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Influence of process variables on the drum drying of mango pulp

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ABSTRACT

Response surface methodology was used to evaluate the best process conditions in the drum drying of mango pulp regarding the physicochemical properties and nutrients content. The independent variables in the central composite rotatable design were: temperature (119–151°C) and residence time (9–41 s), while the concentrations of process additives used were: 3% corn starch and 0.5% glyceryl monostearate. Mathematical models for moisture content, total carotenoid content, and color parameters of the reconstituted pulp (a^*, Hue, and ΔE) were obtained. The optimized conditions were combinations of residence time between 10 and 25 s, and temperature from 120 to 135°C, in which the flakes showed moisture content ranging from 2 to 5%, besides a more intense yellow color and total carotenoid retention between 90 and 96%.

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Introduction

Food industry has been guiding its activities in response to a growing demand for healthier, clean label, and natural products. In this context, fruits have been used as macroingredients in new formulations, attending the current consumer demand related to healthiness and naturalness.[1] Furthermore, the addition of fruits gives quality aspects, such as sweetness, texture, color, flavor, fiber content, vitamins, and antioxidants, and can also result in the minimization of the use of additives.[2,3]

Fruits in the form of dehydrated powder, or flakes, are more conveniently used by the industry due to their easier transport, storage and standardization. However, the production of fruit powders/flakes presents some important technological challenges. The main one is the low stability during the processing and the storage of the dry product due to the low glass transition temperatures (T_g), since ripe fruits have high concentrations of sugars and organic acids with low molecular weight.[4] This fact results in high stickiness and hygroscopicity of the powdered/flaked fruits. As an alternative, a high concentration of additives, such as starches, dextrins, and gums, varying from 40 to 60% (dry basis), are used in the spray drying process, which is the most commercially used technique for powder production.[4–6] These additives have high molecular weight, increasing T_g values and fruit stability regarding the moisture absorption.

Therefore, drum drying is an alternative for the production of fruit and vegetables powders/flakes, since it usually requires a smaller amount of additives. Pua et al.[7] used around 3% soy lecithin and 10% gum arabic for the production of drum dried jackfruit powder. Setyadjit and Sukashih[8] worked with 10% cassava starch and 1% maltodextrin in the drum drying of shallot puree, while Sonthipermpoon et al.[9] used approximately 1–3% maltodextrin in a study with drum drying of banana pulp.

Despain[10] stated that drum drying is suitable for fruit powders, increasing their cost-effectiveness without compromising quality. The nutrients retention in the process, as well as the powder solubility and their stability throughout the storage, are important aspects that stand out in the technology.[3,11,12] The drum drying shows operational benefits, such as high drying rates, high energy efficiency, flexibility, and production profitability.[13–14] However, in spite of short processing times, high temperatures in drum drying may promote browning and flavor changes in the final product.

The drum dryer consists of a metal cylinder with horizontal axis internally heated with saturated steam. The equipment can be provided with a single or double cylinder. In the different types, the cylinders rotate at variable speeds, and the drying is performed by the application of a thin film of the raw material in the

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liquid or paste form. The removal of the film is done through the use of a scraper blade or knife, longitudinally positioned. According to the design of the equipment, the performance depends mostly on the following variables: pool level, process temperature, residence time (rotation speed of the cylinder), and feed concentration.[15] Drum drying is widely used for the drying of purees and paste-like products, as well as in the processing of bakery products, cereals and dairy products.[14] Nevertheless, some studies also point out the technical feasibility of drum drying in the production of fruit powders or flakes, such as banana,[9] jackfruit,[7] tamarind,[16] and goldenberry.[17]

Mango is a very appreciated fruit due to its exotic flavor. Moreover, it is a nutritious food, a source of carotenoids, fibers, and minerals, with considerable concentration of vitamins and total polyphenols, as well as antioxidant compounds.[18–20] Caparino et al.[21] compared different drying methods of mango pulp (refractance window, freeze-drying, and drum drying). The authors observed quality losses during the drum drying, but they pointed out the high solubility in water of the final product. Although the process temperature was mild (105°C), the residence time used in the study was high (52 s). Besides, no process additives were used, which made it difficult the formation of the dry film. Tonin et al.[22], in a study evaluating the performance of different additives, reported that the use of 3% corn starch and 0.5% glyceryl monostearate (GMS) in the drum drying of mango pulp (150°C/15 s) resulted in powders with good retention of vitamin C (61%), total carotenoids (98%), and β-carotene (90%). Travaglini et al.,[23] in a similar work with drum drying of mango puree with 4% starch and 1% GMS, varied the retention time conditions (10–40 s) and the process temperature (130–150°C), fixing the other variables. The authors determined the process yield and the final moisture content, as well as other powder properties. However, as they did not use an experimental design, the process optimization was not performed.

In this context, the purpose of this work was to evaluate the effect of the variables in the drum drying of mango pulp, in particular the temperature and the residence time, in terms of the drying parameters and the quality retention of the raw material, to determine the best process conditions.

Materials and methods

Materials

Frozen whole mango pulp, produced with Tommy Atkins and Ubá varieties (approximately 1:1), was obtained directly from the manufacturer (De Marchi, Campinas, Brazil). The physicochemical properties of the raw material are presented in Table 1. The drum drying additives were: corn starch (Amisol 3408, Ingredion, Mogi Guaçu, Brazil) and glyceryl monostearate (Synth, São Paulo, Brazil).

Experimental design

The experiments were performed according to a central composite rotatable design, based on the response surface methodology.[30] The factors (independent variables) used were residence time (t) and process temperature (T), while their levels were chosen from the results obtained in preliminary tests.

The tests were performed in a random manner, using in each one approximately 25 kg of mango pulp. Based on the previous study,[22] 3% corn starch and 0.5% glyceryl monostearate were used as process additives. The pulp was thawed at room temperature (approximately 25°C), formulated with the additives and homogenized in a colloid mill (Meteor, REX 2-AL, São Paulo, Brazil).

Drying tests were performed in a pilot drum dryer (Richard Simon & Sons, D139, Nottingham, England), provided with a single cylinder, with indirect heating by saturated steam, and an applicator cylinder, containing a total drying area of approximately 0.5 m^2. The other drum drying variables were set: clearance of 0.15 mm; pool level of 10 mm (±400 mL). The dried product, obtained as a film, was flocculated in a flocculator (Fabbe, S508, São Paulo, Brazil), using a 2.5 mm

Table 1. Physicochemical properties of the mango pulp used in this study.

