DOI: 10.1111/jfpp.14658

ORIGINAL ARTICLE

Journal of Food Processing and Preservation

WILEY

Study of three drying methods in production of nutritious flours from the fermentation slurry of orange-fleshed sweet potato

Cristiane Martins Schwein	berger 💿 Juliano Antônio S	ebben 💿
Pedro Henrique Schultz	Jorge Otávio Trierweiler 匝	Luciane Ferreira Trierweiler 匝

Department of Chemical Engineering, Federal University of Rio Grande do Sul (UFRGS), Porto Alegre, Brazil

Correspondence

Juliano Antônio Sebben, Department of Chemical Engineering, Federal University of Rio Grande do Sul (UFRGS), Rua Engenheiro Luiz Englert s/n, Prédio 12204, Porto Alegre, Rio Grande do Sul, 90040-040, Brazil. Email: julianosebben@hotmail.com

Abstract

When producing ethanol from sweet potatoes, the fermentation generates slurry with nutritional content but underutilized for human consumption. Therefore, the nutritional properties of the flours obtained from the fermentation slurry of orange-fleshed sweet potato (rich in carotenoids) was investigated. Three drying methods were carried out to produce slurry flours: convective using a hot-air (60°C), micro-wave, and lyophilization. For carotenoids determination, were applied a conventional method and the Raman spectroscopy, a modern and alternative technique. Regarding the drying methods, the microwave was the most promising since it produced flour presenting higher levels of carotenoids (β -carotene 849.7 µg/g and lycopene 548.4 µg/g). Also, the microwave drying time was 15 min, being much faster than the other methods. In the centesimal analysis of macronutrients, the slurry flours presented high fiber content, around 55 g/100 g. Finally, biscuits containing parcels of the flours demonstrated potential acceptability, by sensory analysis evaluating aroma and appearance.

Practical applications

Since ethanol is a product of worldwide importance, and it is produced mainly from corn and sugar cane, the production from sweet potatoes as an alternative raw material has interested researchers from different countries. Knowing that the ethanol production from sweet potato generates a residual slurry, this study focuses on adding value to this slurry, producing a nutritious flour. The potential for industrial application is promising, assuming it is a residue of an industrial infrastructure already installed. For the industry, obtaining more than one product in the same process represents a financial advantage and resource optimization.

1 | INTRODUCTION

Approximately 90% of the world's ethanol production comes from corn and sugar cane (Gupta & Verma, 2015); this has stimulated the research regarding alternative feedstocks, such as sweet potato. It has high starch content and favorable agronomic characteristics, so its application in ethanol production has been studied by several authors from different countries (Cao, Tian, Yao, & Yuan, 2011; Dewan, Li, Han, & Karim, 2013; Duvernay, Chinn, & Yencho, 2013; Huang et al., 2014; Lareo et al., 2013; Masiero, Peretti, Trierweiler, & Trierweiler, 2014; Schweinberger, Putti, Susin, Trierweiler, & Trierweiler, 2016; Srichuwong et al., 2012; Zhang et al., 2011). 2 of 11

Journal of

Food Processing and Preservation

However, when more products are exploited in the process besides ethanol, the resources are better used, increasing profitability, and business safety. Petterle (2015) analyzed the economic viability of sweet potato microusins for the Rio Grande do Sul state (Brazil), concluding that the ethanol as a single product would lead to narrow profit margins. Therefore, the diversification of the product is crucial to assure financial viability.

The fermentation with sweet potato has the peculiarity of forming a high amount of residual slurry, which is mainly formed by pectic substances, and also by cellulose and hemicellulose. The high viscosity caused by these polysaccharides is a problem during fermentation, so enzymes have been employed to disintegrate them (Puligundla, Smogrovicova, Obulam, & Ko, 2011). Schweinberger et al. (2016) reduced the viscosity of a sweet potato mash by 81% using a pectinase. Although it was notable the degradation of the solid, when the wine was filtered, there were still 43% of the mass retained in the slurry. Hence, this slurry is an opportunity that can be harnessed in the production of value-added food.

Sweet potatoes present low-fat content, around 0.17% (Bradbury & Holloway, 1988), and more than 90% of the carbohydrates are converted into ethanol. Thus, it is expected that the flour of the fermentation slurry presents interesting potential in the food market of fiber sources and low-calorie. Moreover, due to the industrial interest in the application of stable natural dyes, methods of extracting phenolic compounds, antioxidant activity, anthocyanins stability, and in vitro simulated gastrointestinal digestion of purple sweet potatoes have been objects of recent studies (Meng, Tan, & Feng, 2019; Quan et al., 2019; Yang, Tang, Zhang, Zhou, & Zhang, 2019).

Considering this scenario, the presented study is a continuity of the research developed by Schweinberger et al. (2016), but aiming the flour production from the slurry, instead of ethanol production. The orange-fleshed sweet potato was used because this variety contains significant amounts of carotenoids, especially β-carotene, and lycopene (Teow et al., 2007), which adds even more nutritional value to the flour.

Concerning the carotenoids' benefits, the β -carotene acts as vitamin A precursor, contributing to supply the lack of such nutrient, which is a world health problem (World Health Organization, 2009). Also, the high consumption of vegetables and fruits, which are sources of β-carotene, is associated with the reduced risk of cancer, especially lung cancer (Naves, 1998). Moreover, the lycopene possesses high antioxidant activity in quenching singlet oxygen as well as in reacting with the phenoxyl radicals (Baranska, Schütze, & Schulz, 2006).

In the literature, it is common to find studies about DDG (Dried Distillers Grains), which is the residue of corn ethanol production. However, it was not found in the literature a work using the slurry from the alcoholic fermentation of sweet potato, including the focus on carotenoid content. Thus, the presented study contributes to evaluating this subject, where sweet potato is a tuber that has many differences compared to grains.

