

Moisture sorption isotherms of raw and extruded wholemeal sorghum flours studied by the dynamic and salt slurry methods

Isotermas de sorção de farinhas integrais cruas e extrudadas estudadas pelos métodos dinâmico e de solução saturada

Melicia Cintia Galdeano¹, Renata Valeriano Tonon¹, Carlos Wanderlei Piler de Carvalho^{1*} ,
Neuri Santos Menezes¹, Regina Isabel Nogueira¹, William Ferreira Leal-Junior¹, Adriana Paula Silva Minguita¹

¹ Embrapa Agroindústria de Alimentos, Rio de Janeiro/RJ - Brasil

*Corresponding Author:

Carlos Wanderlei Piler de Carvalho, Embrapa Agroindústria de Alimentos, Av. das Américas, 29501, CEP: 23020-470, Rio de Janeiro/RJ - Brasil, e-mail: carlos.piler@embrapa.br

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Abstract

The saturated salt slurry method has been a standard method for many years, but there are several drawbacks, including long equilibration times, extensive labour and mould growth at high water activity (aw) values. The validity of the dynamic method (automated sorption instrument) was investigated by comparing it with the equilibrium isotherms obtained using the saturated salt slurry method for two food materials (whole sorghum raw flour and whole grain sorghum extrudates), as well as their diffusion coefficients. The sorption isotherms ranged between 11.3% and 84.3%. The equilibration time for the highest water activity (0.843) was about 30 days using the saturated salts method, considerably longer than using the dynamic method (300 min). For raw sorghum flour, the dynamic method values were consistent with the data obtained using the traditional method, which can be confirmed by the similar curves and monolayer values obtained (0.038 and 0.040 g water / g solids for the salt slurry and dynamic methods, respectively). For the sorghum extrudates, the equilibrium moisture values were higher using the dynamic method, which could be related to the low diffusion coefficient of the material (order of 10⁻¹⁰). The authors suggest the use of the dynamic method as a better alternative to the saturated salt method in order to determine the sorption isotherms of whole grain sorghum-based food materials.

Keywords: Automated sorption instrument; Cereal flour storage; Diffusion coefficient; Thermoplastic extrusion; Sorption isotherm; Moisture equilibrium.

Resumo

O método de solução saturada tem sido um método padrão por muitos anos, mas existem várias desvantagens, incluindo longos períodos de equilíbrio, trabalho extensivo e crescimento de fungo em valores altos de atividade de água (aw). Foi investigada a validade do método dinâmico (instrumento de sorção automatizado), comparando-o às isotermas de equilíbrio solução saturada de dois alimentos (farinha crua e processada por extrusão de sorgo inteiro), bem como seus coeficientes de difusão. As isotermas de sorção variaram entre 11,3% e 84,3%. O tempo de equilíbrio para a maior atividade de água (0,843) foi de cerca de 30 dias para o método de solução saturada, consideravelmente mais longo que o método dinâmico (300 min). Para a farinha de sorgo crua, os valores do método dinâmico foram consistentes com os dados obtidos a partir do método tradicional, que podem ser confirmados pelas curvas e pelos valores monocamadas semelhantes (0,038 e 0,040 g de sólidos de água/g, para suspensão de sal e métodos dinâmicos, respectivamente). Para a farinha de extrudados de sorgo, os valores de umidade de equilíbrio foram maiores usando o método dinâmico, o que pode estar relacionado ao baixo coeficiente de difusão do material (ordem para 10⁻¹⁰). Sugere-se o uso de um método dinâmico como uma melhor alternativa ao método da solução saturada, a fim de determinar as isotermas de sorção de materiais alimentares à base de sorgo integral.

Palavras-chave: Instrumento de sorção automatizado; Armazenamento de farinhas de cereais; Coeficiente de difusão; Extrusão termoplástica; Isoterma de sorção; Equilíbrio de umidade.



