

Hygroscopic behavior of spray dried acerola and seriguela mixed juice powder stored

Comportamento higroscópico de suco misto de acerola e seriguela em pó armazenado

Christine Maria Carneiro Maranhão Ribeiro^[1], Larry Oscar Chañi Paucar^[2], Enayde de Almeida Melo^[3], Josivanda Palmeira Gomes^[4], Josilene de Assis Cavalcante^[5], Flávio Luiz Honorato da Silva^[6], Maria Inês Sucupira Maciel^[7]

[1] tina_maranhao@yahoo.com.br. Universidade Federal da Paraíba (UFPB). [2] larry.76728@gmail.com. Universidade Estadual de Campinas (UNICAMP). [3] enayde@hotmail.com. Universidade Federal Rural de Pernambuco (UFRPE). [4] josivanda@gmail.com. Universidade Federal de Campina Grande (UFCG). [5] josy_cavalcante@yahoo.com.br. Universidade Federal da Paraíba (UFPB). [6] flavioluizh@yahoo.com.br. Universidade Federal da Paraíba (UFPB). [7] m.inesdcd@gmail.com. Universidade Federal Rural de Pernambuco (UFRPE).

ABSTRACT

Spray drying is an alternative to extend the shelf life of fruits. However, fruit juice powders present problems during their storage because they are hygroscopic. The objectives of this study were to determine adsorption isotherms of mixed juice powder of acerola and seriguela at different temperatures, to identify the best-fit isotherms models and to assess their physicochemical properties. Following physics properties were assessed: apparent and absolute density, solubility, porosity, flowability, cohesiveness, morphology, mean particle size, surface analysis and pore distribution, thermal properties and antioxidant activity. Static-indirect method was used to determine the adsorption isotherms. Experimental data of the water activity and equilibrium moisture were fitted to five mathematical models. The models best fitted to the adsorption curves were the Guggenheim-Anderson-de Boer (GAB), Linearized Brunauer-Emmett-Teller (BET) and Oswin models, $P < 10\%$. The value of the solubility was 79.45%, the absolute density was 1.03 g cm^{-3} , the porosity was 84.47%, and the apparent density was 0.16 g cm^{-3} . The mixed juice powder showed an irregular surface morphology and shape. The results indicated that the mixed juice powder has high thermal stability and can be used as a source of antioxidant with market potential by the food industry.

Keywords: Thermal analyses (TG/DSC). Adsorption curves. Physicochemical properties.

RESUMO

A secagem é uma alternativa para estender a vida útil dos frutos. No entanto, os sucos de frutas em pó apresentam problemas durante o armazenamento por serem higroscópicos. Os objetivos deste estudo foram determinar isotermas de adsorção do suco misto de acerola e seriguela em diferentes temperaturas, identificar o modelo de melhor ajuste e avaliar as propriedades físico-químicas. As seguintes propriedades foram avaliadas: densidade aparente e absoluta, solubilidade, porosidade, fluidez, coesividade, morfologia, tamanho médio de partícula, análise de superfície, distribuição de poros, propriedades térmicas e atividade antioxidante. O método estático-indireto foi utilizado para determinar isotermas de adsorção. Dados experimentais da atividade de água e umidade de equilíbrio foram ajustados a cinco modelos matemáticos. Os modelos mais adequados às curvas de adsorção foram os modelos Guggenheim-Anderson-de Boer (GAB), Brunauer-Emmett-Teller (BET) linearizado e Oswin, $P < 10\%$. A solubilidade foi 79,45%, densidade absoluta $1,03 \text{ g cm}^{-3}$, porosidade 84,47% e densidade aparente $0,16 \text{ g cm}^{-3}$. O suco misto em pó apresentou morfologia e forma irregular na superfície. Os resultados indicaram que o suco misto em pó apresenta alta estabilidade térmica e pode ser considerado uma fonte de compostos antioxidantes, com potencial mercadológico para ser utilizado pela indústria de alimentos.

Palavras-chave: Análise térmica (TG/DSC). Curvas de adsorção. Propriedades físicas.

1 Introduction

The genus *Spondias* has seventeen species, of which 10 species are considered edible, including *S. purpúrea* (Seriguela). The seriguela (*S. purpúrea*) has several quality traits, such as an attractive orange-reddish color, flavor and thickness that are useful in the fruit processing industry (MALDONADO-ASTUDILLO *et al.*, 2014). In Brazil, some species of the *Spondias* genus are used to produce jams, ice creams and juices (CAVALCANTE *et al.*, 2018) the aim of this study was to determine desorption isotherms and isosteric heat of yellow mombin (*Spondias mombin* L.). Although this fruit has an excellent flavor and is a great source of nutrients, it is still underused.

Acerola has been used as an enrichment agent in various food products due to its high vitamin C content, which ranges from 1190.65 to 2187.06 mg/100 g (MORAES *et al.*, 2019). However, this fruit is highly perishable and seasonal, which has hindered their marketing.

The purpose of mixing acerola with seriguela is to enhance the nutritional value as well as contribute to improving the use of and diversifying mixed fruit products. Currently, powdered products obtained from fruit pulp are increasingly used by the national food industry (SILVA FILHO *et al.*, 2018). Dehydration is as an alternative to increase its shelf-life.

Spray drying is an old technique that has been used to transform liquid foods into more stable products (ISLAM *et al.*, 2016). It is very important to choose proper wall material because it affects the surface morphology and stability of microcapsules. Maltodextrin is a hydrolyzed starch with a DE value below 20%. It is widely used as a carrier agent in the drying process because it has excellent properties such as good stability, aroma and neutral flavor, low viscosity in high concentration, and it is inexpensive and suitable for ingredients that undergo oxidation (SHAO *et al.*, 2019).

However, the physical properties, including the density, moisture and particle size, are affected by the temperature and carrier agent (TONTUL; TOPUZ, 2017). Another property that affects the quality of fruit juice powder is the glass transition temperature. Glass transition temperature (T_g) can use as a reference parameter to characterize the properties, quality and stability of food (ISLAM *et al.*, 2016).

Hygroscopicity is a physical parameter that also affects the quality of powders. Highly hygroscopic

powders usually have some handling and storage problems (TONTUL; TOPUZ, 2017).

The fruit juice powder usually presents some problems by being highly hygroscopic that causes an effect called agglomeration and other undesirable effects (RIBEIRO; COSTA; AFONSO, 2019).

Determining the relationship between water activity (a_w) and equilibrium moisture (U) known as sorption isotherm is widely used when it is desired to guarantee a better microbiological, physical chemical stability of the food and to extend the shelf life of food products (AKSIL *et al.*, 2019).