Also, three drying methods were studied: hot-air, microwave oven with rotary drum, and lyophilization. The hot-air drying is a traditional method, and usually demands small investment; the lyophilization is a method known that better preserve the characteristics of the food, where the water removal mechanism is different, which the ice goes to steam without going through the liquid state, but the freeze-drying is more expensive. For this reason, Ahmed, Thomas, and Khashawi (2020) and Corrêa et al. (2011) studied these methods comparatively when producing green banana flour and marolo flour, respectively.

Microwave drying is modern and significantly faster than the other methods. Thus, increasingly studies are emerging to consolidate this technique in the food industry for different purposes. Ahmed et al. (2020) with samples of green banana flour, and Zhu, Guo, Wu, and Wang (2012) with samples of chestnut flour, determined their dielectric properties, which are important parameters in the design of a microwave oven; Bai-Ngew, Therdthai, Dhamvithee, and Zhou (2015) studied the microwave vacuum drying and hot-air drying on the physicochemical properties of durian flour comparatively. Therefore, the effect of those three drying methods on macro and micronutrients (β -carotene and lycopene) in the produced flours was evaluated in this work.

The carotenoids contents were determined by the conventional method with visible spectrophotometry. The application of Raman spectroscopy as an alternative analytical method for carotenoids determination was also investigated. The advantage of the Raman spectroscopy is that it is nondestructive, a handy tool for both quantitative and qualitative analysis, and it allows structural fingerprint with its narrow and highly resolved bands. It has become a popular technique with accurate and rapid results, which facilitates real-time analysis (Boyaci et al., 2015; Jehlička et al., 2014), whereas current methods applied to carotenoids quantification are time-consuming and require destructive extractions.

Finally, the application of the produced flours was evaluated from the consumer perspective: in the biscuits production, by partially replacing the wheat flour.

MATERIALS AND METHODS 2

The orange-fleshed sweet potatoes used in the experiments were provided by a single farmer from Mariana Pimentel city (Brazil). Figure 1 shows a summary of the transformation steps of the sweet potato.

2.1 | Fermentation

The fermentation was carried out applying the method Preheating followed by Simultaneous Viscosity reduction, Hydrolysis, and Fermentation (P-SVHF), proposed in (Schweinberger, Trierweiler, & Trierweiler, 2019). The sweet potato with peel was preheated to 76°C, then, crushed with water, the ratio potato: water was 1.5 kg: 1

WILEY



FIGURE 1 Process steps: (a) Orange-fleshed sweet potatoes; (b) preparing to the preheating, (c) simultaneous viscosity reduction, hydrolysis, and fermentation, (d) slurry after centrifugation and arranged for drying

L. The cultivation process was done during 19 hr at 34°C and pH 4. The experiments were performed on Erlenmeyers, which were placed in an air incubator under orbital stirring of 175 rpm.

For the starch hydrolysis, the commercial enzyme mixture Stargen 002 (produced by DuPont) was used; it contains an *Aspergillus kawa-chi* α -amylase expressed in *Trichoderma reesei* and a glucoamylase from *Trichoderma reesei*. The Pectinex Ultra AFP enzyme (a pectin lyase obtained from strains of *Aspergillus aculeatus* and *Aspergillus niger*) produced by Novozymes was used for pectic substances degradation, contributing to reduce the mash viscosity. The yeast was the *Saccharomyces cerevisiae* Angel Thermal Resistance (produced by Angel). Doses by mass of sweet potato were: Stargen 002 1 μ l/g, Pectinex Ultra AFP 0.1 μ l/g, yeast 3.33 mg/g, and doxycycline 0.28 mg/g.

It is important to understand that the fermentation methodology followed precisely as it was done in Schweinberger et al. (2016), that work focused on only the ethanol as the product of the process, so the antibiotic doxycycline was used, which was also used in this work. For this reason, and also because there was no specific area for food production in the research group lab, due to the potential risk of cross-contamination by other chemical products, the produced flours were not applied for human consumption in this work.

Although initially there was no human consumption, it was possible to verify the operational possibility of substituting part of the wheat flour by the slurry flour in a biscuit formulation. Then, it was also possible to analyze the sensory acceptance by evaluating the criteria aroma and appearance (methodology described in Section 2.6).

Note that this work is the first experimental study of the research group that aims to add value to the fermentation slurry. The results and discussions reported in this paper indicate the ideal direction for the transformation of the byproduct; besides that, it corresponds to information that can support a subsequent optimization of the process. Future works intend to carry out tests with human consumption (producing in an area only for foods), and then, evaluating the replacement of the antibiotic by potassium metabisulfite, which is an additive commonly used for the prevention of microbiological contamination in wine production (also is used in beer production).

2.2 | Slurry separation

After the SVHF process, the medium was centrifuged at 3,600 rpm for 5 min. The slurry and fermented liquid were separately weighted to quantify the slurry yield. A small liquid sample was collected for the quantification of the ethanol production (analytical details are given in Section 2.4).

2.3 | Drying

The slurry was dried by three different drying methods: hot-air, microwave, and lyophilization.

Hot-air drying was performed at 60°C with an oven (model A3 DGtemp, 48 L, De Leo, Brazil) with 1,100 W nominal power and without forced air circulation. This drying temperature was used to avoid the occurrence of shrinkage and darkening or "hot spots" in the sample. The microwave drying was performed in a prototype elaborated by Sebben (2015). It consists of a domestic microwave oven (model BMS45BBANA, 30 L, 2,450 MHz, Brastemp, Brazil), with 820 W of nominal power and with a rotary drum made of acrylic installed into the resonant cavity, as can be seen in Figure 2. The rotation velocity was 60 rpm. Moreover, the process was operated with reduced pressure at 79 kPa, through the connection to a vacuum pump, aiming to remove the steam. Throughout the microwave drying, the temperature inside the cavity was monitored with the use of a thermographic camera, to avoid overheating. When the temperature reached between 80°C-90°C, the oven was turned off, and when the system reached the ambient temperature, the mass was measured to construct the drying curve. The process restarted after each drying point. Lyophilization, on the contrary, worked at a higher vacuum. For lyophilization, the sample was frozen in an ultra-freezer at -40°C, and then the drying occurred at 25°C.