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1 Introduction

The relationship between the water activity (a_w) and moisture content of a material at a given temperature is called the moisture sorption isotherm. This relationship is complex and unique for each product. Sorption isotherms are sigmoidal in shape for most foods, although isotherm curves of foods that contain large amounts of sugar have a J-type shape (FENNEMA, 1996). Sorption isotherms can be divided into three zones. Zone I ($a_w < 0.25$) represents the monolayer water, which is strongly associated with the food material, unable to freeze and not easily removed by drying. Zone II ($0.25 < a_w < 0.75$) represents the water that is adsorbed or absorbed in multilayers within foods and solutions of soluble components. It is slightly less mobile than bulk water. As water is added at the low-moisture end of this region, it exerts a significant plasticizing action on the solutes, lowering their glass transition temperatures and causing swelling of the solid matrix. Zone III ($a_w > 0.75$) represents the “free” water, which is freezable and easily removed by drying. This water is available for microbial growth and enzymatic activity (FENNEMA, 1996).

The methods used to determine sorption isotherms can be divided into static and dynamic techniques. The static gravimetric method, also called the traditional method, is described in both the European (ISO, 2013) and American (ASTM, 2010) standards (GARBALIŃSKA et al., 2017). In this method, dried (desorption), hydrated (adsorption) or native (working) samples are placed in closed chambers (desiccators) where saturated salt slurries provide different relative humidity (RH) values at constant temperature. The mass is weighed at specific intervals until equilibrium is achieved (LABUZA; ALTUNAKAR, 2007; GARBALIŃSKA et al., 2017). Since each saturated salt slurry provides only one RH value, different RH values are obtained by choosing a series of salts. When a material is placed in a desiccator, it gains or loses moisture until reaching equilibrium with the saturated slurry, and after this time no further weight change is observed. The moisture content, calculated based on the final sample weight, is referred to as the equilibrium moisture content (PENNER, 2013).

This method has the advantage of producing good results as well as allowing many samples to be measured simultaneously at low cost (PENNER, 2013). However, it presents some limitations, such as: 1) long period of time to reach equilibrium, 2) great variation in the measurements due to the need to frequently remove samples from the chambers for weighing, 3) conditions inside the desiccators are disrupted by the periodic openings to take measurements, and 4) mould growth may occur on samples at high RH values during long equilibration times (LEWICKI; POMARANSKA-LAZUKA, 2003; BOHN et al., 2005).

Thus the dynamic method could overcome the disadvantages found in the saturated salt slurry method.

Relatively new in the food industry, dynamic systems have been used in the pharmaceutical industry since their introduction in 1994. Commercially available equipment for dynamic analysis include the Dynamic Vapour Sorption (DVS) (Surface Measurement Systems Ltd., UK), the SGA-100 Symmetric Gravimetric Analyser (VTI Corporation, USA), the Cisorp Water Sorption Analyser (C.I. Electronics Ltd., UK), the Q5000SA Sorption Analyser (TA Instruments, USA) and the AquaSorp (Decagon Devices, USA) (TEOH et al., 2001; YU et al., 2008). However, the data from the latter instrument cannot be compared with the water sorption from the conventional water sorption method because its principle includes the generation of an airflow that continuously passes over the sample for a determined time, resulting in controllable changes of water activity (NURHADI; ROOS, 2016).

The dynamic analyses do not require the use of saturated salt solutions in order to provide the desired RH, but instead use mixtures of dry nitrogen and saturated water vapour, whose proportions are precisely controlled by mass flow controllers, to produce any desired water activity environment in the chamber. Due to the high precision and sensitivity, the automated instruments require very small amounts of sample. Thus, due to the sample size, small chamber and continuous flow of dry nitrogen and saturated water vapour mixture over the sample, equilibrium is reached faster, which avoids the risk of mould growth at high RH. In addition, the instruments maintain the RH and temperature conditions of the material throughout the duration of the experiment. However, the best advantage of the instrumental methods is related to the time of analysis. The instruments provide faster results compared to the traditional technique, besides being less laborious (YU, 2007; YU et al., 2008; BINGOL et al., 2012; PENNER, 2013).

Although the use of automated sorption instruments is becoming standard practice in many industries, there are few studies comparing the traditional salt slurry and dynamic methods (TEOH et al., 2001; ARLABOSSE et al., 2003; YU, 2007; BINGOL et al., 2012). Therefore, considering the above-mentioned advantages of the instrumental method over the traditional one and the lack of studies in the literature comparing both methods to obtain sorption isotherms for different materials, the aim of this study was to compare the salt slurry and dynamic methods to obtain sorption isotherms for two whole grain sorghum-based food materials.

