the water group (Table 3). All the tested models in this study have been reported to fit the moisture sorption behaviour of starch foods quite well (PENG et al., 2007; CHISTÉ et al., 2012).

The models of Oswin or GAB seems to be the best mathematical equations to perform the simultaneous prediction of the sorption isotherms of cassava flour from dry group (Figure 1b and c), as well as the models of Halsey or GAB for cassava flour from water group (Figure 2b and c). Thus, it is strong recommend the use of GAB equation to predict efficiently the sorption isotherms (adsorption and desorption) for cassava flour from dry and water groups at the entire range of tested a_w.

CONCLUSION

The hygroscopic behaviour of cassava flour from dry and water groups at 25°C showed that both flour presented type II isotherms and a type-H3 hysteresis loop between adsorption and desorption isotherms. According to the adsorption isotherms, the moisture content of both cassava flour should not be higher than 11.3% to ensure the theoretical microbiological stability of the products

Table 3 - Parameters of mathematical modelling of sorption isotherms of cassava flour from dry and water groups at 25°C.

	Mathematical models	--------------Cassava flour from dry group---------------			-------------Cassava flour from water group-------------		
		Equation parameters	R²	P (%)	Equation parameters	R²	P (%)
Adsorption	Halsey	a = 36.06; b = 1.76	0.99	5.6	a = 25.65; b = 1.60	0.99	3.4
	Handerson	a = 0.05; b = 1.14	0.96	17.8	a = 0.07; b = 1.01	0.95	24.6
	Kuhn	a = 2.10; b = 5.93	0.94	21.6	a = 2.61; b = 5.36	0.98	13.9
	Mizrahi	a = -6.79; b = -4.68	0.94	22.8	a = -6.42; b = -3.79	0.98	15.2
	Oswin	a = 9.62; b = 0.48	0.99	4.5	a = 9.73; b = 0.53	0.99	11.1
	Smith	a = 2.21; b = 10.96	0.98	8.4	a = 1.71; b = 12.48	0.96	15.4
	BET	m_o = 4.07; c = 24,146.07; n = 19.81	0.97	11.7	m_o = 4.17; c = 3,699.70; n = 23.37	0.98	9.6
	GAB	m_o = 5.06; c = 31.61; k = 0.91	0.99	5.7	m_o = 4.90; c = 70.86; k = 0.94	0.99	5.1
Desorption	Halsey	a = 230.06; b = 2.26	0.97	14.9	a = 118.06; b = 2.02	0.99	5.6
	Handerson	a = 0.01; b = 1.57	0.99	4.8	a = 0.02; b = 1.37	0.96	12.8
	Kuhn	a = 1.95; b = 9.65	0.86	30.1	a = 2.40; b = 8.96	0.95	17.2
	Mizrahi	a = -10.46; b = -8.50	0.85	30.9	a = -9.97; b = -7.55	0.94	17.9
	Oswin	a = 13.50; b = 0.37	0.99	8.2	a = 13.10; b = 0.41	0.99	4.9
	Smith	a = 5.44; b = 10.93	0.98	10.7	a = 4.67; b = 12.05	0.98	7.7
	BET	m_o = 6.86; c = 23,07; n = 9.48	0.96	9.9	m_o = 5.78; c = 9,428.90; n = 13.84	0.96	12.1
	GAB	m_o = 9.00; c = 12.86; k = 0.79	0.98	6.9	m_o = 7.22; c = 44.17; k = 0.87	0.99	6.7

The a_w range in adsorption was 0.07 – 0.93 and 0.93 – 0.08 for desorption; R² = coefficient of determination; P = mean relative deviation modulus.

during storage at 25°C. Finally, GAB equation can be highlighted to be able to predict the sorption isotherms for both cassava flour.

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