4 of 11



FIGURE 2 Microwave oven with rotary drum used in the drying experiment

The kinetic drying curves for hot-air and microwave methods were built with experiments performed in duplicate. Lyophilization drying curves were not made due to operational impossibilities. The moisture relation (MR, Equation (1)) was calculated for the kinetic drying curves:

$$MR = \frac{X_t - X_e}{X_o - X_e} \tag{1}$$

where X_t means the moisture at any instant of time, X_e is the equilibrium moisture and X_o is the initial moisture, all these moistures are given on a dry basis, and they were measured by weighing the samples. In hot-air drying, approximately 2 g of sample was placed (a thin layer) in a drying capsule. In the microwave drying, due the rotary drum was larger, more sample was needed, that is, approximately 200 g.

It is well known that the drying kinetics behaves as an exponential model (Saavedra et al., 2017):

$$MR = a \cdot \exp\left(-k_d t\right) \tag{2}$$

where *a* is a correlation parameter, k_d is the kinetic constant (min⁻¹), and *t* is the drying time (min). The experimental points obtained with hot-air drying were adjusted according to Equation (2); on the contrary, the points obtained with the microwave drying were better adjusted to a straight line.

At the end of the drying methods, the dried slurry presented lumps, thus, to standardize the flours, it was ground to a particle size of 48 mesh.

2.4 | Chemical analysis

The analytical methods employed were reported in this section, emphasizing that all described measurements were performed in triplicate.

The moisture analyzes were performed by thermogravimetry (oven drying at 105°C). The ethanol and sugars determination were carried out by HPLC with the Hi-Plex H Agilent column. The column temperature was 60°C, with Refractive Index Detector. The mobile phase flow rate was 0.6 ml/min; the injected sample volume was 20 μ l. Water was the mobile phase.

The Total Reducing Sugars (TRS), that is, fructose (free and from sucrose) + glucose (free, from sucrose, and starch), gives the potential substrate that can be converted into ethanol. Therefore, to quantify the starch content, the sweet potato was hydrolyzed using HCl in an autoclave at 1 atm for 2 hr, following the method proposed by Masiero (S. S. Masiero, 2012). Then, the glucose released was measured by HPLC as described for sugars above. To know the corresponding starch content from the glucose measured, the stoichiometric ratio starch/glucose is applied, that is, 162/180.

The protein analysis was done applying the Kjeldahl method (factor of calculation from nitrogen to proteins: 6.25), the lipid analysis was done by extraction with hexane through the Soxhlet apparatus, the ash content was determined gravimetrically by the decomposition of the organic matter in a muffle (560°C), and finally, the fiber content was calculated by difference, subtracting the contents of the other macronutrients. All these methods were previously validated for its precision, accuracy, and stability according to the protocol described by Instituto Adolf Lutz (2008).

2.4.1 | β-carotene and lycopene determination: Conventional method

The carotenoids content was determined in the slurry and the slurry flours. The extraction method was based on the methodology described by Rodriguez-Amaya and Kimura (2004). The first step was the extraction with acetone using a mortar and pestle. 1 g of the sample was transferred to a mortar, and 3 g of Hyflo supercel (celite) was added. This mixture was ground with 50 ml of acetone and filtered with Buchner funnel with filter paper. After solid-liquid extraction, it was made the partition with petroleum ether (PE). 5 ml of PE with the acetone extract was added in a separtory funnel of 250 ml. Distilled water (100 ml) was slowly added to avoid the formation of an emulsion. Two phases were separated and the aqueous phase was discarded. The funnel was washed four times with distilled water to remove residual acetone. The PE phase was collected in a volumetric flask, making the solution pass through a small funnel containing anhydrous sodium sulfate to remove the remaining water.

The sweet potato extracts were analyzed by visible spectrophotometry in the 450 and 470 nm to β -carotene and lycopene, respectively. The UV-1600 Spectrophotometer (Pró-Análise, BR) was used in these analyses. Equation (3) was used to calculate the total carotenoid content. Journal of Food Processing and Preservation

where, A, absorbance; V, volume total of the extract; $A_{1cm}^{1\%}$ absorption coefficient of β -carotene in PE (2592) or lycopene in PE (3450).

2.4.2 | β -carotene and lycopene determination: Raman spectroscopy

Raman analyses were performed on an iHR550 spectrometer Raman (Horiba Jobin Yvon S.A.S., France) coupled with a cooled chargecoupled device (CCD) detector and a 532 nm laser source. The output power of the laser was adjusted to 50 mW. The spectral data were recorded between 900 and 1,600 cm⁻¹ with constant measurement parameters and diffraction grating of 1,800 grooves/mm. Three accumulations of a 10 s counting time were used. The software package LabSpec 5 (Horiba Jobin Yvon S.A.S., France) was used for spectral data acquisition. Spectra were recorded three times for each sample.

Pretreatments were used for spectral data: a polynomial was fitted through specific points on the spectrum to baseline correction.

2.5 | Morphological properties of the flours

The morphological features of the slurry sweet potato flours were observed via scanning electron microscopy (*SEM*) (JSM 6060). The samples were put in a metal stub and coated with gold powder to make the sample conductive. After, the images were obtained at an accelerating potential of 15 kV.

2.6 | Biscuits production and sensory analysis

Biscuits were produced using the obtained flours to evaluate their culinary applicability. Thus, the slurry flours replaced 20% of the wheat flour in the formulation (this replacement was determined in preliminary tests, where it was possible to perceive the difference caused by the slurry flour in the biscuits). Also, two other biscuits were produced for comparison; the first one had the formulation with only wheat flour, and on the other 20% of the wheat flour was replaced by a commercial sweet potato flour. The biscuits formulation were: water 3.04%, sugar 38.67%, egg powder 3.48%, powdered milk 1.30%, oil 5.46%, chemical ferment 0.19%, when using the slurry flours: slurry flour 9.57% and wheat flour 38.29%, when using the commercial sweet potato flour: commercial sweet potato flour 9.57% and wheat flour 38.29%, and when using only wheat flour: wheat flour 47.85%. The biscuits were baked at 200°C for 18 min.

