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SPRAY DRYING AND PROCESS OPTIMIZATION OF SOUR ORANGE JUICE

L. Cuevas-Glory[a], M. Bringas-Lantigua[b], E. Sauri-Duch[a], O. Sosa-Moguel[a], J.A. Pino[b]* and H. Loría-Sunsa[a]

[a]Technological Institute of Merida. Merida, Yucatan. Mexico
[b]Research Institute for Food Industry. Carretera a Guatao km 3½, Havana 19200. Cuba

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In this study, production of sour orange juice powder utilizing a spray dryer was investigated. To prevent stickiness, maltodextrin DE 12 was used as a drying agent. While feed flow rate, feed temperature, and air flow rate were kept constant, inlet air temperature (120–160 °C) and maltodextrin content (maltodextrin dry solids/100 g feed mixture dry solids; 10–20%, w/w) were selected as the independent variables. Product properties investigated included ascorbic acid, volatile compounds, and moisture content. Ascorbic acid retention, volatiles retention, and moisture content were used in optimization of the process by response surface methodology. The optimum inlet air temperature and maltodextrin content were 156 °C and 20% w/w maltodextrin, respectively. This study revealed that by applying these optimal conditions, sour orange juice powder with 81.5% ascorbic acid retention, 5.5%, w/w moisture content, and 78% volatiles retention was produced.

Keywords: sour orange, spray drying, optimization, ascorbic acid retention, volatiles retention

Sour orange (*Citrus aurantium* L.), also known as bitter or Seville orange, is native to South-eastern Asia, but actually grows in various other parts of the world, where its special products are of commercial importance. The sour orange is usually too sour to be directly enjoyed out-of-hand like sweet orange, but it has other food and medicinal uses. The greatest use of sour oranges as food is in the form of marmalade, and its juice is valued as a flavouring on meat during cooking. Additionally, sour orange juice has antiseptic, anti-bilious properties and is haemostatic. Sour orange juice contains vitamins, principally vitamin C or ascorbic acid (0.55−1.04 g kg^{-1} juice), and also contains vitamin A (as carotenes), plus B-complex vitamins (Morton, 1987).

Spray drying is widely used in both food and pharmaceutical manufacturing processes. This technique offers short contact times, allowing certain properties of foods, such as flavour, colour, and nutrients, to be retained in high percentages (Phisut, 2012). However, the main problem during spray drying of sugar-rich foods, such as fruit juices, is their thermoplastic behaviour. Fruit juice powders obtained by spray drying might present certain problems, such as stickiness, hygroscopicity, and solubility, due to the presence of low molecular weight sugars and acids, which have a low glass transition temperature (Muzaffar et al., 2015). Thus, they can adhere to the dryer chamber wall during drying, leading to low product yield and operational problems. Some additives, such as starch, gum arabic, and maltodextrins, are commonly used as carrier agents to prevent stickiness of product during spray drying (Phisut, 2012; Krishnaiah et al., 2014). Of these additives, maltodextrins offer a good compromise between cost and effectiveness (Phisut, 2012).

* To whom correspondence should be addressed.
Phone: +53 72793956; e-mail: jpino@iiia.edu.cu

Spray drying can be applied to turn sour orange juice into powder that has a longer shelf life and is readily available throughout the year. However, although there are reports pertaining to the drying of concentrated sweet orange juice (Chegini et al., 2008; Goula & Adamopoulos, 2010), there is relatively limited scientific literature concerning spray drying of sour orange juice (Zare et al., 2012). Therefore, the objectives of this work were to produce sour orange juice in powder form by spray drying of unclarified sour orange juice, to determine the optimum drying conditions for the production process and end product quality, and to investigate physical and chemical properties of the powders produced.

1. Materials and methods

Fresh mature sour oranges were obtained from the local market in Merida, Yucatan, Mexico. Chemicals were obtained from Merck (Darmstadt, Germany) and maltodextrin 9≤DE≤14 was supplied by IMSA (Guadalajara, Mexico).

Fruit (100 units) were washed in cold tap water and drained, manually cut apart, and the juice that is localized in the sacs was manually extracted and filtered through a 400 mesh stainless steel sieve. It was immediately stored at −20 °C in 500-ml bags until used. The general characteristics of sour orange juice were the following: total soluble solids 8.5±0.2 °Brix, total acidity expressed as citric acid 35.3±3 g kg^{-1}, and ascorbic acid content 1.20±0.03 g kg^{-1} (data are given as average±standard deviation, n=3).