There are several mathematical models to determine the sorption isotherm. The accuracy of mathematical models depends on water activity ranges or food types. Each food has a type of isotherm and therefore the use of several mathematical models (CONEGERO *et al.*, 2017). Once the adsorption isotherms curves are obtained, the isosteric sorption heat is evaluated, which evaluates the amount of energy required to remove water from the food, contributing to estimate the physical changes occurring at the food surface (CAVALCANTE *et al.*, 2018).

Thus, the objective of this study was to determine the hygroscopic behavior at different temperatures, to define the best-fit model towards determining parameters to study the storage of this material and to assess the physical properties as well as antioxidant activity of mixed juice powder (MJP) of acerola and seriguela.

2 Materials and methods

Seriguela (*Spondia purpurea* L.) and acerola (*Malpighia emarginata* DC) of the Sertaneja variety were purchased from the Central de Abastecimento de Pernambuco, CEASA/PE at km 70 of the BR 101 Highway South. Carrier agent used was maltodextrin with 9–12% DE (10DE) (MOR-REX® 1910 from Corn Products, Mogi-Guaçu, Brazil).

The fruits were selected based on the color and soluble solids content: yellow-orangish for seriguela and red-purplish for acerola and 11 and 7 °Brix soluble solids, respectively. Soluble solids were measured using a handheld Atago n° 01 Refractometer (0–32 °Brix) with measurements expressed as °Brix. The fruits were washed in running water, sanitized with Hidrosteril (50 ppm of active chlorine) and pulped in a semi-industrial stainless steel pulping machine (Bonina Compacta, Itabauna/BA). The pulps were packed in 20 x 30 cm low-density polyethylene Ziploc bags (Talge

brand) and stored at $-18\text{ }^{\circ}\text{C}$. The solution was prepared from a mixture of the fruit pulps 60% of acerola and 40% of seriguela and maltodextrin and water in a 1:1 pulp-to-water ratio.

Atomization was performed in a laboratory dryer with a nozzle atomization system (Mini-Spray-Dryer LM model MSD 1.0, LABMAQ, Brazil LTDA) under the following conditions: $140\text{ }^{\circ}\text{C}$ temperature, 0.60 L/h flow rate, nozzle of 1.2 mm in diameter, air flow of $30\text{ M}^3/\text{h}$, air pressure of 0.6 bar and 14% maltodextrin 10DE according to Ribeiro *et al.* (2019). After atomization, the powders were weighed, stored in hermetically sealed glass jars (250 mL) and kept in the dark as well as a dry environment with silica gel as a desiccant at a $22 \pm 2\text{ }^{\circ}\text{C}$ temperature until performing the tests.

The apparent density was calculated according to the procedure described by Caparino *et al.* (2012). The apparent density was calculated according to Eq. 1, and the result was expressed in g cm^{-3} :

$$\rho_{ap} = \frac{m}{v} \quad (1)$$

wherein m is the mass of the sample (g) and V is the total volume of the powder in the graduated test tube (mL).

The absolute density was determined at $25\text{ }^{\circ}\text{C}$ in a pycnometer with a thermometer according to the method described by Souza *et al.* (2009). The porosity was calculated according to Eq. 2:

$$\varepsilon = \frac{1 - \rho_{ap}}{\rho_{abs}} \quad (2)$$

wherein ρ_{ap} is the apparent density (g cm^{-3}) and ρ_{abs} is the absolute density (g cm^{-3}) of the sample.

The solubility was assessed according to the method described by Cano-Chauca *et al.* (2005). A total of 1 g of sample was diluted in 100 mL of distilled water, followed by stirring on a magnetic stirrer (Fisatom, model 752, São Paulo/SP) for 5 min and subsequent centrifugation in a Cientec centrifuge (model CT-6000R, Charqueada/SP) at 3000 rpm/5 min. A 25 mL aliquot of the supernatant was plated in a previously weighed Petri dish and incubated in a Marconi oven (model MA-035, Piracicaba/SP) with circulation and air renewal at $105\text{ }^{\circ}\text{C}$ for 5 h. After completing the drying process, the plate was weighed in an analytical balance, and the solubility was assessed by calculating the difference in weight.

The flowability and cohesiveness of the MJP were calculated using the Carr index and the Hausner ratio according to the method described by Jinapong, Suphantharika and Jamnong (2008). The flowability was calculated using the Carr index according to Eq. 3. The cohesiveness was assessed using the Hausner ratio calculated according to Eq. 4.

$$CI = \frac{(\rho_{abs} - \rho_{ap})}{\rho_{abs}} \times 100 \quad (3)$$

wherein ρ_{tapped} is the absolute density and ρ_{bulk} is the apparent density.

$$HR = \frac{\rho_{abs}}{\rho_{ap}} \quad (4)$$

The particle size of the MJP was assessed in a Microtrac FLEX 10.6.2 particle size analyzer by dynamic light scattering using water as the settling liquid. The morphology of the particles were obtained by MEV, samples were fixed in metal specimen holders (stubs) with conventional conductive double-sided adhesive tape. Then, they were plated with a gold alloy on a LEICA EM SCD 500 metallizer (Leica, Wetzlar, Germany). The samples were then observed in an FEI QUANTA 200F scanning electron microscope.

High-purity nitrogen gas adsorption using a liquid nitrogen bath under cryogenic conditions ($T = -196\text{ }^{\circ}\text{C}$), while applying the BET model, was the method used to assess the specific surface area and pore diameter. An Accelerated Surface Area and Porosimetry System, ASAP 2420 (Micromeritics), equipped with its own software was used to determine the surface area (SBET) and pore size and volume. The multipoint specific surface area was calculated from the adsorption isotherm fitted to the BET equation (BRUNAUER; EMMETT; TELLER, 1938). The total pore volume or cumulative volume ($\text{cm}^3 \cdot \text{g}^{-1}$) and mean pore diameter (nm) were determined at a relative pressure of 0.99. The pore size distribution was calculated using the BJH method from the adsorption isotherm.

The thermal analyses (TG/DSC) were performed in a thermogravimetric analyzer model Netzsch STA 449F3 calibrated with a reference standard of pure indium with 99.99% purity. The mass used in the analysis was 15.60 mg under a 50 mL min^{-1} synthetic air flow and heated from $30\text{ }^{\circ}\text{C}$ to $500\text{ }^{\circ}\text{C}$ at a $10\text{ }^{\circ}\text{C}/\text{min}$ heating rate.