2 Materials and methods

2.1 Materials

Two materials were selected for use in this study: raw sorghum flour (92.33 μm) and sorghum extrudates (3.43 \pm 0.49 mm diameter). Sorghum grains (genotype SC319), provided by Embrapa Milho e Sorgo (Sete Lagoas, MG), were ground using a model LM3600 disc mill

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(Perten Instruments AB, Huddinge, Sweden), resulting in raw whole grain sorghum flour. The raw whole grain sorghum flour was extruded in a model Evolum HT 25 co-rotating twin-screw extruder (Clextral, Firminy, France). The screw speed (600 rpm), moisture content (14%) and temperature profile (30, 30, 60, 90, 100, 100, 120, 120, 150 and 150 °C) were maintained constant and the die had four holes, each 3.8 mm in diameter. The extrudates were collected, placed in trays and dried in a fan oven at 60 °C for 4 h. After drying, they were stored in plastic bags at 25 °C (± 2 °C) until further analysis.

2.2 Sorption isotherms

2.1.1 Traditional saturated salt slurry method

The sorption isotherms were first determined by the gravimetric method according to Yu (2007). Eight saturated salt slurries were prepared at 60 °C (LiCl, CH₃COOK, MgCl₂, K₂CO₃, Mg(NO₃)₂, KI, NaCl and KCl) according to Nyqvist (1983), in order to provide relative humidity values of 11.3%, 22.6%, 32.8%, 43.2%, 52.9%, 68.9%, 75.3% and 84.3%, respectively (GREENSPAN, 1977). All the salts were acquired from Sigma-Aldrich and the water activity of each saturated solution was verified at 25 °C using the AquaLab Lite equipment (Decagon, Pullman, USA). Prior to analysis, the samples were dried in a desiccator with anhydrous calcium chloride for 20 days according to Galdeano et al. (2009). Triplicate 1 g samples were equilibrated over the saturated slurries in 2.4 L borosilicate glass desiccators with 160 mm diameters placed in controlled temperature model HY350U BOD incubators (Hydrosan, Belo Horizonte, MG) at 25 \pm 2 °C. Each sample was weighed on a model AUX 320 balance (Shimadzu, Tokyo, Japan) at regular intervals for seven days until equilibrium was achieved, which was considered to be when the change in sample weight did not exceed 0.1%. The equilibrium moisture content was calculated from the increase in mass of the samples after equilibration at a given RH, following the methodology described by Ascheri et al. (2006). The data were also fitted to the modified BET model as described below.

2.1.2 Dynamic method

The experiments were carried out using a Q5000SA Sorption Analyser (TA Instruments, New Castle, USA), equipped with a high-accuracy microbalance (0.1 mg \pm 1%). The accuracy of the system was $\pm 1\%$ for the RH over a range from 0 to 98% and ± 0.1 °C for the temperature stability. To take a measurement, the samples (3 to 5 mg) were first dried in the instrument at 60 °C and 0% RH, and then subjected to the same RH values used in the traditional method (11.3, 22.6, 32.8, 43.2, 52.9, 68.9, 75.3 and 84.3%). The RH was increased step by step during adsorption and the samples were maintained at each humidity level until no further weight change was detected. The time required

to reach equilibrium moisture varied for each RH and each material. The mass of the sample was determined continuously during the test. All experiments were carried out in triplicate at 25 °C with a total gas flow of 200 mL/min and the data were also fitted to the modified BET model as described below.

2.1.3 Mathematical modelling

One of the equations most widely used to describe sorption isotherms is that of the Brunauer-Emmett-Teller (BET) model (Equation 1), since it has a theoretical background and the parameters provide a physical meaning related to the sorption process, different from the empirical models. The BET model is considered to be an effective method for estimating the amount of bound water at specific polar sites in dehydrated food systems (SAHIN; GÜLÜM, 2006).

$$X_e = \frac{X_m C_{BET} a_w}{(1 - a_w)(1 - a_w + C_{BET} a_w)} \quad (1)$$

However, the BET model is not adequate for water activities above 0.5 (JONQUIÈRES; FANE, 1998; GOULA et al., 2008), and is unable to accurately predict the sorption behaviour in such cases (Equation 1). In their original publication, Brunauer et al. (1938) derived a modified model with three parameters, considering a limited number of adsorbed layers, which allowed for the modelling with water activities of up to 0.9 (Equation 2).

$$X_e = \frac{X_m C_{BET} a_w [1 - (n + 1)(a_w)^n + n(a_w)^{n+1}]}{(1 - a_w)[1 + (C_{BET} - 1)a_w - C_{BET}(a_w)^{n+1}]} \quad (2)$$

Where: X_e is the equilibrium moisture content at a given water activity (a_w), X_m is the monolayer value (g water/g solids) and C_{BET} and n are model constants.

