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Influence of milling and extrusion on the sorption properties of sorghum

Influência dos processamentos de moagem e de extrusão nas propriedades de sorção do sorgo

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Abstract

The processing to which a material is subjected can affect its physical or chemical structures, resulting in products with different hygroscopic behaviours. The present work studied the water adsorption properties of sorghum subjected to different types of processing: raw flour (produced by milling whole grain), extrudates (obtained using a double screw extruder) and extruded flour (obtained by milling the extrudates). The isotherms were obtained using an automated instrumental method. The tests were run in duplicate at 25 °C with relative humidity values ranging between 11 and 84%. The water adsorption data fitted the GAB model well, showing high coefficients of determination. The estimated water contents of the adsorption monolayer ranged from 5.3 to 6.9 g of water per 100 g of dry material. The sorption isotherms were affected by the type of processing, extrusion cooking resulting in products with less water in the monolayer (less hygroscopic). The milling process yielded high water contents in the monolayer, probably due to the breakdown of some polymer-polymer interactions, which exposed the binding sites. To ensure microbiological stability, the water contents in the materials should not exceed 6.9 g of water per 100 g of dry material for raw sorghum flour, 5.3 g of water per 100 g for sorghum extrudates and 6.7 g of water per 100 g for extruded sorghum flour.

Keywords: *Extruded flour; Extrudates; Raw flour; Sorghum bicolor L.; Sorption isotherms.*

Resumo

O processamento ao qual um material é submetido pode afetar sua estrutura física ou química, resultando em produtos com diferentes comportamentos higroscópicos. No presente trabalho, objetivou-se estudar as propriedades de adsorção de água do sorgo em diferentes formas de processamento: farinha crua (produzida pela moagem de grãos inteiros), extrusados (produzidos em extrusora de duplo parafuso) e farinha extrusada (obtida pela moagem dos extrudados). As isotermas foram obtidas usando o método instrumental automatizado. Os testes foram realizados em duplicata, a 25 °C, com valores de umidade relativa variando entre 11 e 84%. Os dados de adsorção de água foram bem ajustados ao modelo de GAB, mostrando altos coeficientes de determinação. O teor estimado de água da monocamada variou de 5,3 a 6,9 g de água por 100 g de material seco. As isotermas de sorção foram afetadas pelo tipo de processamento, sendo que o cozimento por extrusão resultou em produtos com menor valor de monocamada (menos higroscópico). O processo de moagem produziu valores de monocamada elevados, provavelmente devidos à degradação de algumas interações polímero-polímero, que expuseram sítios de ligação. Para garantir a estabilidade microbiológica, o teor de água nos materiais não deve ser superior a 6,9 g de água por 100 g de material seco para a farinha crua de sorgo; 5,3 g de água por 100 g para os extrusados de sorgo, e 6,7 g de água por 100 g para a farinha extrusada.

Palavras-chave: *Farinha extrusada; Extrusado; Farinha crua; Sorghum bicolor L.; Isotermas de sorção.*

1 Introduction

The interaction of water vapour (moisture) with solid materials has an impact on a wide range of industries, including the pharmaceutical (ÖZENGIN; ELMACI, 2016), food (FENNEMA; REIDE, 2010) and polymer (ANSELL, 2015) industries. This interaction is measured by means of the sorption isotherms.

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. The moisture sorption isotherm curve (moisture content *versus* water activity) is generated from adsorption or desorption processes and is obtained from the equilibrium moisture contents determined at several aw values (LABUZA; ALTUNAKAR, 2007).

All foods have their own characteristic moisture sorption isotherm, which can be useful for a variety of processing and product stability research projects, ranging from studies on the inhibition of microbial growth to those involving the chemical and physical stability of foods, besides providing help when formulating food mixtures. The Isotherms are also important to determine the moisture barrier properties required by packaging materials to limit moisture gain or loss in a package (DAMODARAN et al., 2008; ALVES et al., 2015).