Property	Mean value ± SD	Method
Moisture content (%)	85.78 ± 0.01	Instituto Adolfo Lutz[24]
Soluble solids (°Brix)	13.25 ± 0.10	Instituto Adolfo Lutz[24]
pH	4.28 ± 0.03	Instituto Adolfo Lutz[24]
Titratable acidity (g citric acid/100 g)	0.36 ± 0.03	Instituto Adolfo Lutz[24]
Vitamin C (mg/100 g db)	47.81 ± 0.98	Instituto Adolfo Lutz[24]
Antioxidant activity DPPH (μmol TE/g db)	42.61 ± 1.12	Brand-Williams et al.[25]
Antioxidant activity ABTS (μmol TE/g db)	73.27 ± 0.49	Rufino et al.[26]
Total phenolic compounds (mg GAE/100 g db)	606.24 ± 0.07	Benvenuti et al.[27]
Total carotenoids (mg/100 g db)	16.24 ± 0.42	Carvalho et al.[28]
β-Carotene (mg/100 g db)	6.47 ± 0.56	Carvalho et al.[28]
Reducing sugar (g/100 g db)	23.20 ± 1.54	Carvalho et al.[29]
Nonreducing sugars (sucrose) (g/100 g db)	64.20	Carvalho et al.[29]
Total sugars (g/100 g db)	87.48	Carvalho et al.[29]
Color parameter L^*	46.43 ± 0.54	CIELAB Color System
Color parameter a^*	2.64 ± 0.17	CIELAB Color System
Color parameter b^*	40.36 ± 1.64	CIELAB Color System

Each value represents the mean of three replicates ± standard deviation. db, dry basis.

sieve. Then, the products were separated in adequate amounts for the analyses and frozen at −18°C. Low-density polyethylene (LDPE) packaging (0.15 mm thick) was used for each sample, and they were stored in polyester/aluminum/low-density polyethylene (PET/Al/LDPE) bags, with 70 µm nominal thickness. The outer bags were sealed in a vacuum sealer (P300 MCB01 218021, Multivac, São Paulo, Brazil). Mass flow rate (MFR), expressed as (kg dry product/h m^2), was determined by collecting and weighing the dry material, at intervals of 5 m, according to Equation (1). The measurements were performed in quadruplicate

$$\mathrm{MFR} = \frac{m_{\mathrm{ps}}}{A} \times \frac{60}{\Delta t} \tag{1}$$

where m_{ps} = mass of dry product obtained (kg); Δt = time interval (min); and A = cylinder drying area (m^2).

Analytical methods

Mango flakes obtained in the different drying experiments were subjected to the following analyses: moisture content, water activity, total phenolic compounds, vitamin C, total carotenoids, and β-carotene content, antioxidant activity (DPPH and ABTS methods). The percentage of nutrient retention (vitamin C, total phenolic compounds, total carotenoids, and β-carotene) was calculated from the concentrations determined in the flakes and the raw material, both on dry basis. The flakes were also analyzed with respect to: hygroscopicity, solubility, wettability, color, particle size distribution, and scanning electron microscopy, according to the following methods.

Moisture content

The moisture content of the mango flakes was determined gravimetrically. Samples were weighed and dried in a vacuum oven at 70°C for 24 h.

Water activity

Water activity was evaluated using a digital hygrometer (Aqualab 3 TE, Decagon Devices Inc, Pullman, USA) at 25°C.

Solubility

This analysis was done according to Cano-Chauca et al.[31] About 1 g of the sample was added to a centrifuge vessel containing 100 mL of distilled water, operating at high speed for 5 min. Then, the solution was centrifuged at 3000× g, during 5 min. An aliquot of 25 mL of the supernatant was removed and transferred to the oven (Solab, SL104/27, São Paulo, Brazil) at 105°C for 5 h. Solubility was calculated by weight difference.

Hygroscopicity

Hygroscopicity was evaluated based on the method recommended by Cai and Corke,[32] with some changes. Approximately 1 g of mango flakes were placed in a container at 25°C with a NaCl saturated solution (75.29% RH). Samples were weighed when the equilibrium was reached (after 1 week), and hygroscopicity was calculated according to Jaya and Das[33] (Eq. 2):

$$H = \frac{\frac{\Delta m}{M} + U}{1 + \frac{\Delta m}{M}} \tag{2}$$

where H = hygroscopicity (%); Δm = increase in weight of the powder after equilibrium (g); M = initial mass of the powder (g); and U = initial moisture content of the powder (%).

Wettability

Wettability was determined according to the method described by Vissotto et al.,[34] considering the time (s) required for 7.0 g of mango flakes deposited on the liquid surface to become completely submerged in 400 mL of distilled water at 23–25°C.

Vitamin C

The vitamin C content of the mango flakes was evaluated using Tillmans' titration method,[24] based on the reduction of the 6-dichlorophenolindofenol-sodium indicator (DCFI) by ascorbic acid. Vitamin C was expressed as mg ascorbic acid/100 g dry basis.

Total phenolic compounds

The total phenolic compounds of the mango flakes were determined using Folin Ciocalteau's spectrophotometry method, according to Benvenuti et al.[27] Results were expressed as mg gallic acid equivalent/100 g of total solids (dry basis).

Total carotenoids and β-carotene

The total carotenoids and β-carotene contents were determined according to Carvalho et al.[28] Total carotenoids were quantified in a spectrophotometer (Cary 50 UV–Vis, Agilent Technologies, Richardson, USA), at

the maximum absorption wavelength of β-carotene (453 nm) using the absorption coefficient of 2592. The detection and quantification of β-carotene was performed using high-performance liquid chromatography (HPLC) (Infinity 1260 Quaternary LC, Agilent Technologies, Richardson, USA), and analytical column (LiChrospher 100 RP-18, 125 × 4 mm^2, 5 µm, Merck, Kenilworth, USA) and β-carotene standard (C4582, Sigma-Aldrich, St. Louis, USA) were used.

Antioxidant activity (DPPH and ABTS methods)

The determination of the antioxidant activity by DPPH method (2,2-diphenyl-1-picrylhydrazyl) was done according to Brand-Williams et al.[25] ABTS method is based on the capture of the 2,2′-azinobis (3-ethylbenzothiazoline-6-sulfonic acid) radical (ABTS•+), which can be generated by a chemical reaction with potassium persulfate, in accordance with the adaptation of the methodology proposed by Rufino et al.[26] All the results were expressed as micromoles of Trolox equivalent per gram (dry basis).