The method described by Sanchez-Moreno, Larrauri and Saura-Calixto (1998) was used to determine the 2,2-diphenyl-1-picrylhydrazyl radical (DPPH) scavenging capacity. The DPPH analysis was performed in mixed juice powder (MJP) of acerola and seriguela at 0.02 g mL⁻¹, 0.04 g mL⁻¹ and 0.08 g mL⁻¹ before drying and at 0.04 g mL⁻¹, 0.08 g mL⁻¹ and 0.16 g mL⁻¹ after drying. Readings were performed at a 515 nm wavelength to monitor the reaction in a spectrophotometer (Shimadzu UV-1650PC) until stabilization. It was expressed as EC₅₀ which is defined as effective concentration of juice extract to decrease the initial DPPH concentration by 50%. The method described by Miller *et al.* (1993) was used to determine the antioxidant activity by 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) radical scavenging. The antioxidant activity was assessed at different concentrations of aqueous extract of MJP (13.3, 26.6 and 53.3 mg L⁻¹) and in mixed juice of acerola and seriguela before drying (6.6, 13.3 and 26.6 mg L⁻¹). Readings were performed after 6 min at a wavelength of 734 nm. Using the line equation, 1000 µM Trolox absorbance was calculated, and the results were expressed as µmol Trolox g⁻¹ sample on a dry basis. The antioxidant activity was performed before and after drying on the same day.

To determine the adsorption isotherms of MJP, the static-indirect method was used. Approximately 1 g of sample was weighed in a plastic capsule and placed in a desiccator with distilled water to create a saturated environment. The water activity was measured every 30 min at 10, 20, 30, 40 and 50 °C using Termoconstanter Novasina TH-200 equipment, where the samples remained until stable readings. After measuring the water activity, the samples were weighed to assess the water gain. The equilibrium moisture (X_{eq}) was assessed using Eq. 5:

$$X_{eq} = \frac{m_{eq} - m_s}{m_s} \quad (5)$$

where: X_{eq}: equilibrium moisture (g/g), dry basis; m_{eq}: equilibrium mass of the sample (g); and m_s: dry mass of the sample (g).

The experimental data of the water activity (a_w) and equilibrium moisture (X_{eq}) were fitted to six mathematical models (Table 1) to obtain the adsorption isotherms. The experimental data were modeled by non-linear regression using the Leven Berg Marquardt interaction algorithm with the Origin software version 8.1 (OriginLab).

Table 1 – Models used to define the isotherm behavior and respective equations

Models	Equations
Linearized Brunauer–Emmett–Teller- BET	$\frac{a_w}{(1-a_w)X_{eq}} = \frac{1}{X_m C_{BET}} + \frac{a_w(C_{BET}-1)}{X_m C_{BET}} \quad (6)$
Guggenheim-Anderson-de Boer-GAB:	$X_{eq} = \frac{X_m C K a_w}{(1-K a_w)(1-K a_w + C K a_w)} \quad (7)$
Henderson	$X_{eq} = \left[\frac{-\ln(1-a_w)}{b} \right]^{\frac{1}{a}} \quad (8)$
Oswin	$X_{eq} = A \left(\frac{a_w}{1-a_w} \right)^B \quad (9)$
Smith	$X_{eq} = k_1 + (k_2 \log(1 - a_w)) \quad (10)$

where: X_{eq}: equilibrium moisture, g H₂O g⁻¹; X_m: water content in the molecular monolayer, g H₂O g⁻¹; a_w: water activity; n: number of molecular layers; C, K: sorption constants; and a, b: adjustment parameters.

The goodness-of-fit of the experimental data was assessed for each mathematical model by calculating the coefficient of determination (R²) and the relative mean deviation (P). The relative mean deviation (Eq. 11) is commonly used in the literature as a selection criterion for an appropriate model (BARBOSA *et al.*, 2016).

$$P (\%) = \frac{100}{N} \sum_{i=1}^N \frac{|V_{exp} - V_p|}{V_{exp}} \quad (11)$$

The isosteric adsorption heat (q_{st,n}) was determined by linear regression using the Clausius–Clapeyron equation (Eq. 12), as proposed by Tsami (1991), where ln(a_w) is plotted as a function of 1/T and the value of -q_{st,n}/T is determined after regression. This value is multiplied by the gas constant (R) to assess the value of q_{st,n}. The effect of the equilibrium moisture on the isosteric heat of the MJP was shown by plotting the q_{st,n} versus the equilibrium moisture at which the q_{st,n} was determined.

$$\ln \ln (a_w) = - \frac{q_{st,n}}{R} \frac{1}{T} + cste \quad (12)$$

Where: a_w: water activity; R: gas constant; T: temperature and cste: constant.

3 Results and discussion

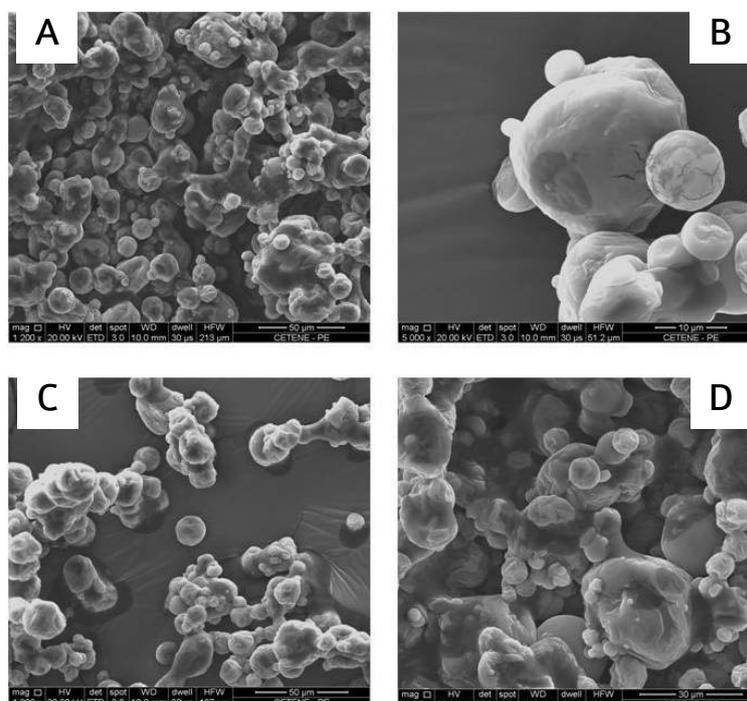
The MJP density affects its storage and stability and therefore its quality. Tontul and Topuz (2017) explain that the apparent density depends on the size, shape and surface of properties. The apparent density of the MJP was $0.16 \text{ g cm}^{-3} \pm 0.003$. This value was lower than that found by Santhalakshmy *et al.* (2015), who reported values ranging from 0.24 to 0.28 g cm^{-3} in jamun fruit juice powder. Those authors explain that a very low apparent density increases the likelihood of oxidation reactions due to the increased air occlusion and consequently decreases the shelf-life of the product. The absolute density ($1.03 \text{ g cm}^{-3} \pm 0.0002$) was higher than the apparent density. Mitra *et al.* (2017) flow and flow-related properties namely morphology, angle of repose, moisture sorption, glass transition and sticky point temperature of basundi mix powder were determined. The bulk and tapped densities decreased from 690.71 to 622.71 kg/m^3 and 819.10 to 729.31 kg/m^3 , respectively as moisture content increased from 3 to 9% (d.b. describe that a high value of the absolute density and a low apparent density allowing smaller particles to fit into the voids left by the larger particles, most likely due to repulsive forces between particles with higher moisture content.