Processing changes the chemical and physical structures of foods (CHESSON et al., 2002) and consequently affects their sorption isotherms (RAMANATHAN; CENKOWSKI, 1995). Extrusion cooking and milling are processes widely used in the food industry that can also affect water mobility and affinity, and hence the moisture sorption of the materials due to modifications that occur in the starch and in other constituents present, such as dietary fibre (CHÁVEZ et al., 2017). Previous studies have examined the effect of different processes on the isotherms of products (VALDEZ-NIEBLA et al., 1993; RAMANATHAN; CENKOWSKI, 1995; JAMROZ et al., 1999; SUKAINAH et al., 2013).

The traditional method used to determine food sorption isotherms using saturated salt slurries of known relative humidity values inside closed chambers (desiccators), produces good results, as well as allowing many samples to be measured simultaneously at low cost (PENNER, 2013). However, it presents some limitations such as long periods of time to reach equilibrium, great variation in the measurements and mould growth on samples at high relative humidity values during long equilibration times (GARBALIŃSKA et al., 2017). In order to solve these drawbacks, some automated instrumental methods (also called dynamic methods) are being used in the food industry.

There are a number of advantages of these instruments as compared to the traditional saturated salt slurry method. The instruments provide faster results (equilibration time is about 10 to 100 times faster than the standard method), besides being less laborious (fully automated). In addition, the instruments maintain the relative humidity and temperature conditions of the material for the duration of the experiment. Furthermore, studies comparing the traditional salt slurry and the instrumental methods have shown good agreement between them (PENNER, 2013).

The aim of this work was to assess the effect of milling and extrusion on the moisture sorption isotherms of sorghum products.

2 Material and methods

2.1 Material

Sorghum grains (genotype SC319), provided by *Embrapa Milho e Sorgo* (Sete Lagoas, MG, Brazil) were milled using a model LM3600 disc mill (Perten Instruments AB, Huddinge, Sweden) to obtain the raw sorghum flour.

2.2 Sorghum extrudate production

The raw sorghum flour was extruded in an Evolum HT 25 co-rotating twin-screw extruder (Clextral, Firminy, France) with a screw diameter of 25 mm, length:diameter ratio of 40:1 and ten temperature zones. The screw speed (600 rpm), moisture content (14%) and temperature profile (30, 30, 60, 90, 100, 100, 120, 120, 150 and 150 °C) were kept constant. The die had four holes, each 3.8 mm in diameter. The extrudates were collected, placed in trays and dried in a fan oven (60 °C for 4 h). After drying, they were stored in plastic pots at 25 °C (± 2 °C) until further analysis.

2.3 Extruded sorghum flour production

Part of the sorghum extrudates were milled in a model LM3600 disc mill (Perten Instruments AB, Huddinge, Sweden) to yield the extruded sorghum flour. The material was then stored in a plastic pot at 25 °C until analysed.

2.4 Sorption isotherms using the instrumental 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 ±1%). The accuracy of the system was ±1% for the relative humidity (RH) over a range from 0 to 98% and ±0.1 °C for temperature stability. During a measurement, the samples (3 to 5 mg) were dried in the instrument at 60 °C and 0% RH and exposed to a series of relative humidity values (11, 22, 32, 42, 52, 57, 75 and 84%) at constant temperature. The RH was increased step by step during adsorption. The time required to reach equilibrium moisture was different for each RH and material. The mass of the sample was measured continuously during the test and the equilibrium moisture content calculated from the increase in mass after equilibration at a given RH. All experiments were carried out in quadruplicate at 25 °C with a gas flow of 200 mL min^{-1}. The GAB model was used to fit the sorption isotherm data and can be expressed as follows: $M=m_0CKa_w/(1-Ka_w)(1-Ka_w+CKa_w)$, where M is the equilibrium moisture content at a water activity of (a_w), m_0 is the monolayer value (g water/g solids) and C and K are the GAB constants.