Color parameters

Color parameters of mango flakes and reconstituted pulps were determined using a colorimeter (CR300, Minolta, Osaka, Japan), through the CIELAB system (D65 illuminant). L^* lightness ($L^* = 0$ for black and $L^* = 100$ for white) and chromaticity parameters a^* (green [−] to red [+]) and b^* (blue [−] to yellow [+]) were measured.

The reconstituted pulps were obtained from the homogenization of the flakes with distilled water in a Turratec grinder (Tecnal, TE-102, Piracicaba, Brazil). The amounts of water were calculated to obtain the same total solids contents of the whole pulp. *Chroma* (color intensity) and Hue were determined for the reconstituted pulps, according to Eqs. (3) and (4), respectively. The color difference (ΔE) was also calculated for the reconstituted pulps with respect to the whole pulp, using Eq. (5):

$$\text{Chroma} = (a^{(*2)} + b^{(*2)})^{(1/2)} \quad (3)$$

$$\text{Hue} = \arctan(b^*/a^*) \quad (4)$$

$$\Delta E = ((\Delta L^*)^2 + (\Delta b^*)^2 + (\Delta a^*)^2)^{(1/2)} \quad (5)$$

Particle size distribution

This analysis was performed by laser light diffraction instrument (Partica, model LA950V2, Horiba, Tokyo, Japan).[35] The test was performed six times, using the dry module of equipment, considering the values of the medians (D50) as the average diameter of the sample. D50 was calculated by Horiba's software, and represents the size in microns which splits the particle size distribution profile (frequency × mean diameter), with half above and half below this diameter.

Scanning electron microscopy

Scanning electron microscopy (SEM) analysis was performed with the mango flakes obtained in a validation test performed under the optimal conditions and described later. Samples were attached to SEM stubs using a double-sided adhesive tape and coated with gold/palladium under vacuum in a Polaron Sputter Coater (model SC7620, VG Microtech, Ringmer, UK) at a coating rate of Å/s, 3–5 mA, 1 V, and 0.08–0.09 mbar for 180 s, according to the method reported by Ferrari et al.[5] The coated samples were observed in a LEO440i scanning electron microscope (LEICA Electron Microscopy Ltd., Oxford, England). The SEM was operated at 22 ± 2°C, 20 kV and 100 pA with a magnification of 45 and 100×.

Statistical methods

A polynomial regression equation was used to describe the behavior of the response surface for each response (Yi), according to the following model[7]:

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{12} x_1 x_2 \quad (6)$$

where Y is the response calculated by the model; β_0, β_1, and β_2 are the regression coefficients for the linear terms; β_{11} and β_{22} are the quadratic terms; β_{12} is the interaction term; x_1 and x_2 represent the coded values of the independent variables, residence time and temperature, respectively.

Table 2 shows the coded and real values of the independent variables in the central composite rotatable design, as well as the experimental results of the significant responses.

Results were statistically evaluated for each response using multiple regression analyses with software Statistica 8.0 (StatSoft, Inc., Tulsa, USA). The quality of the fit was analyzed by the coefficient of determination R^2 (>0.70). Analysis of variance (ANOVA), F-test, and p-value were used to determine the significant terms for each model, considering a significance level of 5%. The surface response graphics were built from the polynomial models, with the significant terms.[36–38]

Table 2. Coded and real values of the independent variables in the central composite rotatable design, and the experimental results of the significant responses.

Test	Residence time (t)	Temperature (T)	Moisture content (%)	Total carotenoids content (mg/100 g) (db)	a^*	Hue	ΔE
1	14 (−1)	124 (−1)	2.53 ± 0.02	15.23 ± 0.15	2.74 ± 0.22	85.73 ± 0.32	3.69 ± 0.43
2	36 (1)	124 (−1)	2.06 ± 0.05	14.80 ± 0.35	3.22 ± 0.31	85.07 ± 0.51	3.28 ± 0.37
3	14 (−1)	146 (1)	1.22 ± 0.01	14.08 ± 0.15	3.90 ± 0.13	83.92 ± 0.24	3.96 ± 0.40
4	36 (1)	146 (1)	0.74 ± 0.08	13.94 ± 0.28	4.31 ± 0.18	82.76 ± 0.33	7.35 ± 0.30
5	25 (0)	135 (0)	1.29 ± 0.04	15.19 ± 0.02	3.68 ± 0.09	84.42 ± 0.25	3.20 ± 0.53
6	25 (0)	135 (0)	1.22 ± 0.09	14.45 ± 0.34	3.23 ± 0.23	84.86 ± 0.36	4.74 ± 0.44
7	25 (0)	135 (0)	1.37 ± 0.02	14.31 ± 0.28	3.76 ± 0.17	84.06 ± 0.28	4.31 ± 0.23
8	25 (0)	135 (0)	1.22 ± 0.01	14.33 ± 0.34	3.70 ± 0.14	84.17 ± 0.23	4.62 ± 0.52
9	9.44 (−1.41)	135 (0)	2.13 ± 0.08	14.51 ± 0.22	3.05 ± 0.31	83.33 ± 0.31	0.84 ± 0.22
10	25 (0)	150.6 (1.41)	0.97 ± 0.07	13.53 ± 0.38	4.20 ± 0.17	85.67 ± 0.41	4.78 ± 0.38
11	40.6 (1.41)	135 (0)	1.55 ± 0.03	13.89 ± 0.46	3.57 ± 0.03	84.30 ± 0.04	4.77 ± 0.28
12	25 (0)	119.4 (−1.41)	3.04 ± 0.07	15.76 ± 0.58	3.46 ± 0.12	84.73 ± 0.20	3.05 ± 0.34

Moisture content: mean ± standard deviation ($n = 4$).
Total carotenoids content: mean ± standard deviation ($n = 3$).
Hue, color difference (ΔE), a^* parameter: mean ± standard deviation ($n = 9$).

Results and discussion

Models and response surfaces

Response surfaces were obtained ($p \leq 0.05$), according to the established criteria, for the following dependent variables: moisture content and total carotenoid content of mango flakes; a^*, Hue, and ΔE color parameters of the reconstituted pulps. No valid models were obtained for the other investigated properties. This result indicates that the remaining response variables are unaffected by the independent variables in the tested range. Nevertheless, this fact might be also related to the difficulties for the absolute control of all process conditions, since the tests were performed on a pilot scale.