The porosity affects powder stability. The porosity of the MJP was $84.5\% \pm 0.003$. The value of porosity assessed in this study was similar to that found by Islam *et al.* (2017). High porosity values indicate the increased likelihood of oxidation reactions. Solubility is a physical property that affects the juice powder quality because it affects juice powder reconstitution. The solubility of the juice was $79.4\% \pm 0.68$. Similar findings were reported by Santhalakshmy *et al.* (2015) when assessing the effect of the inlet temperature on the spray drying process of jamun juice. Those authors found solubility values ranging from 87.7 to 99.7%.

The Carr index and Hausner index were $87.5\% \pm 0.34$ and $6.4\% \pm 0.14$, respectively. The high Carr index indicates that the flowability of MJP was very poor, and the high Hausner ratio indicates that the juice had high cohesiveness. A similar result was found by Santhalakshmy *et al.* (2015).

Figures 1A and 1B show that the particles were spherical, with various sizes and agglomerated. These results are in line with the findings of Islam *et al.* (2016) when assessing the effect of the maltodextrin concentration on the morphology of orange juice powder. They observed that the lower the maltodextrin concentration is, the higher the tendency to agglomerate into larger particles. Ferrari *et al.* (2012) explain that the higher this agglomeration

Figure 1 – Micrographs of particles of MJP



where: A: Overview of the distribution of particles as agglomerates (1200x); B: Smaller particles over smaller particles (5000x); C: Details of the agglomerate formed by isolated and agglomerated particles of different sizes (1300x); D: Particles with rough and smooth surfaces (2400x).

or adherence is, the lower the powder exposure to oxygen will be, thereby avoiding oxidation reactions. These irregularities observed on the surface of particles, including roughness with cavities and surface cracks are possibly due to the process of removing water during the spray drying.

The particle diameters ranged from 0.57 μm to 209 μm , with a mean diameter of $19.63 \pm 5.93 \mu\text{m}$, and showed a unimodal distribution. The mean particle diameter found in this study is similar to that found by Negrão-Murakami *et al.* (2017) which is a rich source of phenolic compounds, was microencapsulated by spray drying using three different DE (dextrose equivalent when assessing the effect of the dextrose-equivalent (DE) value of maltodextrin on the spray-dried concentrated mate.

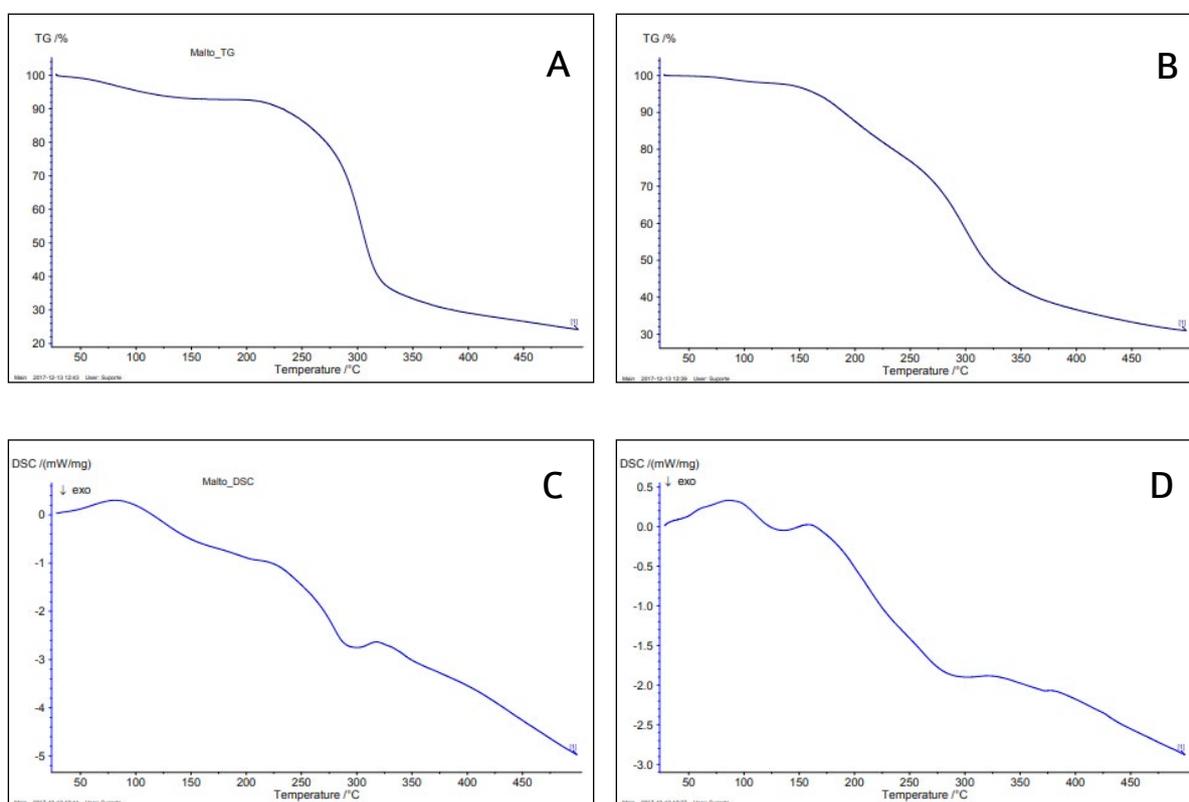
The BET equation was used to calculate the surface area. Sun-Waterhouse and Waterhouse (2015) found BET values of $0.99 \text{ m}^2 \text{ g}^{-1}$ for green kiwi juice powder and $0.86 \text{ m}^2 \text{ g}^{-1}$ for yellow kiwi juice powder. Those values were higher than that found in this study, which was $0.66 \text{ m}^2 \text{ g}^{-1}$. The value found in this study for the BJH adsorption pore diameter was 48.2 nm, which is classified as a mesopore. Pores with diameters ranging

from 2 nm to 50 nm are classified as mesopores (SILVA *et al.*, 2019). The volume of micropores in the present study was $0.0087 \text{ cm}^3 \text{ g}^{-1}$, which is considerably lower than that found by Hidayat and Sutrisno (2016), which ranged from 0.15 to $0.24 \text{ cm}^3 \text{ g}^{-1}$.