2.5 Particle size distribution

The particle sizes of the raw and extruded flours were assessed by laser diffraction using a S3500 (Microtrac Inc., Montgomery Ville, USA). The samples were dispersed in isopropyl alcohol (refractive index of 1.377) and slowly added to the compartment of the equipment containing the same dispersant. The analyses were carried out in triplicate. The refractive index of the particles was 1.50 and the results were expressed as D[4,3], D(v 0.1), D (v 0.5) and D (v 0.9). For the extrudates, the medium diameter (mm) was measured using a digital micrometer.

2.6 Scanning Electron Microscopy (SEM)

The materials were allowed to dry in a desiccator containing anhydrous calcium chloride. The SEM analyses were carried ouy using a TM3000 scanning electron microscope (Hitachi, Tokyo, Japan) and all samples were examined under low vacuum with a 15 kV accelerating voltage.

2.7 X-ray diffraction

The X-ray diffraction profile was determined in a D2 Phaser X-ray diffractor (Bruker AXS, Rheinfelden, Germany) equipped with a copper tube and operating at 30 kV and 10 mA, producing CuKα radiation with a wavelength of 0.154 nm. The samples were scanned from 2 to 32° (2θ) at a rate of 0.15° min^{-1}, with a step size of 0.02°, a divergence slit width of 0.6 mm, a scatter slit width of 0.6 mm and a receiving slit width of 0.2 mm. The Diffrac.Suite EVA-XRD version 3.1 software (Bruker AXS, Rheinfelden, Germany) was used to analyse the diffractograms.

2.8 Statistical analysis

All the statistical analyses were carried out using the STATISTICA 7.0 (Statsoft, Tulsa, USA) software. The data was subjected to an analysis of variance (ANOVA), Tukey's test and the t-Student test ($p < 0.05$). The sorption isotherm data were fitted to the GAB model using the Solver algorithm of Microsoft Excel (Microsoft, Redmond, USA).

3 Results and discussion

The thermoplastic extrusion process consists of a high temperature/short time heat treatment (CHÁVEZ et al., 2017). This process promotes the disruption of the granular structure of starch, denaturing and re-directing proteins to form a final product with modified physical characteristics. Due to the changes in the raw material imposed by the extrusion cooking process, it is important to understand the water absorption properties (ASCHERI et al., 2016).

Dry cereal products are hygroscopic due to their chemical composition, porosity and the presence of starch in its amorphous state. For expanded products, where crunchiness is a determining factor, the phase changes that occur as a result of moisture adsorption can cause a loss of crunchiness, affecting the sensory quality (WANI; KUMAR, 2016).

The moisture equilibration times and the moisture sorption isotherms of the raw sorghum flour, sorghum extrudates and extruded sorghum flour are shown in Figures 1 and 2, respectively, and the parameters of the GAB model are presented in Table 1.

For each RH, the time necessary to reach these apparent equilibration conditions, ranged from 120 to about 210 min for raw flour, 130 to about 300 min for extrudates and 115 to about 230 min for extruded sorghum flour as determined using the dynamic instrumental method (Figure 1). The time taken to obtain the experimental sorption equilibrium data using the automated instrumental method was significantly reduced. To obtain the complete isotherm the run took approximately 2 days. Similar ranges in equilibration times (using the dynamic method) were also observed in other studies (ROMAN-GUTIERREZ et al., 2002; BINGOL et al., 2012).

The GAB model was chosen to fit the data, since it is the most used model, mainly for covering the isotherm in a greater range of water activity (between 0.05 and 0.9) (LABUZA; ALTUNAKAR, 2007). The sorption isotherms (Figure 2) presented the sigmoidal form of type II. According to the classification of the International Union of Pure and Applied Chemistry (IUPAC, 1985) the type II isotherm is typical of hydrophilic surfaces, such as products processed by thermoplastic extrusion (McMINN et al., 2007). The GAB model parameters are shown in Table 1 and the goodness of fit evaluated (high determination coefficient, R^2).

All the samples showed high moisture sorption (Figure 2), which is a typical behaviour of hydrophilic samples. Furthermore, an increase in equilibrium moisture content with increase in water activity at a constant temperature was observed, which could be due to the hydrophilic nature (carbohydrates and protein) of the components present.