Table 2 presents the central composite rotatable design, coded and real values of the independent variables (process temperature and residence time), as well as the experimental results of the significant responses. The obtained models (Eqs. 7–11, Table 3) show different fit levels, with determination coefficients (R^2) varying from 0.72 to 0.96.

Equation (7), which represents the variation of moisture content, is a quadratic model regarding the independent variables, without the interaction term of both variables. The quadratic and linear terms are positive and negative, respectively, with a predominant effect of the temperature. The surface shows a region of minimum values, located in the combination of $T > 0$ and $t > 0$ (Figure 1a). According to the model, drum drying tests of mango pulp performed under the combined conditions of higher temperatures and longer residence times ($T > 135$°C and $t > 25$ s) resulted in products with a moisture content around 1%. The process performed at lower temperatures and residence times ($T < 135$°C and $t < 25$ s) led to the formation of drum dried mango flakes with higher moisture content (ranging from 2 to 4%). Similarly, Pua et al.,[7] working with the optimization of drum drying process parameters for production of jackfruit powder, observed that high process temperatures resulted in lower moisture content, while the increase of cylinder speed, that is, shorter residence times, led to higher moisture contents. The moisture content values cited by the authors (between 5 and 10%) were higher than those found in the present study.

Equation 8 indicates that the total carotenoid content of the drum dried mango powder only depends on the process temperature, in a linear form. Besides, the temperature negatively affected this parameter. The use of lower temperatures (between 120 and 135°C) resulted in higher total carotenoids content (from 14.4 to 15.2 mg/100 g), with retention varying from 90 to 96% (Figure 1b). According to the model, the drum drying tests done with higher temperatures (from 135 to

Table 3. F values, coefficients of determination (R^2) and second-order polynomial models of the significant responses: Moisture content, total carotenoids content, a^*, Hue, and ΔE.

Properties	R^2	F_c	F_{tab}	Second-order polynomial equation	Number
Moisture content (%)	0.96	47.05	4.12	$MC = 1.271 - 0.443t + 0.427t^2 - 1.389T + 0.590T^2$	(7)
Total carotenoids (mg/100 g db)	0.73	27.25	4.96	$TC = 14.500 - 1.290T$	(8)
a^*	0.73	11.96	4.26	$a^* = 3.567 + 0.407t + 0.826T$	(9)
Hue	0.76	14.58	4.26	$Hue = 84.419 - 0.941t - 1.523T$	(10)
ΔE	0.72	6.95	4.07	$\Delta E = 4.048 + 2.136t + 1.696T + 1.895Tt$	(11)

F_c, F calculated; F_{tab}, F tabulated ($p \leq 0.05$); R^2, coefficient of determination; T, coded temperature; t, coded time; a^*, Hue, and ΔE, color parameters of the reconstituted pulp.

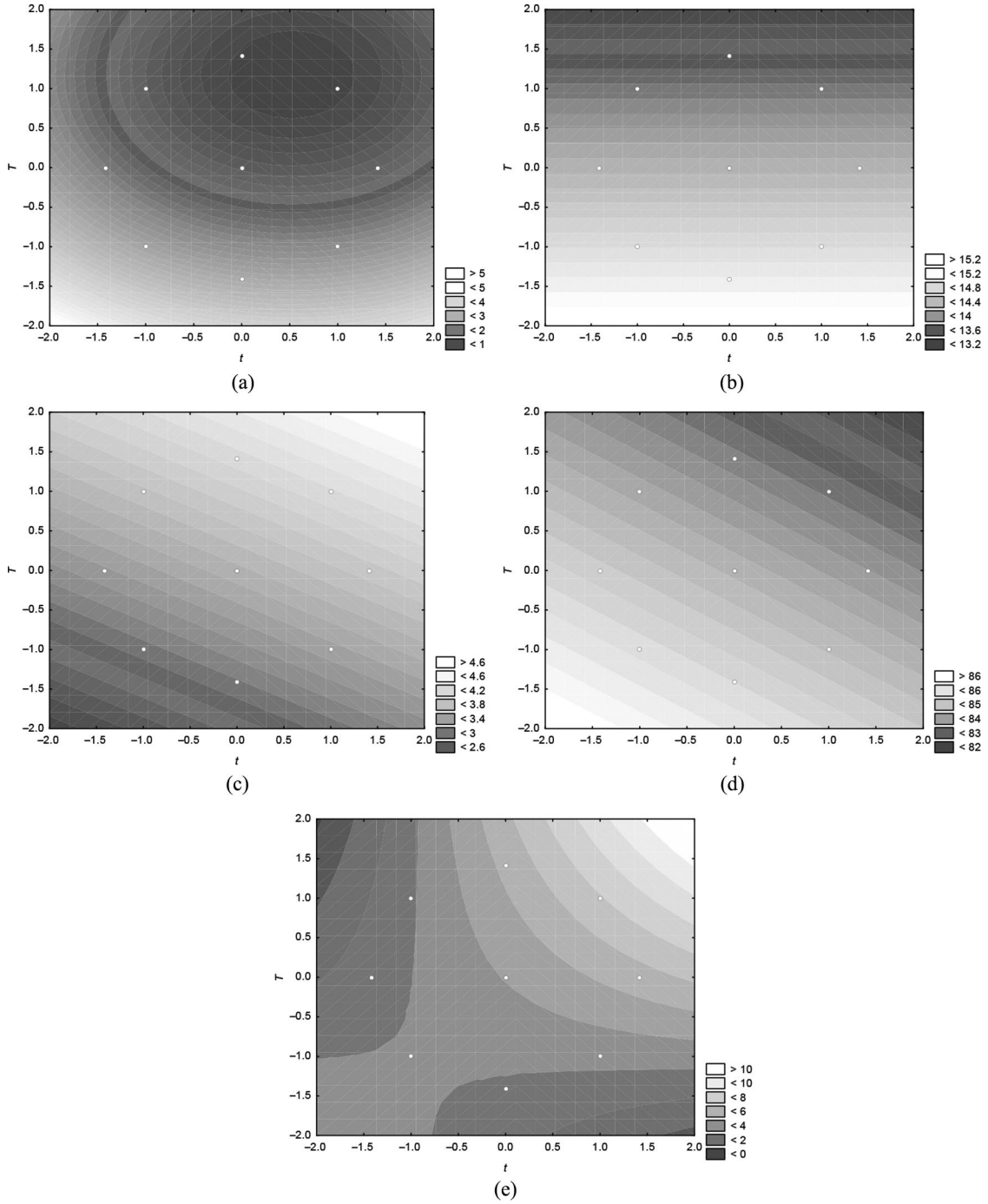


Figure 1. Contour curves of the obtained surface responses: (a) moisture content (%); (b) total carotenoids (mg/100 g); (c) *a** parameter of the reconstituted pulp; (d) Hue of the reconstituted pulp; and (e) color difference (*ΔE*) of the reconstituted pulp.