Thus, in the present work, the sorption isotherms data were modelled according to the modified BET model (Equation 2), using the Solver algorithm of Microsoft Excel (Microsoft, Redmond, USA).

2.1.4 Diffusion coefficient

The effective diffusion coefficients (D_{eff}) of water into raw sorghum flour and into sorghum extrudates were determined according to Fick's second law, applied to an infinite slab and to a sphere, respectively. The analytical solutions of the diffusion equation for these two cases, as described by Crank (1979), are expressed by Equations 3 and 4.

$$\left(\frac{X_t - X_e}{X_0 - X_e} \right) = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp \left[-(2n-1)^2 \frac{\pi^2 D_{eff} t}{L^2} \right] \quad (3)$$

$$\left(\frac{X_t - X_e}{X_0 - X_e} \right) = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp \left[-n^2 \frac{\pi^2 D_{eff} t}{r^2} \right] \quad (4)$$

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Where: X_t = the moisture content at time t , X_0 = the initial moisture content, X_e = the equilibrium moisture content, D_{eff} = effective diffusion coefficient for water ($\text{m}^2 \text{s}^{-1}$), L = thickness of the sorghum flour layer (m), r = extrudate radius and n = number of terms in the series.

Eighteen terms were used to determine the moisture diffusivity ($n = 18$) and "shrinkage" of the monolayer was neglected, i.e., L and r were considered constant ($L = 0.001 \text{ m}$ and $r = 0.0017 \text{ m}$) throughout the sorption isotherms determination.

3 Results and discussion

3.1 Moisture equilibration time

Water adsorption is characterized by an increase in sample mass after changing the RH conditions, tending to reach a constant value (equilibration conditions) after a certain time (ROMAN-GUTIERREZ et al., 2002). In the present study, the time needed to reach these apparent equilibration conditions (plateau) using the automated instrumental method ranged, for each RH, from 120 to 210 min for the raw flour and from 130 to 300 min for the extrudates (Figure 1). The equilibration time for the highest water activity (0.84) was about 30 days for the saturated salts method, considerably longer than for the dynamic method at the same RH (300 min).

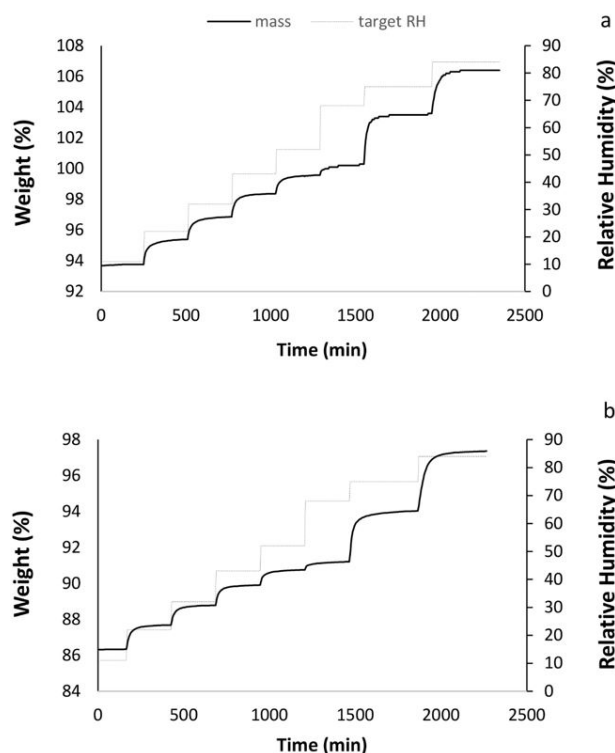


Figure 1. Time to reach the equilibrium moisture content at different RH levels for (a) raw sorghum flour and (b) sorghum extrudates.