It was observed that the type of processing to which the material was subjected influenced its sorption

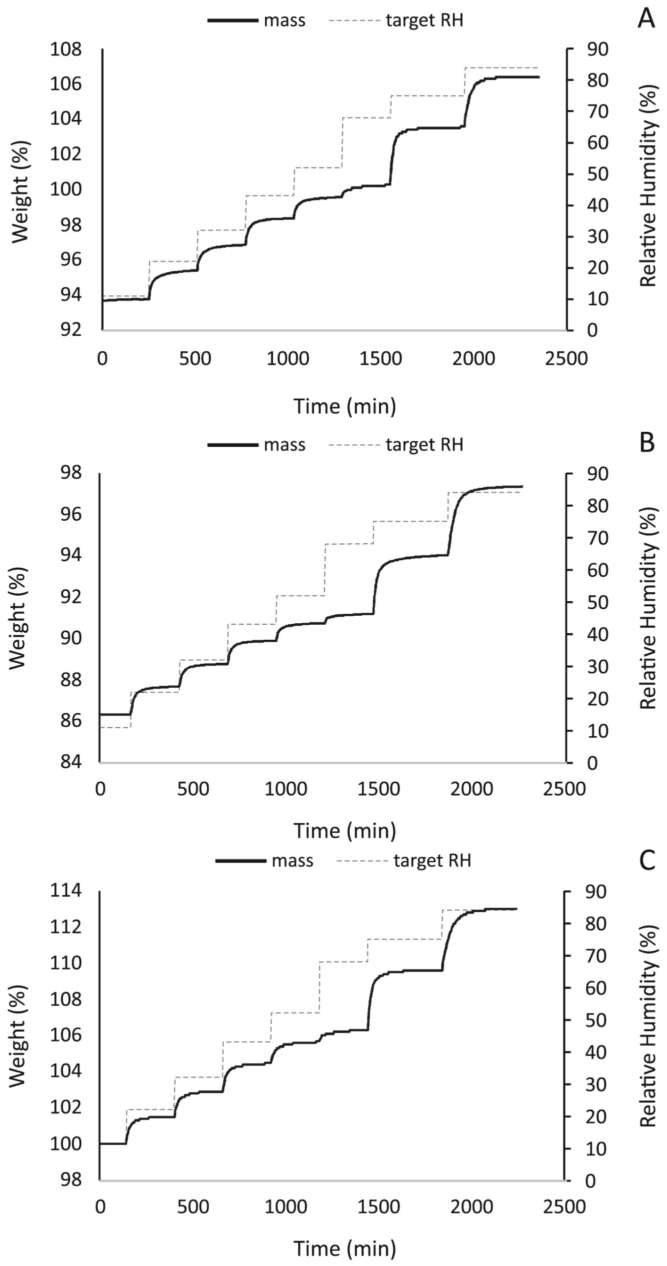

Figure 1. Equilibration time to reach equilibrium moisture at different RH levels. (A) raw sorghum flour, (B) sorghum extrudates and (C) extruded sorghum flour.

properties. Raw sorghum flour had the largest monolayer value (0.069 g water / g solids), followed by the extruded flour (0.067 g water / g solids) and the extrudates (0.053 g water / g solids). The value of the monolayer indicates the maximum amount of water that can be adsorbed by the primary layer and it is a measure of the sorption sites. In the monolayer, or below it, the water is tightly bound and unavailable for reactions, corresponding to the optimal moisture content for food preservation (CRUZ, 2000). The greater the number of active sites in the monolayer, the greater the hydrophilicity of the material, and therefore the greater the sensitivity to environmental changes.