150.6°C) provided total carotenoids retention around 82–88%. Working with drying of mango pulp by spray drying and cast-tape drying, Zotarelli et al.[4] reported lower retention of total carotenoids (about 40 and 29%), in comparison to the current study. The authors attributed this behavior to the high sensitivity of carotenoids to oxidation, suggesting that adjustments in the process conditions (temperature and process time) are important to minimize the degradation of carotenoids. However, Desobry et al.[12] found losses of carotenoids around 14% during drum drying (140°C/45 s) of model solutions (pure β-carotene), using maltodextrin 25 DE (40%). The authors stated that the degradation of the pigments in the process is mainly due to the collapse of the solid matrix, and less to the pigment thermosensitivity, since the melting point of the carotenoid (approximately 193°C) was not reached under the conditions used.

Concerning a^* color parameter (red) of the reconstituted pulp, Eq. (9) describes a linear variation model with temperature, as well as residence time, showing a predominant influence of the temperature. In accordance with the contour curve (Figure 1c), the higher the residence time ($t > 25$ s) and the process temperature ($T > 135$°C), the higher is a^* value, that is, the product is more reddish. Equation (10) shows that Hue angle of the reconstituted pulp depends linearly on the two factors evaluated, with a higher effect on the process temperature. Hue equals to 90° represents the pure yellow color, while 0° represents the pure red. Lower residence time ($t < 25$ s) and temperature ($T < 135$°C) resulted in more yellowish reconstituted pulps (Figure 1d), with Hue angle closer to 90°. However, combinations of higher residence times ($t > 25$ s) and temperatures ($T > 135$°C) led to more reddish reconstituted pulps, with Hue angle values furthest from 90°. The model obtained for the color difference (ΔE) of the reconstituted pulp (Eq. 11) presents the linear terms of each independent variable, as well as the interaction term of both factors. According to Figure 1e, lower residence times ($t < 25$ s) and temperatures ($T < 135$°C) resulted in lower ΔE values (around 2–4). These values are well below those reported by Jaya and Das[33] during the vacuum drying of mango pulp, which under optimized conditions was from 5.8 to 7.9. In the present study, combinations of higher process times and temperatures promoted greater color change, with ΔE ranging from 6 to 10 (Figure 1e). Caparino et al.,[21] working with drum drying of mango pulp ($T = 152$°C and $t = 54$ s) obtained ΔE of 9.22 between the reconstituted pulp and the pure pulp. The better results of the present study may be related to the milder process conditions (temperatures from 124 to 151°C and residence times between 14 and 41 s), and also to the protective effect of the additives used in the drum drying process. For Setyadjit and Sukasih,[8] the additives in drum drying additionally offer a protective effect to denaturation and losses of natural compounds. In general, the results of the color parameters of the reconstituted pulp in the current work are consistent with the literature, and the possible causes of their variation are nonenzymatic browning (Maillard reaction) and caramelization of sugars in the drying process.

To determine the best process conditions, a graphical analysis of the obtained surfaces was made, considering the models for moisture content (Eq. 7), total carotenoid content (Eq. 8) and color difference (ΔE) of the reconstituted pulp (Eq. 11). Although the models for a^* and Hue are also statistically significant (Table 3), they were not included in this analysis, since they are correlated with ΔE. The graphical approach is also reported in some drying studies, and consists of a superimposition of different surfaces for the determination of the experimental condition that results in the desired effects.[7,39,40]

Figure 2 illustrates the superimposed contour curves of the evaluated surfaces. The gray region shows the best combinations of the factors, resulting in the most interesting product characteristics: total carotenoid content between 14.4 and 15.6 mg/100 g (which corresponds to total carotenoids retention ranging from 90 to 96%); color difference ΔE of the reconstituted pulp with respect to the whole pulp between 2 and 4); and moisture content from 2 to 4%. Therefore, according to this analysis, the obtained optimal area is delimited by temperatures between 120 and 135°C and residence time varying from 10 to 25 s.

To evaluate the models, an experimental test was performed using a combination of process variables within the optimal area: temperature of 135°C and residence time of 20 s. The experimental and predicted values by the mathematical models are shown in Table 4. The differences between the observed and the predicted values were below 20% for all the responses, except for ΔE, being lower for moisture content and Hue parameter. Concerning the total carotenoids content and a^* parameter of reconstituted pulp, the differences were about 10 and 20%. Small variations in the characteristics of the raw material as well as in the experimental conditions may have contributed to these results. With respect to ΔE, the error is possibly due to the sum of the experimental variations of L^*, a^*, and b^* values for its calculation (Eq. 5).

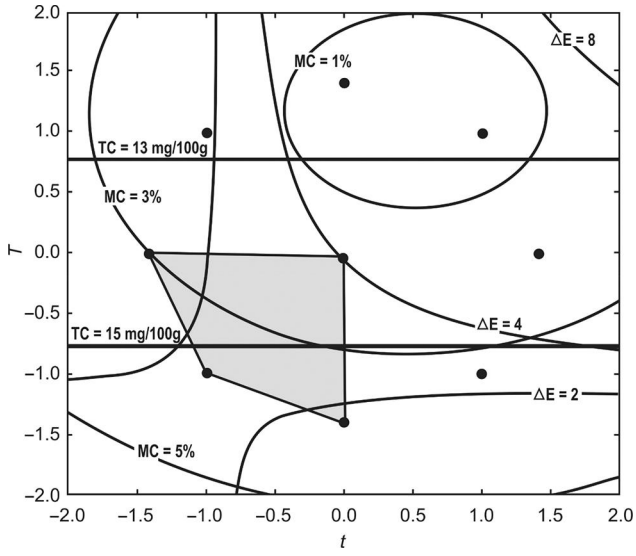


Figure 2. Superimposed contour plot of moisture content (MC), total carotenoid content (TC) and color difference (ΔE) as a function of the coded variables temperature (T) and time (t): delimited area in gray is the optimized region is the optimized region.

Table 4. Predicted and observed values of the significant responses in the drum drying of mango pulp performed under the optimized conditions.