Figure 2A shows three thermal events for maltodextrin 10DE. The first thermal event occurred in the temperature range from 36 to 186 $^{\circ}\text{C}$ and is related to a 6.9% water loss. The second thermal event corresponds to a more pronounced mass loss of 61.7% in the temperature range from 205 to 371 $^{\circ}\text{C}$. This loss of mass is likely due to the reaction of the cleavage of long molecular chains and from isomerizations associated with dehydration (LIU *et al.*, 2013). The last thermal event occurred from 371 to 493 $^{\circ}\text{C}$ with a 6.3% mass loss. According to Otálora *et al.* (2015), this loss results from the volatilization of decomposed materials.

The TG curve of the MJP in Figure 2B shows four stages of mass loss, and the largest loss occurred in the temperature range from 205 to 371 $^{\circ}\text{C}$ with 61.7%. These results are in line with the findings of Bisinella *et al.* (2016). These authors attribute this loss of mass to dehydration reactions at the beginning and to sample decomposition and organic acid oxidation reactions.

Figure 2 – Thermogravimetric curves (TG/DSC) of maltodextrin 10DE and MJP. A) TG of maltodextrin 10DE; B) TG of MJP; C) DSC of maltodextrin 10DE; D) DSC of MJP



The DSC curve of maltodextrin 10DE in Figure 2C shows two transitions: an exothermic and an endothermic transition with peak temperatures of 88 °C of 296 °C, respectively. Nunes *et al.* (2015) 30%, and 40% found two endothermic peaks at temperatures ranging from 196 °C to 205 °C. These authors attribute these peaks to the melting point displacement of *Ilex paraguariensis* extract microcapsules. Bisinella *et al.* (2016) explain that this exothermic transition is probably due to the maltodextrin crystallization process. Conversely, the MJP showed two endothermic transitions at peak temperatures of 131 °C and 292 °C. Villacrez, Carriazo and Osorio (2014) explain that the endothermic peak results from the process termed

gelatinization, which occurs when starch is heated in an aqueous environment.

The glass transition temperature of the medium is 67.2 °C for maltodextrin 10DE and 173.2 °C for the MJP. Therefore, the addition of maltodextrin 10DE to MJP increased the transition temperature. Negrão-Murakami *et al.* (2017), when studying the effect of maltodextrin DE values on the glass transition temperature of mate concentrate, observed that the thermal stability of the concentrate increased with the addition of maltodextrin 10DE.

The EC50 of 6.97 ± 0.01 significantly increased after drying process which means that there was a reduction of the antioxidant action before drying which

Table 2 – Parameters used to fit the mathematical models to the adsorption isotherms of MJP at different temperatures

Parameters						
Model	T (°C)	X _m	C	K	R ²	P (%)
GAB	10	1.817	0.053	0.657	0.959	19.56
	20	0.055	2.432	1.012	0.997	6.05
	30	0.068	1.457	0.991	0.998	4.39
	40	0.054	2.784	1.016	0.998	4.61
	50	0.084	0.593	0.975	0.994	3.84
Linearized BET*		X _m	C _{BET}			
	20	0.0663	1.2097		0.9952	7.56
	30	0.0624	2.0399		0.9982	4.36
	40	0.0649	1.5222		0.9972	4.93
	50	0.0626	1.2003		0.9966	4.06
Henderson		a	b			
	10	1.0113	8.1054		0.9063	29.54
	20	0.4581	2.8792		0.9785	18.38
	30	0.5566	3.157		0.9925	10.65
	40	0.5359	3.0540		0.9885	15.74
Oswin		A	B			
	10	0.1105	0.3928		0.8225	39.89
	20	0.0682	0.9960		0.9949	8.21
	30	0.0806	0.9034		0.9980	4.47
	40	0.0756	0.9461		0.9966	5.63
Smith		k ₁	k ₂			
	10	-0.0135	0.1326		0.9100	17.88
	20	-0.1262	0.2921		0.8822	20.68
	30	-0.0795	0.2499		0.9309	18.07
	40	-0.0794	0.2522		0.9017	27.71
50	-0.0854	0.2474		0.9381	4.48	

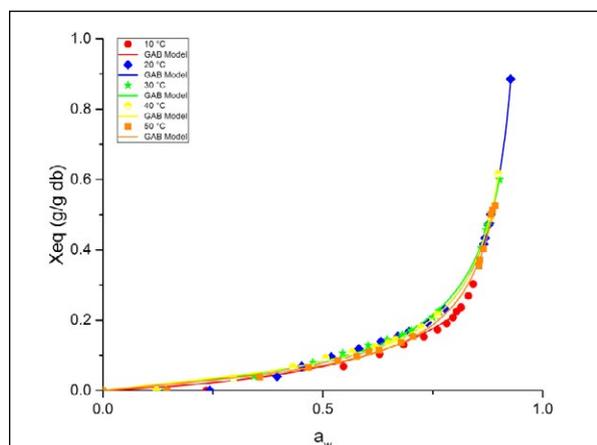
where: X_m: moisture content in the molecular monolayer (g·g⁻¹ in a dry basis); C: BET's constant, related to the sorption heat in the molecular layer; K: sorption constant; a and b: adjustment parameters; R²: coefficient of determination; P: the values of relative mean error. * The BET 10 °C was unable to adjust the experimental data for this isotherm in this model.

showed EC50 of 0.05 ± 0.03 . Medina-Torres *et al.* (2016) explain that the feed rate used in drying process can also contribute to this reduction since it is related to the contact time of the material with hot air.

Saikia, Mahnot and Mahanta (2015) also found decreased antioxidant activity in watermelon juice powder. These authors explained that changes in the antioxidant activity and in phenolic compounds depend on the individual constituents of phenolic acids. Mixed juice of acerola and seriguela showed antioxidant activity of $35.74 \mu\text{mol Trolox g}^{-1} \pm 1.01$ and $441.05 \mu\text{mol Trolox g}^{-1}$ (bs) ± 0.75 , respectively, before and after drying process according to the ABTS method.