The sensory acceptance of the biscuits was evaluated by applying the hedonic scale test. For reasons of acuity (prevention of cross-contamination by chemicals, and also due to the use of an antibiotic), the biscuits were not consumed (more details about this issue are discussed in Section 2.1), and then, the criteria evaluated in the sensorial analysis were the aroma and visual presentation (attractiveness by appearance and color).

The hedonic scale was structured with points from 1 to 9 (I immensely disliked to I immensely liked). Thirty judges evaluated the biscuits. This experiment was approved by the ethics committee (CAAE: 10325419.5.0000.5347).

2.7 | Statistical analysis

Statistica software was used to evaluate the equality of means through the Tukey test.

It is noteworthy that in the macronutrient results, the Tukey test was not applied because they were also compared with data of other flours from the literature (which there was not enough information to use the Tukey test). Thus, the experimental results of the macronutrients are presented only with the standard deviation.

3 | RESULTS AND DISCUSSION

3.1 | Fermentation and slurry separation

The sweet potato used in the experiments showed high moisture content (83.3%), therefore low TRS (12.04%), leading to an ethanol potential of 4.98% (vol/vol). Experimentally was achieved the ethanol content of 4.19% (vol/vol). Thus, the fermentative efficiency was 84% (the residual glucose content was 0.01 g/L).

Concerning the slurry yield, it corresponded to 26% of the fermented mass, and its moisture was 87.43%. This value of yield is lower than the one quantified by Schweinberger et al. (2016), that is, 43%. A reason that can be assigned to this difference is that the sweet potato used in the referenced work had a higher solids content (75%–77% of moisture), which increases the susceptibility to the slurry formation.

3.2 | Drying

The kinetic drying curves can be seen in Figure 3a,b, for hot-air (kiln), and microwave, respectively. In Figure 3a, the kinetics of drying is fitted to an exponential model achieving a determination coefficient higher than 0.985. Such behavior is expected for the hot-air drying, where on the first stage, the solid surface is entirely covered by a film of water, resulting in high drying velocity (part of the curve that is similar to a straight line). At the moment that the surface begins to dry, the drying rate is governed by the diffusion of the water through the solid, which reduces the drying velocity, justifying the exponential behavior.

In Figure 3b, the microwave drying was better explained with a linear behavior. It happens because the phenomenon of heating is different from what occurs with hot-air drying. The electromagnetic waves penetrate the food, exciting and heating, mainly the water (because it is the predominant dipole molecule). Due to the

WILEV



FIGURE 3 Kinetic drying curves: hot-air at 60°C (a) and microwave (b). The curves were built with experiments performed in duplicate

penetration capacity of the microwaves, all the volume is heated, and the drying velocity is not limited by diffusion, leading to a linear drying curve (Chandrasekaran, Ramanathan, & Basak, 2013).

As already known, the temperature distribution inside a microwave cavity is heterogeneous (Chandrasekaran et al., 2013; Sebben, Trierweiler, & Trierweiler, 2016; Zhu et al., 2012); this fact is responsible for placing the mass to be heated inside of a rotary drum, as done in this work. The maximum and minimum temperatures of the slurry for each sampling point, in two distinct experiments, were recorded and can be visualized in Figure 4a. Additionally, Figure 4b shows an example of temperature recording, which was done using a thermographic camera. Initially, there is a higher difference between the maximum and minimum temperatures; however, with the progress of the experiment, the difference was reducing. The electromagnetic energy is mainly absorbed by water. Thus, at the beginning of the drying, the sample had high moisture content, and an uneven microwave incidence leads to points significantly hotter than others. At the end of drying, the temperature differences are smaller because the moisture content is reduced. The behavior observed in Figure 4a can be related to what is seen in Figure 3b. In Figure 4a, when the upper and lower temperatures become closer (after measurement point no. 13), the MR value is < 0.3 (Figure 3b).

3.3 | β-carotene and lycopene analysis

Figure 5 shows the average of Raman spectra obtained for the fermentation slurry and the flours produced with different drying methods.



FIGURE 4 (a) Maximum (red lines) and minimum (blue lines) temperatures observed in the rotary drum at different sampling points during the drying. (b) Example of temperature recording, measured using a thermographic camera. The color scale indicated that the sample presented regions with different temperatures. In the image, the yellow and orange tones indicate the surface of the heated sample, while the regions in blue correspond to the unheated acrylic walls of the rotating drum



FIGURE 5 Raman spectra of the slurry and flours obtained by hot-air drying, lyophilization, and microwave drying

For all samples, the three characteristic peaks of carotenoids were identified. The Raman spectrum of carotenes as β -carotene and ly-copene is composed of three prominent vibrational bands at 1,520, 1,157, and 1,006 cm⁻¹. The peak at 1,006 cm⁻¹ occurs due to methyl in-plane rocking modes. The high-intensity peaks at 1,157 and 1,520 cm⁻¹ were attributed to the in-phase symmetric stretching of C-C and C=C of the carotenoid polyene chain, respectively (Cintă Pinzaru et al., 2015; Huo et al., 2011; Killeen et al., 2013).