Parameters	Predicted	Observed	CV (%)
Moisture content (%)	1.56	1.73 ± 0.02	9.91
Total carotenoids content (mg/100 g db)	14.50	13.67 ± 0.22	6.11
a^*	3.38	2.90 ± 0.14	16.46
Hue	84.85	85.62 ± 0.20	0.90
ΔE	3.08	4.11 ± 0.11	25.24

a^*, Hue, and ΔE, color parameters of the of the reconstituted pulp.

In this context, despite the observed differences, it is reasonable to consider the obtained optimal region as a good indication of the best conditions for the drum drying of mango pulp. This area contains possible drum drying operation conditions with good results in terms of moisture content, carotenoid retention and color of reconstituted pulp. According to Madamba and Lopez[41] and Delgado-Nieblas et al.,[39] the central point of the optimal area should be stated as the optimal condition for the process. Therefore, temperature of 127.2°C and residence time of 17.2 s could be pointed as the optimal combined conditions in the current study.

Variation of physicochemical properties and nutrients contents

Tables 5 and 6 show the mean results of physicochemical properties and nutrients contents, respectively, of drum dried mango flakes, whose values were not adjusted by the models. However, the evaluation of these results, together with the models obtained, help to better understand the drum drying of mango pulp.

According to Table 5, color parameters values of the flakes ranged approximately from: $52 < L^* < 59$; $12 < a^* < 14$; $66 < b^* < 71$. Comparing to the color of the original pulp (Table 1), a slight whitening was observed in the process, as well as an increase of the red color and an intensification of the yellow color. Chaux-Gutiérrez et al.[42] also observed an increase in L^* parameter in mango powder ($78 < L^* < 85$) obtained by foam-mat drying using a pulp with similar characteristics of the current work. The authors attributed the results to the incorporation of air into the mango pulp during the foaming process. According to Caparino et al.,[21] the higher L^* values usually observed in powder/flakes might be attributed to the reflection of the light by the irregular particles. Regarding the increase of red color (a^*), enzymatic browning (Maillard) and caramelization of sugars, as previously mentioned, might be the possible causes. The dominant color in mango pulp is yellow, represented by the chromatic parameter b^*. The drum drying of mango pulp resulted in higher values of this parameter, which is expected due to the loss of water. These results are similar to those reported by Caparino et al.[21] for mango flakes obtained by refractance window and freeze-drying.

The mass flow rates of the different tests varied between 2 and 6 kg/h m^2. Travaglini et al.,[23] in a similar work with drum drying of mango pulp using 4% corn starch and 1% glyceryl monostearate, reported mass flow rates from 4 to 6 kg/h m^2 under different process conditions. Vallous et al.,[43] in a theoretical study about the drum drying process, obtained mass flow rates ranging from 2 to 8 kg/h m^2. The authors used residence times of 14–41 s and drying area equivalent to that of the present study.

Concerning the water activity, the values were between 0.27 and 0.34, indicating that the mango flakes show microbiological stability, according to Labuza.[44] Similarly, Jittanit et al.[16] found water activity values around 0.30 in a drum drying study of tamarind juice using maltodextrin (40% wet basis). With respect to

Table 5. Physicochemical properties of the flakes obtained in the different tests of the central composite rotatable design.

Test	L^*	a^*	b^*	Mass flow rate (kg/h m^2)	Water activity	Hygroscopicity (%)	Solubility (%)	Wettability (s)	Mean particle diameter (mm)
1	52.93 ± 2.52	13.40 ± 1.06	68.72 ± 3.98	5.34 ± 0.18	0.269 ± 0.001	21.45 ± 0.08	83.03 ± 0.09	15 ± 0.6	1.33 ± 0.21
2	58.84 ± 0.85	12.87 ± 0.28	70.02 ± 1.23	2.52 ± 0.25	0.314 ± 0.003	21.14 ± 0.10	78.87 ± 0.05	29 ± 1.0	1.12 ± 0.21
3	58.50 ± 1.75	12.21 ± 0.39	68.46 ± 1.18	6.00 ± 0.05	0.272 ± 0.003	19.73 ± 0.12	78.65 ± 0.04	38 ± 1.5	1.08 ± 0.06
4	56.50 ± 1.23	14.09 ± 0.36	66.33 ± 1.09	4.14 ± 0.00	0.327 ± 0.001	19.15 ± 0.11	77.85 ± 0.34	16 ± 1.2	1.09 ± 0.04
5	59.26 ± 0.89	13.61 ± 0.46	70.61 ± 1.25	3.42 ± 0.15	0.320 ± 0.002	21.20 ± 0.08	78.78 ± 0.15	32 ± 0.6	1.14 ± 0.05
6	56.65 ± 2.58	12.69 ± 0.36	69.05 ± 1.00	5.15 ± 0.12	0.341 ± 0.001	20.18 ± 0.05	78.32 ± 0.14	39 ± 1.5	1.13 ± 0.06
7	57.36 ± 1.73	13.25 ± 0.45	67.18 ± 2.16	4.94 ± 0.09	0.240 ± 0.001	20.59 ± 0.09	78.63 ± 0.04	33 ± 1.7	1.27 ± 0.04
8	57.75 ± 1.30	13.79 ± 0.47	69.19 ± 1.90	4.98 ± 0.08	0.341 ± 0.005	19.99 ± 0.09	78.54 ± 0.04	24 ± 0.0	1.16 ± 0.04
9	58.18 ± 1.10	13.42 ± 0.43	69.09 ± 1.30	4.86 ± 0.10	0.319 ± 0.002	19.07 ± 0.01	78.79 ± 0.04	20 ± 0.6	1.40 ± 0.18
10	58.28 ± 1.85	13.97 ± 0.60	67.87 ± 2.50	4.28 ± 0.13	0.309 ± 0.001	20.56 ± 0.08	78.81 ± 0.12	34 ± 0.6	1.20 ± 0.04
11	59.14 ± 1.29	12.76 ± 0.52	68.45 ± 1.79	5.67 ± 0.03	0.312 ± 0.003	19.02 ± 0.05	78.65 ± 0.03	34 ± 1.7	1.17 ± 0.03
12	56.52 ± 0.84	12.40 ± 0.59	67.43 ± 1.39	1.70 ± 0.39	0.335 ± 0.004	19.79 ± 0.06	79.06 ± 0.08	22 ± 0.6	1.25 ± 0.08

L^*, a^*, and b^* parameter: mean ± standard deviation ($n = 9$).
Mass flow rate, water activity, hygroscopicity, solubility, wettability: mean ± standard deviation ($n = 4$).
Mean particle size: mean ± standard deviation ($n = 6$).