The results outlined in Table 2 (previous page) show that the mathematical models best fitted to the adsorption curve of the MJP were the GAB, linearized BET and Oswin models at 20 °C to 50 °C temperatures because they had a percentage of deviation lower than 10% and a R^2 close to 1. However, at 10 °C, they presented a relative average error (P) above 10% for all models, indicating that they are not suitable for this temperature. Those results corroborate the findings of Islam *et al.* (2016) when analyzing the adsorption isotherm of orange juice powder. The authors explain that the behavior is typical of a sample with a high content of sugar and a type III curve (J type). It should be noted that seriguela pulp has higher sugar content than acerola, thus causing this type of behavior, as shown in Figure 3.

Figure 3 – Adsorption isotherms of MJP at 10, 20, 30, 40 and 50 °C temperatures fitted using the GAB model

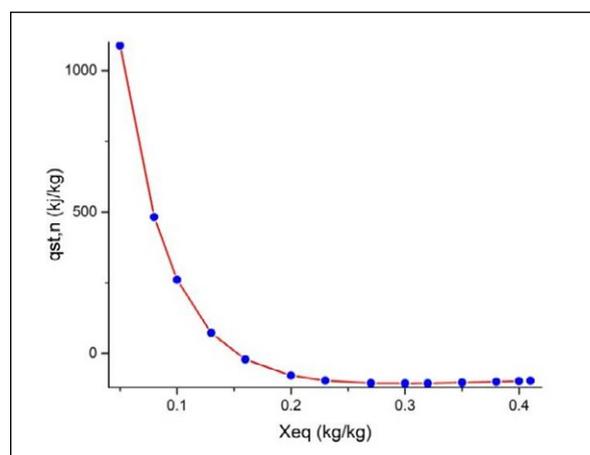


where: Xeq: equilibrium moisture, $\text{g H}_2\text{O g}^{-1}$; aw: water activity.

The values of X_m , which is the quantity of water adsorbed in the monolayer, ranged from 0.084 to 0.054 for the GAB model and from 0.0663 to 0.0624 for the linearized BET model at 20, 30, 40 and 50 °C temperatures. These values are important to assess the stability of the MJP.

As shown in Figure 4, the isosteric heat decreased with the increase in the moisture content. A similar behavior was observed by Aksil *et al.* (2019) the experimental sorption curves of lyophilized Arbutus unedo L. fruit powder (LP which explain that the high isosteric heat at the beginning points to the presence of highly active polar zones on the surfaces of the powder that were filled by a layer of monomolecular water. This reveals that the presence of these polar zones on the surface reduce the mobility of water from within.

Figure 4 – Isosteric adsorption heat as a function of the equilibrium moisture of MJP



where: Xeq: equilibrium moisture, $\text{g H}_2\text{O g}^{-1}$;
qst: integral isosteric heat

4 Conclusions

The adsorption isotherms, pore distribution and thermal properties of MJP were investigated to determine the best conditions to storage this powder juice. Physicochemical properties and antioxidant activity were also determined to characterize the powder juice.

The low apparent density and high porosity indicate that MJP should be stored in vacuum packaging to avoid oxidation reactions. The MEV showed an irregular surface morphology and shape. The BJH adsorption means pore diameter distribution and allows to classify pore sizes as mesopores.

However, this information is very useful for the quality control of food because it affects properties such as wettability and its oxidative stability.

The MJP is a good source of antioxidants and may be used by the food industry as a functional food ingredient. Thermal analysis (TG) showed high thermal stability for MJP, while the DSC revealed that at very high temperatures underwent gelatinization due to the presence of maltodextrin.

The MJP isotherms are well adjusted in the GAB, BET linearized and Oswin models at temperatures 20 to 50 °C and are characterized as type III. The determination of the moisture adsorption isotherms constitutes important information for the processing of powdered juices, since it helps to determine the type of packaging to be used, the conditions of storage and to predict their stability.

Given the above, it can be concluded that acerola and seriguela mixed juice powder presented rapid reconstitution, thermal stability and good antioxidant capacity.

ACKNOWLEDGMENTS

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior – Brasil (CAPES) – Finance Code 001. Authors are also acknowledged with the National Institute of Science and Technology of Tropical Fruits and CNPq for the financial support.

REFERENCES

AKSIL, T. *et al.* Water adsorption on lyophilized *Arbutus unedo* L. fruit powder: Determination of thermodynamic parameters. **Microchemical Journal**, v. 145, p. 35–41, 2019. DOI: 10.1016/j.microc.2018.10.012. Available from: <https://www.sciencedirect.com/science/article/abs/pii/S0026265X18307227>. Access on: 20 jan. 2017.

BARBOSA, K. F. *et al.* Desorption isotherms and isosteric heat of “cajuzinho-do-cerrado” achenes. **Revista Brasileira de Engenharia Agrícola e Ambiental**, v. 20, n. 5, p. 481–486, 2016. DOI: 10.1590/18071929/agriambi.v20n5p481-486. Available from: http://www.scielo.br/scielo.php?script=sci_arttext&pid=S14154366201600050481&lng=en&nrm=iso&tlng=en. Access on: 26 apr. 2017.

BISINELLA, R. Z. B. *et al.* Thermal analysis as screening technique to assess spray-drying process of encapsulated “yacon” juice. **Journal of Thermal Analysis and Calorimetry**, v. 126, n. 3, p. 1841–1849, 2016. DOI: 10.1007/s10973-016-5696-z. Available

from: <https://link.springer.com/article/10.1007/s10973-016-5696-z>. Access on: 26 apr. 2017.

BRUNAUER, S.; EMMETT, P.; TELLER, E. Adsorption of gases in multimolecular layers. **Journal of American Chemical Society**, v. 60, n. 2, p. 309–319, 1938. DOI: 10.1021/ja01269a023. Available from: <https://pubs.acs.org/doi/abs/10.1021/ja01269a023>. Access on: 25 apr. 2018.

CANO-CHAUCA, M. *et al.* Effect of the carriers on the microstructure of mango powder obtained by spray drying and its functional characterization. **Innovative Food Science and Emerging Technologies**, v. 6, n. 4, p. 420–428, 2005. DOI: 10.1016/j.ifset.2005.05.003. Available from: <https://www.sciencedirect.com/science/article/pii/S1466856405000834>. Access on: 25 apr. 2017.