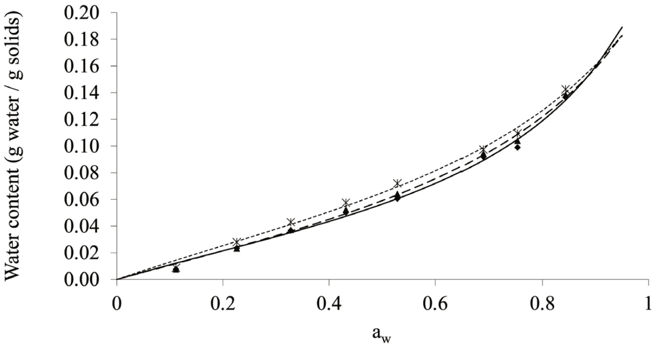

Figure 2. Sorption isotherms of the raw sorghum flour (Ж), sorghum extrudate (▲) and extruded sorghum flour (◆) - the lines were derived from the GAB model.

Table 1. The GAB model parameters for raw sorghum flour, sorghum extrudates and extruded sorghum flour.

Product	GAB parameters*			
	m_0	C	K	R^2
Raw sorghum flour	0.069^a	2.762^a	0.714^a	0.99
Sorghum extrudates	0.053^b	2.740^b	0.788^a	0.99
Extruded sorghum flour	0.067^c	2.192^c	0.730^a	1.00

*$M=m_0CK_{aw}/(1-K_{aw})(1-K_{aw}+CK_{aw})$, where M is the equilibrium moisture content at a determined water activity (aw), m_0 is the monolayer value (g water/g solids), and C and K are the constants. Means in the same column with different letters are significantly different (Tukey test, $p \leq 0.05$).

The smaller monolayer value of the extrudates as compared to the raw material could be explained by the probable complexation of molecules inside the extruder (polymer-polymer interactions), reducing their ability to interact with water. During extrusion, high temperatures and large shearing forces cause disruption of the granular starch structure and a deterioration of the quaternary and tertiary structures of the biopolymers, thus favouring interactions between food components and reducing the number of actives sites for water binding. Biopolymers are aligned according to the flow streamlines, and extra bonding and cross-linking occur (HARPER, 1986).

Besides, since the monolayer is influenced by the exposed sites of broken fibre components (cellulose and hemicelluloses) of greater water affinity, during extrusion these sites might not be available since the process may cause encapsulation of the fibre components by the broken starch molecules, as a consequence of high shearing. Although broken starch has a greater water affinity than raw starch, it is less prone to absorb water than the fibre components (HIETALA et al., 2013).

Wani and Kumar (2016) suggested that the formation of starch structures with different types and degrees of crystallinity, related to the partial or complete destruction of starch granules during the extrusion process, could also explain the smaller monolayer value of the expanded product.

Jamroz et al. (1999) found similar results to those found in the present work; where extruded potato starch yielded a smaller monolayer capacity than its native starch. According to the authors, high mechanical pressure in the extruder could reduce the porosity and moisture diffusivity of the material, explaining the difference between the samples.

Different behaviour was observed in other studies. Lima et al. (2012) reported that raw rice flour adsorbed less water than its extruded flour. In this case, the material was composed essentially of starch and protein since the meal had been removed. According to the authors, the molecular disorder caused by the extrusion cooking of the grain exposed a greater number of adsorption sites. This was because during the extrusion process, the high temperatures, mechanical shear and limited water content promoted starch breakdown, resulting in disruption of the molecular order and causing irreversible changes in their properties, making them soluble in cold water and increasing the availability of hygroscopic OH- groups, which interact with water through hydrogen bonding. They also added that for extruded flours, water adsorption occurred not only in the amorphous region, but also in the crystalline region, due to the molecular breakdown caused by the cooking process, that liberated free hydroxylates.

Compared with the extrudates, milled extrudates (extruded flour) showed an increase in the monolayer value. The milling process may cause the breakage of some polymer-polymer interactions (complexation), thus exposing more binding sites, which resulted in the higher monolayer value (CHESSON et al., 2002).