Table 6. Vitamin C, total phenolic compounds, β-carotene content, and antioxidant activity of the flakes obtained in the different tests of the central composite rotatable design.

Test	Vitamin C content (mg/100 g db)	Total phenolic compounds (mg GAE/ 100 g db)	β-Carotene content (mg/100 g db)	Antioxidant activity—ABTS (μmol TE/g db)	Antioxidant activity—DPPH (μmol TE/g d.b)
1	34.68 ± 0.42	503.49 ± 0.27	7.57 ± 0.28	67.76 ± 1.38	43.79 ± 0.46
2	23.10 ± 0.61	488.18 ± 6.61	6.82 ± 0.08	74.90 ± 0.37	42.17 ± 0.64
3	31.03 ± 0.37	494.53 ± 2.64	5.91 ± 0.12	75.12 ± 0.81	42.72 ± 1.28
4	35.96 ± 1.08	507.87 ± 2.08	6.24 ± 0.22	69.48 ± 0.69	42.96 ± 0.24
5	30.88 ± 0.03	507.21 ± 1.16	7.32 ± 0.16	57.44 ± 1.02	41.20 ± 0.11
6	27.98 ± 0.60	492.56 ± 0.96	6.54 ± 0.31	62.85 ± 0.41	40.38 ± 0.46
7	29.27 ± 0.33	477.05 ± 0.57	6.71 ± 0.15	57.19 ± 0.26	38.62 ± 0.53
8	27.29 ± 0.89	477.13 ± 1.29	6.01 ± 0.01	65.17 ± 1.71	41.18 ± 0.32
9	35.01 ± 0.48	482.07 ± 0.52	6.71 ± 0.15	70.28 ± 1.25	39.01 ± 0.89
10	24.32 ± 0.73	492.07 ± 1.10	7.05 ± 0.08	68.75 ± 0.44	40.34 ± 1.21
11	30.96 ± 0.62	562.47 ± 8.86	6.49 ± 0.01	69.99 ± 0.27	41.31 ± 0.51
12	32.92 ± 0.67	518.82 ± 2.41	7.61 ± 0.07	71.75 ± 0.17	43.55 ± 0.92

db, dry basis.
Mean ± standard deviation ($n = 4$).

hygroscopicity, the results varied from 19 to 21%, which is in accordance with the mean values presented by Caparino et al.[21] (~21%) for mango flakes produced in drum drying without additives.

The solubility of the flakes ranged from 78 to 83% (Table 5). These results were greater than those reported by Cano-Chauca et al.,[31] in the spray drying of mango juice with different process additives. According to the authors, solubility values were around 31% in the test with 12% waxy starch and 9% cellulose (wet basis), and about 72% in the test performed with 12% maltodextrin and 9% cellulose (wet basis). Solubility of the products decreased as a function of the cellulose concentration, and the starches used in their work had low solubility in cold water (between 35 and 40%). Therefore, the high solubility of the mango flakes observed in the current study may be attributed to the low concentration of corn starch used as an additive. However, Caparino et al.[21] stated that the solubility of mango flakes produced with drum drying without additives was around 94%. The authors concluded that the relatively greater solubility of the drum dried powders in comparison to the other methods evaluated (spray drying and refractance window) is due to a higher degree of macromolecular disorganization of the product affected by process conditions.

Regarding the wettability, the mean values ranged from 15 (test 1) to 39 s (test 6). Ferrari et al.,[45] studying the spray drying of blackberry pulp, obtained wettability around 82 and 134 s for the powders produced with maltodextrin and gum Arabic, respectively. According to Gong et al.,[46] spray-dried powders often have a small particle size (<50 μm), with poor handling and reconstitution properties, that is, higher wettability values. With respect to the particle size, the mean diameter varied approximately from 1.1 mm (test 3) to 1.4 mm (test 9), showing a significant variation in the size of the mango flakes obtained (Table 5). Figure 3 shows, as an example, a particle size distribution of the drum dried mango flakes determined in test 7 of the experimental design (Table 2). The curve shows a monomodal distribution behavior, even though it has been interrupted at 3 mm, which is the maximum detection limit of the equipment.

It is possible to make connections between some of the mean diameter results and the respective values of moisture content and wettability. Test 4 (1; 1), whose product showed the lowest moisture content (around 0.75%), as seen in Table 2, resulted in one of the smallest mean diameter (~1.09 mm) (Table 5). In this case, the drier product probably broke down more in flocculation, resulting in smaller particles. However, test 9 (−1.41; 0), whose flakes had one of the highest moisture content (approximately 2.1%) (Table 2), presented the largest mean diameter (1.40 mm) (Table 5), probably due to the lower particle fracture during the flocculation process. Furthermore, the product obtained from test 1 (−1; −1) (Table 2), with one of the largest particle diameters (1.33 mm), had the shortest wettability time (15 s), while the flakes of test 6, with the smallest

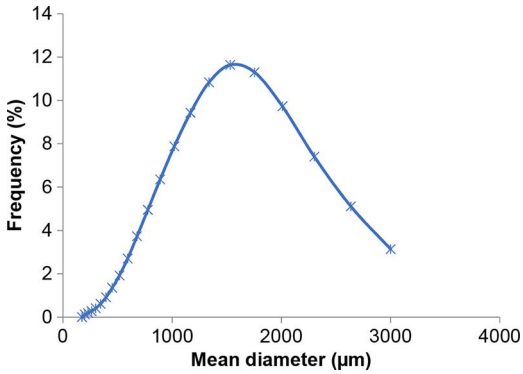


Figure 3. Particle size distribution of the drum dried mango powder obtained using residence time (t) = 25 s and temperature (T) = 135°C (Test 7).

particle diameter (1.13 mm), required a longer time to wet (39 s) (Table 5). These results are consistent with Vissotto et al.,[34] who stated that smaller particles tend to have high wettability times because they form granules in the mixture with water. According to the authors, the wettability can be related to some physical factors, especially size and shape of the particles, being favored by the interstitial spaces presented in the larger particles with an irregular shape.