CAPARINO, O. A. *et al.* Effect of drying methods on the physical properties and microstructures of mango (Philippine “Carabao” var.) powder. **Journal of Food Engineering**, v. 111, n. 1, p. 135–148, 2012. DOI: 10.1016/j.jfoodeng.2012.01.010 Available from: <https://www.sciencedirect.com/science/article/abs/pii/S0260877412000301>. Access on: 25 apr. 2017.

CAVALCANTE, M. D. *et al.* Isotherms and isostatic heat of foam-mat dried yellow mombin pulp. **Revista Brasileira de Engenharia Agrícola e Ambiental**, v. 22, n. 6, p. 436–441, 2018. DOI: 10.1590/1807-1929/agriambi.v22n6p436-441. Available from: http://www.scielo.br/scielo.php?script=sci_arttext&pid=S1415-43662018000600436&lng=en&nrm=iso. Access on: 18 feb. 2019.

CONEGERO, J. *et al.* Hygroscopic trend of lyophilized ‘mangaba’ pulp powder. **Revista Brasileira de Engenharia Agrícola e Ambiental**, v. 21, n. 5, p. 356–360, 2017. DOI: 10.1590/1807-1929/agriambi.v21n5p356-360. Available from: http://www.scielo.br/scielo.php?script=sci_arttext&pid=S1415-43662017000500356&nrm=iso. Access on: 26 apr. 2017.

FERRARI, C. C. *et al.* Influence of carrier agents on the physicochemical properties of blackberry powder produced by spray drying. **International Journal of Food Science and Technology**, v. 47, n. 6, p. 1237–1245, 2012. DOI: 10.1111/j.1365-2621.2012.02964. Available from: <https://ifst.onlinelibrary.wiley.com/doi/abs/10.1111/j.1365-2621.2012.02964.x>. Access on: 18 feb. 2018.

HIDAYAT, A.; SUTRISNO, B. Comparison on pore development of activated carbon produced by chemical and physical activation from palm empty

fruit bunch. **IOP Conference Series: Materials Science and Engineering**, v. 162, n. 1, 2016. DOI: 10.1088/1757-899X/162/1/012008. Available from: <https://iopscience.iop.org/article/10.1088/1757-899X/162/1/012008>. Access on: 15 jul. 2018.

ISLAM, M. Z. *et al.* Effect of vacuum spray drying on the physicochemical properties, water sorption and glass transition phenomenon of orange juice powder. **Journal of Food Engineering**, v. 169, p. 131–140, 2016. DOI: 10.1016/j.jfoodeng.2015.08.024. Available from: <http://www.sciencedirect.com/science/article/pii/S0260877415003775>. Access on: 21 nov. 2015.

ISLAM, M. Z. *et al.* Effects of micro wet milling and vacuum spray drying on the physicochemical and antioxidant properties of orange (*Citrus unshiu*) juice with pulp powder. **Food and Bioprocess Technology**, v. 101, p. 132–144, 2017. DOI: 10.1016/j.fbp.2016.11.002. Available from: <https://www.sciencedirect.com/science/article/abs/pii/S0960308516301535>. Access on: 25 oct. 2019.

JINAPONG, N.; SUPHANTHARIKA, M.; JAMNONG, P. Production of instant soymilk powders by ultrafiltration, spray drying and fluidized bed agglomeration. **Journal of Food Engineering**, v. 84, n. 2, p. 194–205, 2008. DOI: 10.1016/j.jfoodeng.2007.04.032. Available from: <https://www.sciencedirect.com/science/article/abs/pii/S0260877407002853>. Access on: 25 oct. 2018.

LIU, X. *et al.* Thermal degradation and stability of starch under different processing conditions. **Starch/Staerke**, v. 65, n. 1–2, p. 48–60, 2013. DOI: 10.1002/star.201200198. Available from: <https://onlinelibrary.wiley.com/doi/abs/10.1002/star.201200198>. Access on: 25 oct. 2018.

MALDONADO-ASTUDILLO, Y. I. *et al.* Postharvest physiology and technology of *Spondias purpurea* L. and *S. mombin* L. **Scientia Horticulturae**, v. 174, p. 193–206, 2014. DOI: 10.1016/j.scienta.2014.05.016. Available from: <http://www.sciencedirect.com/science/article/pii/S0304423814002799>. Access on: 18 feb. 2016.

MEDINA-TORRES, L. *et al.* Microencapsulation by spray drying of laurel infusions (*Litsea glaucescens*) with maltodextrin. **Industrial Crops and Products**, v. 90, p. 1–8, 2016. DOI: 10.1016/j.indcrop.2016.06.009. Available from: <http://linkinghub.elsevier.com/retrieve/pii/S0926669016303934>. Access on: 7 aug. 2017.

MILLER, N. J. *et al.* A novel method for measuring antioxidant capacity and its application to monitoring the antioxidant status in premature neonates. **Clinical Science**, v. 84, n. 4, p. 407–412, 1993. DOI: 10.1042/

cs0840407. Available from: <https://www.ncbi.nlm.nih.gov/pubmed/8482045>. Access on: 18 apr. 2017.

MITRA, H. *et al.* Influence of moisture content on the flow properties of *basundi* mix. **Powder Technology**, v. 312, p. 133–143, 2017. DOI: 10.1016/j.powtec.2017.02.039. Available from: <https://www.sciencedirect.com/science/article/abs/pii/S0032591017301675>. Access on: 25 oct. 2018.

MORAES, F. P. *et al.* Estimation of Ascorbic Acid in Intact Acerola (*Malpighia emarginata* DC) Fruit by NIRS and Chemometric Analysis. **Horticulturae**, v. 5, n. 12, p. 1–10, 2019. DOI: 10.3390/horticulturae5010012. Available from: <https://www.mdpi.com/2311-7524/5/1/12>. Access on: 15 jul. 2019.

NEGRÃO-MURAKAMI, A. N. *et al.* Influence of DE-value of maltodextrin on the physicochemical properties, antioxidant activity, and storage stability of spray dried concentrated mate (*Ilex paraguariensis* A. St. Hil.). **LWT - Food Science and Technology**, v. 79, p. 561–567, 2017. DOI: 10.1016/j.lwt.2016.11.002. Available from: <http://linkinghub.elsevier.com/retrieve/pii/S0023643816306739>. Access on: 9 aug. 2017.

NUNES, G. L. *et al.* Microencapsulation of freeze concentrated *Ilex paraguariensis* extract by spray drying. **Journal of Food Engineering**, v. 151, p. 60–68, 2015. DOI: 10.1016/j.jfoodeng.2014.10.031. Available from: <https://www.sciencedirect.com/science/article/abs/pii/S0260877414004993>. Access on: 9 aug. 2017.