Compared to the weeks of equilibration time reported in the literature for the static method for most foods, the automated sorption instrument has the advantage of reaching the equilibrium conditions much quicker. These low equilibration times could be associated with the very small sample sizes (3 to 5 mg) and the continuous flow of nitrogen and water vapour mixture over the product. In the present work, eight RH levels were used, and the time required to obtain the complete sorption isotherm was approximately two days. Similar equilibration time ranges were reported in other studies for rice grains and wheat flour (ROMAN-GUTIERREZ et al., 2002; BINGOL et al., 2012).

These rapid equilibration times also have the advantage of obtaining isothermal points at high water activities without concern about microbial degradation, as well as the increase in isothermal modelling accuracy at higher relative humidity values (BOHN et al., 2005).

3.2 Sorption isotherms

Figure 2 shows the raw sorghum flour and sorghum extrudates sorption isotherms obtained by the traditional and dynamic methods, while the BET model parameters are shown in Table 1.

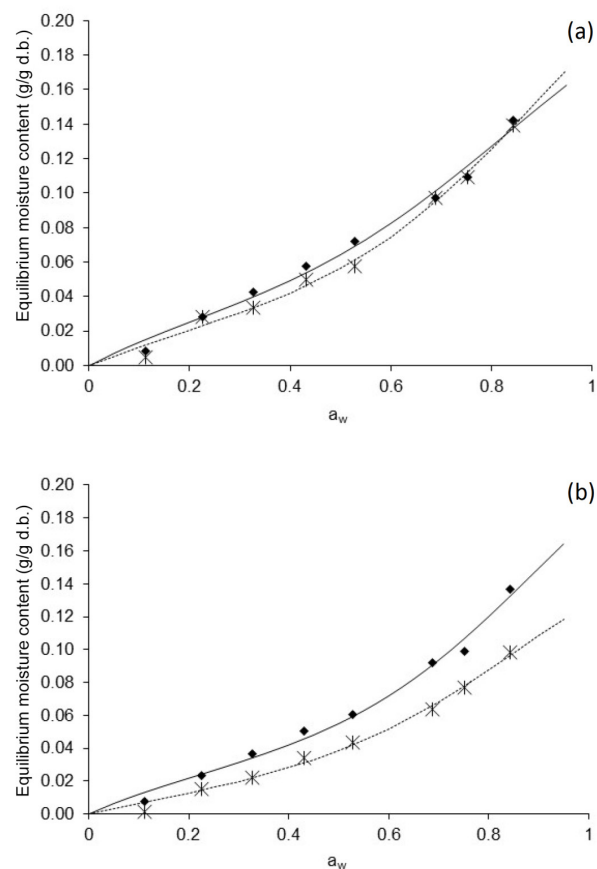


Figure 2. Sorption isotherms for raw sorghum flour (a) and sorghum extrudates (b). The lines were derived from the BET model. (a) Salt slurry and (b) dynamic methods.

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Table 1. Estimated BET parameters for the raw sorghum flour and sorghum extrudates using the saturated salt slurries and dynamic methods of analysis.

Product	Method	BET parameters			
		X_m	C	n	R^2
Raw sorghum flour	Salt slurry	0.038	3.199	8.589	0.993
	Dynamic	0.040	3.813	7.679	0.989
Sorghum extrudates	Salt slurry	0.028	2.293	9.279	0.993
	Dynamic	0.034	4.150	9.656	0.989

X_m = monolayer value (g water/g solids); C and n are BET constants.

Table 2. Effective diffusion coefficients (D_{eff}) of water into raw sorghum flour and sorghum extrudates at different water activities (aw).

Product	D_{eff} ($m^2 s^{-1}$)		
	aw 0.226	aw 0.529	aw 0.843
Raw sorghum flour	1.6×10^{-10}	1.5×10^{-10}	1.6×10^{-10}
Sorghum extrudates	7.3×10^{-10}	5.1×10^{-10}	6.4×10^{-10}

According to the Brunauer classification (BRUNAUER et al., 1938), the sorption isotherms exhibited a type II sigmoid shape. Type II isotherms represent the sigmoidal curve that describes the majority of processed foods, since they are typical of intermediate moisture materials.