As noticed in Table 6, vitamin C content of the flakes varied from approximately 23 mg/100 g (test 2) to 36 mg/100 g (test 4), corresponding to retentions of 48 and 75%, respectively. Caparino et al.[47] reported a vitamin C content of approximately 13 mg/100 g in mango flakes obtained with refractance window. The maximum retention of vitamin C obtained in the present study is close to that found in the drum drying of goldenberry juice at 110°C (around 76%) by Valdenegro et al.[17] Nevertheless, that result might have been influenced by the use of ascorbic acid as an antioxidant agent during the preparation of the pulp to avoid browning. In accordance with Santos,[48] the degradation of vitamin C strongly depends on the time and temperature of the drying; however, the preliminary steps of the process may be also responsible for important losses of this nutrient.

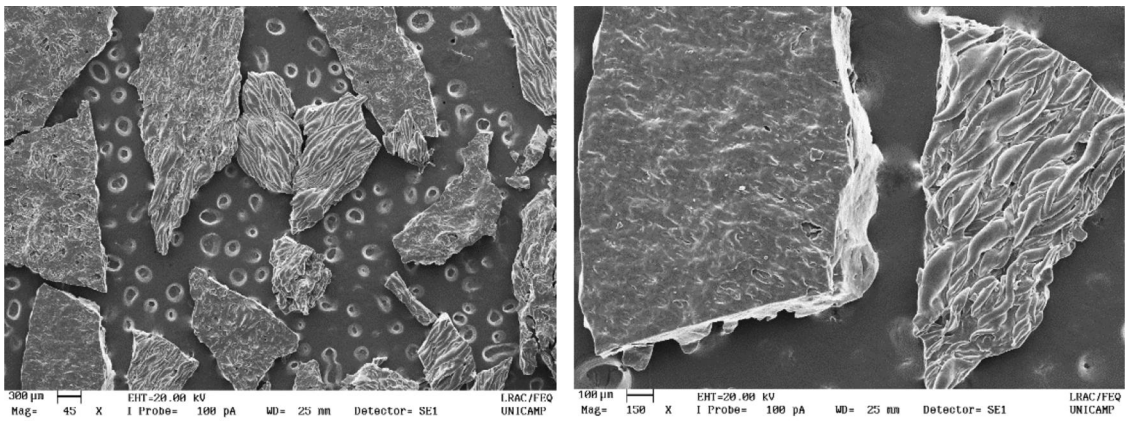
The total phenolic compounds content ranged between 477 and 518 mg GAE/100 g (db), resulting in retentions higher than 95% (Table 6). Sogi et al.[49] studying the drying of mango Tommy Atkins variety using different processes (vacuum drying, hot air drying, freeze-drying, and infra-red drying), obtained concentrations of phenolic compounds above 900 mg GAE/100 g (db). The slightly smaller values observed in the present research may be due to the differences among the drying techniques, but also to the differences in the characteristics of the raw materials, such as varieties, ripening stages, cultivation areas and others. The commercial pulp used in this study showed a mean total polyphenols content around 606 mg GAE/100 g (dry basis), which is in agreement with the range reported by Hung,[50] that is, from 250 to 750 mg GAE/100 g (db) for pulps of different mango varieties.

The antioxidant activity of the drum dried mango flakes obtained by ABTS method was between 57 and 75 μmol TE/g dry basis, with retentions above 78%. With respect to DPPH method, the values were lower (around 39–44 μmol TE/g dry basis), showing retentions higher than 90%. Sogi et al.,[49] in a similar work with vacuum drying and freeze-drying of mango pulp, also observed lower antioxidant activity values for DPPH method in comparison to ABTS method. The results presented in Table 6 are within the range cited by the authors for each method.

For β-carotene content, Table 6 shows a variation from approximately 6 to 7.6 (mg/100 g), with retentions greater than 85%, which is close to the retention reported by Desobry et al.[12] in the drum drying pure β-carotene, using maltodextrin 25 DE. However, Zotarelli et al.[4] observed lower β-carotene retention (around 40%) in a study with spray drying of mango pulp using maltodextrin 10 DE.

Morphological analysis of the drum dried mango flakes

The morphological characteristics of the particles obtained in the validation test of the models, performed at $T = 135$°C and $t = 20$ s, can be seen in Figure 4. A large variability of particle sizes and shapes is noticed (Figure 4a), corroborating the results previously discussed about mean particle diameters (Table 5).



(a) (b)

Figure 4. Scanning electron microscopy micrographs of the flakes obtained using drum drying of mango pulp (temperature = 135°C and residence time = 20 s) with 3% corn starch and 0.5% glyceryl monostearate: (a) magnification = 45× and (b) magnification = 150×.

Figure 4b (with magnification = 150 ×) shows that the particles have sharp edges, which are typical of products produced by grinding or flocculation.[51] Caric and Kalab[51] stated that the thickness of the particles obtained in drum drying is influenced by the process conditions; however, the other dimensions are defined in milling/flocculation. In addition, it is evident that the particles have two distinct sides, one smooth and one wrinkled, in which the smooth side is the one in direct contact with the cylinder surface. Similar behavior was also reported by Caparino et al.[21] in the drum drying of mango pulp. In addition, granular structures are observed at the crinkled side (Figure 4b), which may be related to starch gelatinization. According to Vallous et al.,[43] gelatinization of the starch in the drum drying takes place at the pool, when the liquid comes into contact with the heated surface of the cylinder. The drying starts when the gelatinized material leaves the pool, and forms a thin film on the drum surface, which possibly explains the stretched shape of the granules.

Conclusion

The study showed that the variations of some properties of the drum dried mango flakes can be described by mathematical models with process temperature and residence time as independent variables. A second-order polynomial equation was able to explain the variation in the moisture content of the mango flakes as a function of the investigated factors, in which the temperature was the predominant effect. The minimum values (approximately 1% wet basis) were observed in the region for temperatures higher than 135°C and residence time above 25 s. However, total carotenoids content only depends on the process temperature, linearly and negatively, with retention values of 90–96% in the range of 120–135°C. The models of the color parameters (a *, Hue, and ΔE) of the reconstituted pulp demonstrated the preponderant and deleterious effect of the process temperature, which caused an increase in the red color, differing from the color of the fresh pulp. The combination of these models, aiming at the greater retention of product quality, resulted in the determination of the best process conditions, which were the combination of residence times ranging from 10 to 25 s and temperature between 120 and 135°C. Under these conditions, the mango flakes obtained by drum drying had good quality characteristics, such as high solubility and wettability, and reasonable retentions of other nutrients, such as vitamin C, total phenolic compounds and antioxidant capacity. The central point of this area may be stated as the optimal condition for the drum drying of mango pulp: temperature around 127°C and residence time close to 17 s.

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