OTÁLORA, M. C. *et al.* Microencapsulation of betalains obtained from cactus fruit (*Opuntia ficus-indica*) by spray drying using cactus cladode mucilage and maltodextrin as encapsulating agents. **Food Chemistry**, v. 187, p. 174–181, 2015. DOI: 10.1016/j.foodchem.2015.04.090. Available from: <https://www.sciencedirect.com/science/article/abs/pii/S0308814615006366>. Access on: 9 aug. 2017.

RIBEIRO, L. C.; COSTA, J. M. C.; AFONSO, M. R. A. Hygroscopic behavior of acerola powder obtained by spray-drying. **Acta Scientiarum - Technology**, v. 41, p. 1–9, 2019. DOI: 10.4025/actascitechnol.v41i1.35382. Available from: <http://periodicos.uem.br/ojs/index.php/ActaSciTechnol/article/view/35382>. Access on: 9 jul. 2019.

RIBEIRO, C. M. C. M. *et al.* Optimization of the spray drying process conditions for acerola and seriguela juice mix. **Food Science and Technology**, v. 39, supl. 1, p. 48–55, 2019. DOI: 10.1590/fst.36217. Available from: http://www.scielo.br/scielo.php?script=sci_arttext&pid=S0101-20612019000500048&lng=en&nrm=iso. Access on: 25 apr. 2019.

- SAIKIA, S.; MAHNOT, N. K.; MAHANTA, C. L. Effect of Spray Drying of Four Fruit Juices on Physicochemical, Phytochemical and Antioxidant Properties. **Journal of Food Processing and Preservation**, v. 39, n. 6, p. 1656–1664, 2015. DOI: 10.1111/jfpp.12395. Available from: <https://ifst.onlinelibrary.wiley.com/doi/abs/10.1111/jfpp.12395>. Access on: 25 apr. 2019.
- SANCHEZ-MORENO, C.; LARRAURI, J. A.; SAURA-CALIXTO, F. A procedure to measure the antiradical efficiency of polyphenols. **Journal of the Science of Food and Agriculture**, v. 76, n. 2, p. 270–276, 1998. DOI: 10.1002/(SICI)1097-0010(199802)76:2<270::AID-JSFA945>3.0.CO;2-9. Available from: [https://onlinelibrary.wiley.com/doi/abs/10.1002/\(SICI\)1097-0010\(199802\)76:2%3C270::AID-JSFA945%3E3.0.CO;2-9](https://onlinelibrary.wiley.com/doi/abs/10.1002/(SICI)1097-0010(199802)76:2%3C270::AID-JSFA945%3E3.0.CO;2-9). Access on: 25 apr. 2019.
- SANTHALAKSHMY, S.; DON BOSCO, S. J.; FRANCIS, S.; SABEENA, M. Effect of inlet temperature on physicochemical properties of spray-dried jamun fruit juice powder. **Powder Technology**, v. 274, p. 37–43, 2015. DOI: 10.1016/j.powtec.2015.01.016. Available from: <https://www.sciencedirect.com/science/article/abs/pii/S0032591015000303>. Access on: 9 jul. 2019.
- SHAO, P. *et al.* Encapsulation efficiency and controlled release of Ganoderma lucidum polysaccharide microcapsules by spray drying using different combinations of wall materials. **International Journal of Biological Macromolecules**, v. 125, p. 962–969, 2019. DOI: 10.1016/j.ijbiomac.2018.12.153. Available from: <https://www.ncbi.nlm.nih.gov/pubmed/30572060>. Access on: 9 jul. 2019.
- SILVA, I. C. *et al.* Caracterização, tratamento e utilização do carvão ativado para adequação de parâmetros físico-químicos de efluentes oleosos através da adsorção em banho finito. *Revista Principia - Divulgação Científica e Tecnológica do IFPB*, n. 45, p. 171–179, 2019. DOI: 10.18265/1517-03062015v1n45p171-179. Available from: <https://periodicos.ifpb.edu.br/index.php/principia/article/view/2784>. Access on: 15 jan. 2019.
- SILVA FILHO, E. *et al.* Modelagem matemática para descrição da cinética de secagem do caldo de cana *in natura*. *Revista Principia - Divulgação Científica e Tecnológica do IFPB*, n. 40, p. 28–34, 2018. DOI: 10.18265/1517-03062015v1n40p28-34. Available from: <https://periodicos.ifpb.edu.br/index.php/principia/article/view/1306>. Access on: 15 apr. 2019.
- SOUZA, A. S. *et al.* Influence of spray drying conditions on the physical properties of dried pulp tomato. **Ciência e Tecnologia de Alimentos**, v. 29, n. 2, p. 291–294, 2009. DOI: 10.1590/S0101-20612009000200008. Available from: http://www.scielo.br/scielo.php?script=sci_arttext&pid=S0101-206120090. Access on: 15 apr. 2018.
- SUN-WATERHOUSE, D.; WATERHOUSE, G. I. N. Spray-Drying of Green or Gold Kiwifruit Juice–Milk Mixtures; Novel Formulations and Processes to Retain Natural Fruit Colour and Antioxidants. **Food and Bioprocess Technology**, v. 8, n. 1, p. 191–207, 2015. DOI: 10.1007/s11947-014-1397-4. Available from: <https://link.springer.com/article/10.1007/s11947-014-1397-4>. Access on: 25 apr. 2019.
- TONTUL, I.; TOPUZ, A. Spray-drying of fruit and vegetable juices: Effect of drying conditions on the product yield and physical properties. **Trends in Food Science and Technology**, v. 63, p. 91–102, 2017. DOI: 10.1016/j.tifs.2017.03.009. Available from: <https://www.sciencedirect.com/science/article/abs/pii/S0924224417300729>. Access on: 25 apr. 2019.
- TSAMI, E. Net isosteric heat of sorption in dried fruits. **Journal of Food Engineering**, v. 14, n. 4, p. 327–335, 1991. DOI: 10.1016/0260-8774(91)90022-K. Available from: <https://www.sciencedirect.com/science/article/abs/pii/S026087749190022K>. Access on: 15 apr. 2018.
- VILLACREZ, J. L.; CARRIAZO, J. G.; OSORIO, C. Microencapsulation of Andes Berry (*Rubus glaucus* Benth.) Aqueous Extract by Spray Drying. **Food and Bioprocess Technology**, v. 7, n. 5, p. 1445–1456, 2014. DOI: 10.1007/s11947-013-1172-y. Available from: <https://link.springer.com/article/10.1007/s11947-013-1172-y>. Access on: 15 apr. 2018.