WILEY

Table 1 shows the values of β -carotene and lycopene obtained by the conventional quantification method: extraction followed by visible spectrophotometry measurements. Comparing the values from Table 1, the carotenoid losses were lower to microwave drying, followed by the lyophilization (highlighting that the Tukey test did not accuse significant differences between the results for the microwave and lyophilization), and hot-air at 60°C. The reason for the flour obtained by the microwave method to keep a high carotenoid concentration can be explained by its heating process. It was faster than the others, reducing the time of the slurry exposure to the air, which would lead to oxidation losses. On the contrary, the drying using an oven with hot-air led to higher losses due to the prolonged exposure to the air, and at temperatures more favorable to the oxidation reaction, when compared to freeze-drying. According to Sebben et al. (2018), reduction of around fifty percent of carotenoid content in orange-fleshed sweet potato was obtained after 125 min under hot-air exposure, and also, by 8 min using microwave drying. In the present work, the carotenoids contents, that is, the sum of β -carotene and lycopene, shows a reduction correspondent at around 16% and 43% for the microwave (15 min) and hot-air (200 min), respectively. It is of note that in the present work, the object under study was the slurry of fermentation and its flours, while in Sebben et al. (2018), samples were the

sweet potatoes. Although the comparison is not entirely appropriate because they were different materials, it can be noted that carotenoid losses were lower in this work.

3.4 | Centesimal composition

The centesimal compositions of the flours were determined regarding the macronutrients, and the results are presented in Table 2. Additionally, for comparison purposes, data from the literature of commercial sweet potato flour and wheat flour are presented.

Through the standard deviations presented in Table 2 is possible to note that there is no significant difference between the composition of the three slurry flours. Thus, it can be concluded that drying methods did not influence the macronutrient content. It is expected that the most significant influence will occur on more unstable components, as observed in the carotenoid content, where the losses were significantly higher in the hot-air compared to the other methods.

On the contrary, when comparing the slurry flours (made for this study) with the sweet potato flour and wheat flour (both commercialized flours), differences were noted. Since most of the starch is consumed in the fermentation (the fermentative efficiency was 84%, discussed in Section 3.1), reduced carbohydrates content in the slurry

	β-carotene		Lycopene	
	Content (µg/g) ¹	CV (%) ²	Content (µg/g) ¹	CV (%) ²
Slurry	979.6 ^a ± 145.40	14.85	644.5 ^a ± 88.41	13.72
Slurry flours				
Lyophilization	764.4 ^{a,b} ± 65.25	8.54	462.2 ^{b,c} ± 14.35	3.10
Hot-air at 60°C	573.5 ^b ± 115.30	20.10	349.4 ^c ± 43.66	12.50
Microwave	849.7 ^a ± 74.17	8.73	$548.4^{a,b} \pm 20.16$	3.68

¹Mean values that do not share the same letter are significantly different, according to Tukey test with 95% confidence.

²The coefficient of variation (CV) is calculated as the standard deviation divided by the mean.

TABLE 2 The averaged centesimal composition of the flours and standard deviations. Contents of the components are given in g/100 g (wet basis)

	Flours produced with the following methods:				Wheat
	Hot-air	Microwave	Lyophilization	Sweet potato flour ¹	flour ²
Carbohydrates	8.36 ± 3.8	9.90 ± 2.9	12.71 ± 4.50	73.63	75.10
Proteins	10.34 ± 0.19	10.75 ± 0.25	10.15 ± 0.04	6.85	9.80
Lipids	3.26 ± 0.25	3.82 ± 0.36	3.56 ± 0.35	0.83	1.40
Fibers	57.26 ± 4.39	56.31 ± 3.65	52.9 ± 5.07	9.92	2.30
Ashes	8.14 ± 0.15	8.20 ± 0.14	7.45 ± 0.18	-	0.80
Moisture	12.57 ± 0.31	11.02 ± 0.25	13.23 ± 0.01	-	13.00
Kcal/100 g	104.14 ± 18.21	116.98 ± 15.84	123.48 ± 21.31	329.39	360.00

¹Source: (Sabor em grãos—Farinha de Batata Doce (Granel 100 g), 2018)

²Source: (Lima et al., 2006).

TABLE 1 β-carotene and lycopeneresults (contents given in dry basis)

flours would be expected, which is advantageous because it results in lower caloric content. Also, by reducing the carbohydrates, the proportion of the other nutrients increases, as seen in Table 2, highlighting the positive gain in the high fiber content. As can be seen in the same table, more than 50% of the centesimal composition of the flours were fibers. This result raises the flour produced to the level of potentially nutritious food, since in some countries bread is the primary source of fiber (11%-30% of total fiber), with much smaller contributions from breakfast cereals (5%-8%), biscuits and pastries (3%-11%), and pasta (1%-4%). Furthermore, vegetables contributing from 12% to 21% of fiber intake, potatoes from 6% to 19%, and fruits from 8% to 23% (Gębski, Jezewska-Zychowicz, Szlachciuk, & Kosicka-Gębska, 2019). Increasing fiber intake represents the most crucial objectives for public health worldwide to combat "diet-related" noncommunicable diseases; therefore, changing the composition of food products seems to be an effective method of influencing improvement in the diet (Spiteri & Soler, 2018). Regarding the protein content of the slurry flours, it is not higher than of sweet potato flour, a fact attributed to the consumption of proteins by the yeast during the fermentation.

3.5 | Morphological properties of the flours

Scanning electron micrographs (SEM) of the slurry sweet potato flours are shown in Figure 6. The micrographs showed an irregular structure, with a layered surface. This aspect refers to fibrous material, being coherent with the high fiber contents seen in Table 2. The residual starch would be observed as spheres shape, which was not found in the SEM analysis. The physiological changes between the drying methods are not visible because the microwave power has been controlled, implying that the higher diffusion rates during the drying process lead to intensified evaporation of the water.

3.6 | Biscuits production and sensory analysis

Images of the biscuits produced can be seen in Figure 7, and the results of the sensory analysis are presented in Table 3.

According to the sensorial analysis results (Table 3), the biscuit containing slurry flour from the microwave drying had a visual presentation statistically similar to biscuits containing only wheat flour and the commercial sweet potato flour (all obtained the highest classification, indicated in the group "a" according to Tukey test). Although the visual difference between such biscuits is remarkable (Figure 7), they were in the same acceptance class due to different types of cookies that referred to the evaluators. With the wheat flour, the biscuit resembles a traditional one. The coloring of the sweet potato flour biscuit is attractive, and the biscuit with the slurry flour seems to be nutritious (whole food).