For raw sorghum flour, the dynamic method values were consistent with the data obtained using the traditional method, which can be confirmed by the similar curves observed in Figure 2a. The agreement between the methods was good for the whole range of water activity, as also observed by Arlabosse et al. (2003).

Using the static method, the raw flour showed a moisture content of 0.140 g of water/g of dry matter when submitted to a RH of 84.3%, and 0.143 g of water/g of dry matter using the dynamic method (data not shown). The BET equation yielded similar monolayer values (X_m) when comparing the results obtained using the two methods of analysis (Table 1). The BET model is based on the monolayer moisture concept and provides the value for the monolayer moisture content of the material (X_m) which is considered safe for dried foods during storage, whereas most other models fail to provide this parameter. The monolayer moisture content (X_m) indicates the water that is strongly adsorbed to specific sites on the food surface, and is considered to be an important value to guarantee food stability. In order to maintain the food microbiologically safe during storage, based on the static method the raw sorghum flour should not contain more than 3.8 g of water/g of dry matter, whereas for the sorghum extrudates this value should be even lower, 2.8 g of water/g of dry matter. The X_m values varied from 0.028 to 0.040 g water/g solids, similar to the values observed by Tonon et al. (2009) and Purohit and Rao (2017).

For sorghum extrudates, the equilibrium moisture values obtained at all RH values were higher when using the dynamic method. The reproducibility between the two experimental methods was only good for water activities below 0.2. For higher activity values, a significant difference appeared. Teoh et al. (2001) observed similar behaviour for the sorption isotherms of extruded cornmeal; there was a difference between the curves for all the RH values studied and the dynamic method showed higher equilibrium moisture content values.

The similarities and differences observed between the methods could be explained by the diffusion coefficient of the material (ARLABOSSE et al., 2003). According to these authors, when the diffusion coefficient is low ($< 10^{-9} m^2 s^{-1}$), thermodynamic equilibrium is difficult to reach using the saturated salts method. However, if the apparent diffusion coefficient is above $10^{-9} m^2 s^{-1}$, internal diffusion is not limiting enough to induce a discrepancy between the static and dynamic methods. Thus a comparison between the static and dynamic methods shows consistent results as long as the diffusion coefficient of the material is high. Hence the effective diffusion coefficients (D_{eff}) of water into the sorghum flour and into the sorghum extrudates were calculated for three water activities (0.226, 0.529 and 0.843) and the results are shown in Table 2.

The estimated diffusion coefficients for the extrudates were 7.3, 5.1 and $6.4 \times 10^{-10} m^2 s^{-1}$ for water activities of 0.226, 0.529 and 0.849, respectively. Thus when comparing the methods, the difference found for the extrudates could be related to the low diffusion coefficient of the material. Since the diffusion coefficients were low (order of 10^{-10}) in the present study, the use of a dynamic method to determine the sorption isotherms is suggested as a better alternative to the saturated salt method.

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Using the static method, the extrudates presented a moisture content of 0.098 g of water/g of dry matter when submitted to a RH of 84%, whereas using the dynamic method it increased to 0.137 g of water/g of dry matter under the same RH value (data not shown). Figure 2b shows greater hygroscopicity for the sorghum extrudate evaluated by the dynamic method, as compared to the salt slurry method, which is in agreement with the higher monolayer values calculated by the BET model (Table 1).

For both samples, considering the time required to determine the complete sorption isotherm, equilibrium was reached after 30 days using the saturated salts method and around 30 hours when using the dynamic method it took.

4 Conclusion

The time required to reach the apparent equilibration conditions using the dynamic method was considerably lower than when using the salt slurry method. As a result, a sorption isotherm could be obtained in a few days using the automated sorption instrument, as compared to weeks using the saturated salts method. A comparison between the methods showed consistent results for the raw flour but some differences for the extrudates. The choice of one or the other method to determine the sorption isotherm is not easy. The saturated salts method involves low investment costs and is easy to implement, but the equilibrium criterion is difficult to define and the delay in equilibration can be very long. Using the dynamic method, the experimental time to produce a complete isotherm is much shorter, but the apparatus is expensive and requires some training in order to operate it, besides needing frequent maintenance. Thus a careful analysis should be carried before choosing the method, considering costs, available time and frequency of use.

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