The other GAB constants are also important in the study of isotherms. Parameter C is related to the heat of sorption of the first layer in primary sites. This parameter determines the shape of the curvature in the lower water activity range. On the other hand, the constant *k* is related to the total heat of sorption of the multilayer, regulating the curvature after the plateau. Isotherms with lower *k* values are flatter in the higher water activity range (WANI; KUMAR, 2016). The raw flour had the highest C value, followed by the extruded flour and the extrudates, confirming that the higher the water adsorption capacity, the higher the heat of sorption (ALBARRACÍN et al., 2016). The *K* value was not affected by the different processes.

Particle size is an important parameter that may influence the water sorption of materials. The larger the particle diameter, the lower the contact area exposed, and thus the smaller the amount of water adsorbed. The results for particle size are presented in Table 2. The De Brouckere mean diameter (D4,3) and the Sauter mean diameter (D3,2) were lower for raw sorghum (92.33 and 30.22 µm, respectively), followed by the extruded flour (129.48 and 78.22 µm, respectively) and the extrudates (3.43 mm). The average diameter of the extrudates was about 30 times greater than that of the milled materials. Fifty percent of the raw and extruded flour particles [D (v 0.5)] were smaller than 65.63 µm and 116.29 µm, respectively. The raw and extruded flours, which presented smaller mean diameters, adsorbed more moisture from the environment and presented higher values for the monolayer moisture content.

Table 2. Particle sizes of the raw sorghum flour, sorghum extrudates and extruded sorghum flour.

Product	D[4,3][a] (µm)	D(v 0.1) (µm)	D(v 0.5) (µm)	D(v 0.9) (µm)
Raw sorghum flour	92.33^{a}	14.26^{a}	65.63^{a}	210.68^{a}
Extruded sorghum flour	129.48^{b}	41.43^{b}	116.29^{b}	235.35^{a}

[a]volume *mean diameter or* De Brouckere mean diameter; [b]surface mean diameter or Sauter mean diameter. Means in the same column with different letters are significantly different (t-Student test, $p \leq 0.05$).

The morphology of materials also exerts an influence on their water sorption behaviour. The smoother the surface of a particle, the smaller the contact area exposed, and consequently the lesser the amount of water adsorbed from the environment. Raw flour and extruded flour adsorbed more moisture when compared to the extruded product (Table 1). The milled samples showed particles with rougher surfaces (Figure 3) than the extruded sample, adsorbing more moisture from the environment and resulting in higher monolayer moisture values.

The relative crystallinity of materials may also help explain differences in their water sorption. According to the degree of crystallinity, materials have a greater or lesser affinity for water. Amorphous solids adsorb considerably more water than crystalline solids at low water activities whereas at high water activities both adsorb similar amounts of water (SLOAN; LABUZA, 1975). The extrudates had the highest relative crystallinity value (25.09%) while the raw flour presented the lowest value (21.68%), which is in agreement with their m_0-values (low for extruded material and high for raw material). The extruded flour, despite having a high m_0-value, presented similar relative crystallinity (25.04%) to the extruded material.

Extrusion is known to partially or completely destroy the crystalline structures of the material, and the degree of destruction depends on the process conditions (DONKOR et al., 2012). However, this behaviour was not observed in the present study. With respect to this aspect, Albarracín et al. (2016) postulated that higher relative crystallinity values in extruded materials may be due to the presence of some remaining native starch (which has a semicrystalline structure).

In the present study, the use of X-ray diffraction failed to explain the differences observed between the isotherms of the materials. The effects of particle size and