As the visual appearance of the biscuits is one of the criteria for comparing the drying methods to obtain the flour, it was possible to identify by the appearance that there was no significant difference between the biscuits containing flour obtained by microwave and oven with hot-air, both classified in group B (photographs can be seen in Figure 7).

On the contrary, the biscuits containing slurry flour from the lyophilization had the worst appearance, according to the interviewed individuals. One of the possible reasons for this lower acceptance was that the lyophilized flour had a higher capacity for water absorption compared to the others. Although the amount of water in the formulation for all biscuits was the same, during the handling of the



FIGURE 6 Scanning electron micrographs of the flour obtained from: (a1) hot-air-dried, magnification ×500; (a2) hot-air-dried, magnification ×1500; (b1) lyophilized, magnification ×500; (b2) lyophilized, magnification ×1500; (c1) microwave-dried magnification ×500; (c2) microwave-dried magnification ×1500

FIGURE 7 Biscuits produced with different flours. (a) 100% wheat flour. Replacing 20% of the wheat flour with: (b) commercial sweet potato flour, (c) slurry flour dried by hot-air drying, (d) slurry flour from microwave-drying and (e) slurry flour from freeze-drying (lyophilization)



TABLE 3 Averages of the notes of the sensory analysis (scale having points from 1 to 9)

	Criterion ¹	
Biscuits containing	Aroma	Visual presentation ²
A–Only wheat flour	5.09 ^a	6.83 ^{a,b}
B-Sweet potato flour	5.57ª	7.26ª
Slurry flours		
E–Lyophilization	4.34 ^a	3.94 ^c
C—Hot-air	5.26ª	5.97 ^b
D-Microwave	4.89 ^a	6.49 ^{a,b}

¹Mean values that do not share the same letter are significantly different, according to Tukey test with 95% confidence. ²Attractiveness by appearance and color.

dough containing the lyophilized flour, it has absorbed more water, resulting in the dry, less lubricated, and more brittle dough, which justifies the appearance of the resulting biscuit be less attractive.

Regarding the aroma evaluation, the Tukey test suggested that there were no significant differences in the aroma between all the biscuits analyzed. As presented in Section 2.6, the biscuits had the same formulation base, with 20% of wheat flour replaced by sweet potato flour and slurry flours. Therefore, these formulations did not result in a significant difference in aroma.

According to some interviewed individuals, the biscuits containing slurry flour had an aroma and visual presentation similar to those produced with whole-wheat flour. Since consumption of whole foods has been appreciated for its benefits to human health and nutritional value, it is noticed that the biscuits produced with the slurry flour can pursue easy acceptance by the consumers of that market.

In the first application of flour for the development of bakery products, it was noted that the sensory scores of the biscuits produced with the slurry flour (oven and microwave) were between 5 and 6. In the scale used, which goes from 1 to 9, score 5 means indifference, thereby it was possible to conclude that such flours have the potential to be used in bakery products, such as biscuits. It should be noted, however, that for the safe commercialization for human consumption of the biscuits obtained from this methodology, more in-depth studies are required, and more formulations should be applied, applying broader sensory tests, with texture, and flavor parameters. Besides, financial profitability must also be estimated.

4 | CONCLUSIONS

This work had as purpose to evaluate the potential of adding value to the sweet potato fermentation slurry, aiming the culinary application by producing flour that is also a source of β -carotene and lycopene. Regarding the drying methods, the microwave shown to be most promising since it resulted in higher levels of carotenoids, is faster than the others and is more straightforward than lyophilization.

The centesimal composition of the produced flours presented high fiber contents, which was also verified in the *SEM* images. This result is justified because most carbohydrates are consumed in the fermentation, thus, increasing the fiber concentration. Such characteristics become the flour a promising supplement for food formulations.

In a first approach, which was the biscuits producing, it is concluded that there is potential for sensory acceptance. However, to confirm that possibility, more tests must be carried out for improvements. First, replacing the antibiotic with the potassium metabisulfite and performing the production in a specific area for food. Then, sensory tests could be done with human consumption. In addition, it is essential for the development of more formulations, with a different balance between the ingredients, and more sensorial tests, in order to optimize the culinary application.

Finally, to enrich the information on the slurry flour, the importance of investigating more characteristics in future studies, such as amino acid composition, solubility, density, average particle size, pH, water absorption capacity, and viscosity was highlighted. Journal of Food Processing and Preservation

CONFLICT OF INTEREST

The authors have declared no conflicts of interest for this article.

ORCID

Cristiane Martins Schweinberger ២ https://orcid.

org/0000-0002-2919-9821

Juliano Antônio Sebben ២ https://orcid.org/0000-0001-5499-1469

Jorge Otávio Trierweiler ២ https://orcid.

org/0000-0002-6328-945X

Luciane Ferreira Trierweiler ២ https://orcid.

org/0000-0003-4497-7334

REFERENCES

- Ahmed, J., Thomas, L., & Khashawi, R. (2020). Influence of hot-air drying and freeze-drying on functional, rheological, structural and dielectric properties of green banana flour and dispersions. *Food Hydrocolloids*, 99. https://doi.org/10.1016/j.foodhyd.2019.105331
- Bai-Ngew, S., Therdthai, N., Dhamvithee, P., & Zhou, W. (2015). Effect of microwave vacuum drying and hot air drying on the physicochemical properties of durian flour. *International Journal of Food Science and Technology*, 50(2), 305–312. https://doi.org/10.1111/ijfs.12651
- Baranska, M., Schütze, W., & Schulz, H. (2006). Determination of lycopene and β-carotene content in tomato fruits and related products: Comparison of FT-raman, ATR-IR, and NIR spectroscopy. *Analytical Chemistry*, 78(24), 8456–8461. https://doi.org/10.1021/ac061220j
- Boyaci, I. H., Temiz, H. T., Geniş, H. E., Acar Soykut, E., Yazgan, N. N., Güven, B., ... Dudak Şeker, F. C. (2015). Dispersive and FT-Raman spectroscopic methods in food analysis. *RSC Advances*, 5(70), 56606–56624. https://doi.org/10.1039/C4RA12463D
- Bradbury, J. H., & Holloway, W. D. (1988). Chemistry or Tropical Root Crop: Significance for nutrition and agriculture in the Pacific. ACIAR Monograph No. 6., Canberra.
- Cao, Y., Tian, H., Yao, K., & Yuan, Y. (2011). Simultaneous saccharification and fermentation of sweet potato powder for the production of ethanol under conditions of very high gravity. *Frontiers of Chemical Science and Engineering*, 5(3), 318–324. https://doi.org/10.1007/ s11705-010-1026-3
- Chandrasekaran, S., Ramanathan, S., & Basak, T. (2013). Microwave food processing—A review. Food Research International, 52(1), 243–261. https://doi.org/10.1016/j.foodres.2013.02.033
- Cintă Pinzaru, S., Müller, C., Tomšić, S., Venter, M. M., Cozar, B. I., & Glamuzina, B. (2015). New SERS feature of β-carotene: Consequences for quantitative SERS analysis. *Journal of Raman Spectroscopy*, 46(7), 597–604. https://doi.org/10.1002/jrs.4713
- Corrêa, S. C., Clerici, M. T. P. S., Garcia, J. S., Ferreira, E. B., Eberlin, M. N., & Azevedo, L. (2011). Evaluation of dehydrated marolo (Annona crassiflora) flour and carpels by freeze-drying and convective hot-air drying. *Food Research International*, 44(7), 2385–2390. https://doi.org/10.1016/j.foodres.2011.02.052
- Dewan, A., Li, Z., Han, B., & Karim, M. N. (2013). Saccharification and fermentation of waste sweet potato for bioethanol production. *Journal* of Food Process Engineering, 36(6), 739–747. https://doi.org/10.1111/ jfpe.12042
- Duvernay, W. H., Chinn, M. S., & Yencho, G. C. (2013). Hydrolysis and fermentation of sweetpotatoes for production of fermentable sugars and ethanol. *Industrial Crops and Products*, 42, 527–537. https://doi. org/10.1016/j.indcrop.2012.06.028
- Gębski, J., Jezewska-Zychowicz, M., Szlachciuk, J., & Kosicka-Gębska, M. (2019). Impact of nutritional claims on consumer preferences for bread with varied fiber and salt content. *Food Quality* and Preference, 76, 91–99. https://doi.org/10.1016/j.foodq ual.2019.03.012

- Gupta, A., & Verma, J. P. (2015). Sustainable bio-ethanol production from agro-residues: A review. *Renewable and Sustainable Energy Reviews*, 41, 550–567. https://doi.org/10.1016/j.rser.2014.08.032
- Huang, Y., Jin, Y., Shen, W., Fang, Y., Zhang, G., & Zhao, H. (2014). The use of plant cell wall-degrading enzymes from newly isolated Penicillium ochrochloron Biourge for viscosity reduction in ethanol production with fresh sweet potato tubers as feedstock. *Biotechnology* and Applied Biochemistry, 61(4), 480–491. https://doi.org/10.1002/ bab.1190
- Huo, M. M., Liu, W. L., Zheng, Z. R., Zhang, W., Li, A. H., & Xu, D. P. (2011). Effect of end groups on the raman spectra of lycopene and β-carotene under high pressure. *Molecules*, 16(3), 1973–1980. https://doi. org/10.3390/molecules16031973
- Jehlička, J., Edwards, H. G. M., Osterrothová, K., Novotná, J., Nedbalová, L., Kopecký, J., ... Oren, A. (2014). Potential and limits of Raman spectroscopy for carotenoid detection in microorganisms: Implications for astrobiology. *Philosophical Transactions of the Royal Society* A: Mathematical, Physical and Engineering Sciences, 372(2030), 20140199. https://doi.org/10.1098/rsta.2014.0199
- Killeen, D. P., Sansom, C. E., Lill, R. E., Eason, J. R., Gordon, K. C., & Perry, N. B. (2013). Quantitative Raman spectroscopy for the analysis of carrot bioactives. *Journal of Agricultural and Food Chemistry*, 61(11), 2701–2708. https://doi.org/10.1021/jf3053669
- Lareo, C., Ferrari, M., Guigou, M., Fajardo, L., Larnaudie, V., Ramírez, M., & Martínez-Garreiro, J. (2013). Evaluation of sweet potato for fuel bioethanol production: Hydrolysis and fermentation. *SpringerPlus*, 2(1), 493. https://doi.org/10.1186/2193-1801-2-493
- Lima, D. M., Colugnati, F. A. B., Padovani, R. M., Rodriguez-Amaya, D. B., Salay, E., & Galeazzi, M. A. M. (2006). *Tabela Brasileira de Composição de Alimentos-TACO*. Núcleo de Estudos e Pesquisas em Alimentação-NEPA/UNICAMP.
- Masiero, S. S. (2012). Microusinas de etanol de batata-doce: Viabilidade econômica e técnica. Universidade Federal do Rio Grande do Sul (UFRGS).
- Masiero, S. S., Peretti, A., Trierweiler, L. F., & Trierweiler, J. O. (2014). Simultaneous cold hydrolysis and fermentation of fresh sweet potato. *Biomass and Bioenergy*, 70, 174–183. https://doi.org/10.1016/j. biombioe.2014.08.007
- Meng, X.-J., Tan, C., & Feng, Y. (2019). Solvent extraction and in vitro simulated gastrointestinal digestion of phenolic compounds from purple sweet potato. International Journal of Food Science and Technology, 54(10), 2887–2896. https://doi.org/10.1111/ijfs.14153
- Naves, M. M. V. (1998). Beta—caroteno e câncer. Revista de Nutrição, 11(2), 99–115. https://doi.org/10.1590/S1415-52731998000200001
- Petterle, E. C. (2015). Produção Descentralizada de Etanol Visando suprir a Planta de Eteno Verde da Braskem. Porto Alegre: Federal University of Rio Grande do Sul (UFRGS).
- Puligundla, P., Smogrovicova, D., Obulam, V. S. R., & Ko, S. (2011). Very high gravity (VHG) ethanolic brewing and fermentation: A research update. *Journal of Industrial Microbiology & Biotechnology*, 38(9), 1133–1144. https://doi.org/10.1007/s10295-011-0999-3
- Quan, W., He, W., Lu, M., Yuan, B., Zeng, M., Gao, D., ... He, Z. (2019). Anthocyanin composition and storage degradation kinetics of anthocyanins-based natural food colourant from purple-fleshed sweet potato. *International Journal of Food Science and Technology*, 54(8), 2529–2539. https://doi.org/10.1111/ijfs.14163
- Rodriguez-Amaya, D., & Kimura, M. (2004). HarvestPlus handbook for carotenoid analysis. HarvestPlus Technical Monographs, 59.
- Saavedra, J., Córdova, A., Navarro, R., Díaz-Calderón, P., Fuentealba, C., Astudillo-Castro, C., ... Galvez, L. (2017). Industrial avocado waste: Functional compounds preservation by convective drying process. *Journal of Food Engineering*, 198, 81–90. https://doi.org/10.1016/j. jfoodeng.2016.11.018
- Sabor em grãos–Farinha de Batata Doce (Granel 100g). (2018). Retrieved from https://www.saboremgraos.com.br/farinaceos/farinha-debatata-doce-granel-100g