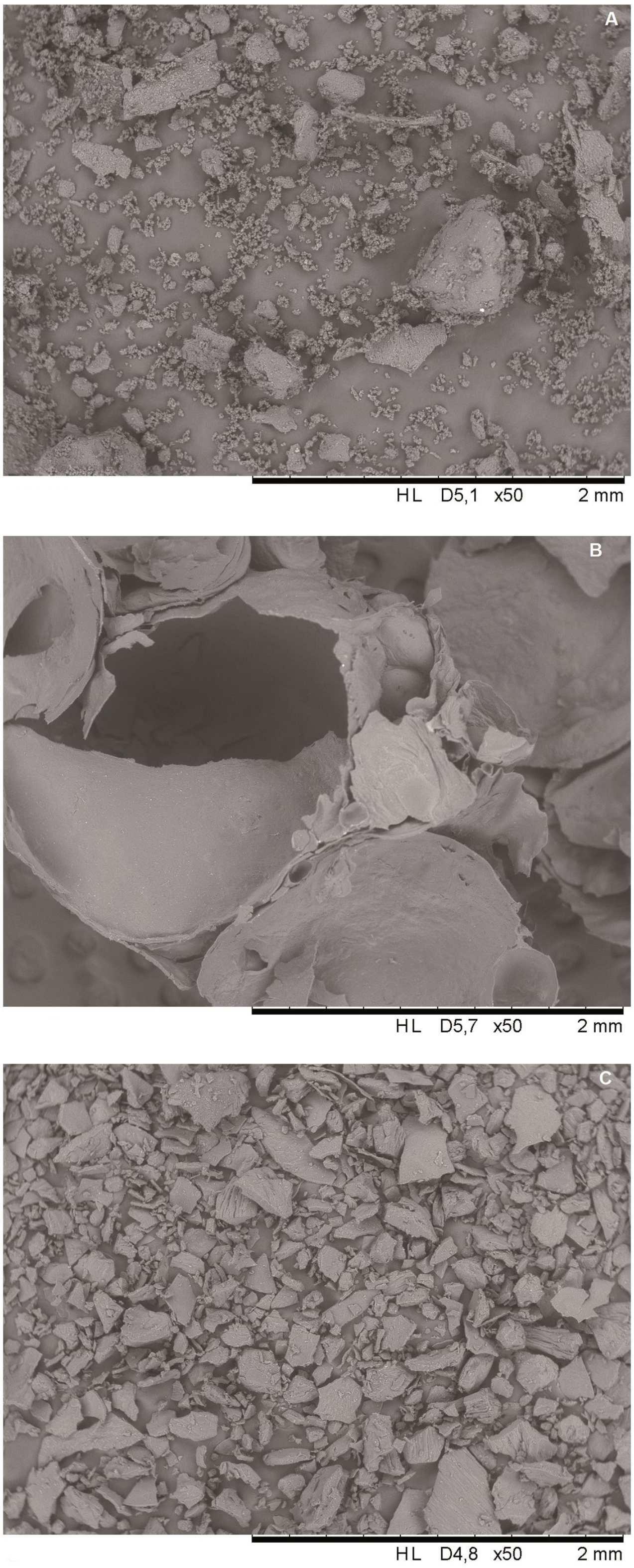

Figure 3. SEM micrographs of (A) raw sorghum flour; (B) sorghum extrudates; and (C) extruded sorghum flour (x50).

the likely encapsulation of the fibres by starch inside the extruder could better justify the results obtained.

The isotherms indicated that, for all three products under the conditions studied, in order to maintain a low moisture content (10%), the relative humidity of the storage environment should be no greater than 70%, which is the recommended limit for the water activity of dehydrated products according to Padula *apud* Sarantopoulos and Oliveira (2001). Under this condition, the sorghum products would be free from microbial attack.

In order to ensure microbiological stability, the water content of the materials should be no higher than 6.9 g of water per 100 g of dry material for raw sorghum flour, 5.3 g of water per 100 g material for the sorghum extrudates and 6.7 g of water per 100 g material for the extruded sorghum flour.

4 Conclusion

The sorption isotherms of the sorghum materials were affected by the type of processing. The extrudates were the least hygroscopic (smaller monolayer), probably due to the effect of polymer-polymer complexation and encapsulation of the fibres resulting from the extrusion cooking process. The milling process exposed more hydrophilic sites by breaking some of the polymer-polymer interactions, which resulted in a larger monolayer. Particle morphology, including size and appearance, influenced water absorption. Both the raw and extruded flours, which presented smaller mean diameters and rough surfaces, adsorbed more moisture from the environment.

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