- Schweinberger, C. M., Putti, T. R., Susin, G. B., Trierweiler, J. O., & Trierweiler, L. F. (2016). Ethanol production from sweet potato: The effect of ripening, comparison of two heating methods, and cost analysis. *The Canadian Journal of Chemical Engineering*, 94(4), 716– 724. https://doi.org/10.1002/cjce.22441
- Schweinberger, C. M., Trierweiler, J. O., & Trierweiler, L. F. (2019). Preheating followed by simultaneous viscosity reduction, hydrolysis, and fermentation: simplifying the process of ethanol production from sweet potato. *Bioenergy Research*, 12(1), 94–102. https://doi. org/10.1007/s12155-018-9953-9
- Sebben, J. A. (2015). Desenvolvimento de tambor rotativo para micro-ondas e aplicação no processamento de batata-doce. Porto Alegre: Federal University of Rio Grande do Sul.
- Sebben, J. A., Trierweiler, L. F., & Trierweiler, J. O. (2016). Orangefleshed sweet potato flour obtained by drying in microwave and hot air. Journal of Food Processing and Preservation, 41, 1–8. https://doi. org/10.1111/jfpp.12744
- Sebben, J. A., da Silveira Espindola, J., Ranzan, L., Fernandes de Moura, N., Trierweiler, L. F., & Trierweiler, J. O. (2018). Development of a quantitative approach using Raman spectroscopy for carotenoids determination in processed sweet potato. *Food Chemistry*, 245, 1224–1231. https://doi.org/10.1016/j.foodchem.2017.11.086
- Spiteri, M., & Soler, L. G. (2018). Food reformulation and nutritional quality of food consumption: An analysis based on households panel data in France. European Journal of Clinical Nutrition, 72(2), 228–235. https://doi.org/10.1038/s41430-017-0044-3
- Srichuwong, S., Orikasa, T., Matsuki, J., Shiina, T., Kobayashi, T., & Tokuyasu, K. (2012). Sweet potato having a low temperature-gelatinizing starch as a promising feedstock for bioethanol production. *Biomass and Bioenergy*, *39*, 120–127. https://doi.org/10.1016/j.biomb ioe.2011.12.023
- Teow, C. C., Truong, V. D., McFeeters, R. F., Thompson, R. L., Pecota, K. V., & Yencho, G. C. (2007). Antioxidant activities, phenolic and ??-carotene contents of sweet potato genotypes with varying flesh

colours. *Food Chemistry*, 103(3), 829–838. https://doi.org/10.1016/j. foodchem.2006.09.033

- World Health Organization. (2009). Global prevalence of vitamin A deficiency in populations at risk 1995–2005 : WHO global database on vitamin A deficiency. *WHO Iris* (p. 55). ISBN 978 92 4 159801 9.
- Yang, Z.-w., Tang, C.-e., Zhang, J.-L., Zhou, Q., & Zhang, Z.C. (2019). Stability and antioxidant activity of anthocyanins from purple sweet potato (*Ipomoea batatas* L. cultivar Eshu No. 8) subjected to simulated in vitro gastrointestinal digestion. *International Journal of Food Science and Technology*, 54(8), 2604–2614. https://doi.org/10.1111/ ijfs.14172
- Zenebon, O., Pascuet, N. S., Tiglea, P. (2008). Métodos físico-químicos para análise de alimentos (p. 1020). São Paulo: Instituto Adolf Lutz.
- Zhang, L., Zhao, H., Gan, M., Jin, Y., Gao, X., Chen, Q., ... Wang, Z. (2011). Application of simultaneous saccharification and fermentation (SSF) from viscosity reducing of raw sweet potato for bioethanol production at laboratory, pilot and industrial scales. *Bioresource Technology*, 102(6), 4573–4579. https://doi.org/10.1016/j.biortech.2010.12.115
- Zhu, X., Guo, W., Wu, X., & Wang, S. (2012). Dielectric properties of chestnut flour relevant to drying with radio-frequency and microwave energy. *Journal of Food Engineering*, 113(1), 143–150. https:// doi.org/10.1016/j.jfoodeng.2012.04.014

How to cite this article: Schweinberger CM, Sebben JA, Schultz PH, Trierweiler JO, Trierweiler LF. Study of three drying methods in production of nutritious flours from the fermentation slurry of orange-fleshed sweet potato. *J Food Process Preserv.* 2020;44:e14658. <u>https://doi.org/10.1111/</u> jfpp.14658