The spray drying process was performed in a laboratory-scale spray dryer LabPlant SD-05 (Huddersfield, England). Initial trials were performed by varying the different conditions for the spray drying of sour orange juice, such as inlet temperature and maltodextrin content, as found in the literature for other juices (preliminary results not shown). Inlet air temperature (°C) and maltodextrin content (w/w %) were the independent variables, and ascorbic acid retention, volatiles retention, and moisture content were the dependent variables. After analysing the effects of different variables on these dependent variables, the maximum and minimum values of each factor were adjusted for the experimental design. The spray dryer inlet air temperatures evaluated were 120, 140, and 160 °C; the feed mixture concentrations (sour orange juice+maltodextrin) evaluated were 10, 15, and 20% w/w. The lower concentration was selected in a preliminary study as the lowest concentration without provoking excessive powder stickiness on the chamber wall. Other processing parameters were fixed for all drying runs. The feed mixture was maintained under magnetic agitation at 20 °C and fed into the main drying chamber through a peristaltic pump with a drying air flow rate of 63 $m^3 h^{-1}$. The feed flow rate employed was 0.52 l h^{-1}. The drying air inlet flow rate, feed flow rate, and feed temperature were maintained constant throughout the experiments. The juice powders were collected from the cyclone separator, packed in polythene bags, and stored in a desiccator at 20 °C before being submitted to triplicate analyses.

Total soluble solids, citric acid, and ascorbic acid content of sour orange juice and reconstituted spray dried juices were determined according to standard methods (AOAC, 2006). The calculation of ascorbic acid retention during spray drying was based on its content on a dry sour orange basis before and after spray drying, i.e., the moisture contents were taken into account, and for spray dried juice, also the drying aid.

Moisture content of powders was determined in an electronic moisture balance Crode (Merida, Mexico). Measurement of water activity was carried out using an Aqualab water activity meter at 25±0.1 °C (model series CX-2, Decagon Devices, Inc., Pullman, WA, USA).

Volatiles retention during drying was expressed as a percentage of the amount of target volatiles in the feedstock before processing (g of volatiles/g of solid) relative to the amount of volatile in the dried microcapsules produced (g of volatiles/g of solid). Dispersions of 1 g powder or an equivalent amount of fresh juice (3.77, 2.96, and 2.38 g, respectively) and maltodextrin (0.42, 0.52, and 0.59 g, respectively), which represented 100, 150, and 200 g kg^{-1} feedstock, 1.4 g of analytical grade sodium chloride, and 7 ml bidistilled water were prepared in a 15-ml vial sealed with a PTFE-lined screw cap together with a magnetic stirring bar. Analyte extraction was performed by exposing the solid phase microextraction fibre with 50/30 mm divinylbenzene/carboxen on polydimethylsiloxane coating (Supelco, Bellefonte, PA, USA) in the vial headspace for 10 min after the sample matrix was vigorously stirred at 40 °C for 30 min. The fibre coating was retrieved back into the syringe and immediately desorbed in the GC inlet at 250 °C for 2 min to quantify the volatiles. Analytes' separation and detection were performed using Perkin-Elmer Clarus 500 (Shelton, CT, USA). The gas chromatograph injector was set at 250 °C and with splitless mode for 2 min. Helium was employed as the carrier gas at 1 ml min^{-1} constant flow rate in a 25 m × 0.25 mm × 0.25 µm AT-5ms (AllTech, Deerfield, IL, USA) fused silica capillary column. Oven temperature was initially maintained at 50 °C for 2 min, than programmed to 240 °C at 4 °C min^{-1}. The gas chromatograph was connected to a mass selective detector through a transfer line at 250 °C. The detector was operated using a 70 eV electron impact and scan mass range within 35–400 m/z. Compounds were preliminarily identified by use of NIST 05, Wiley 6, NBS 75k, and in-house Flavorlib libraries, and then the identities were confirmed by comparison of their linear retention indices with those of reference standards. External standard calibration was used to compare detector responses from the samples to the responses from the target compounds in the calibration standards (equivalent amounts of fresh juice + maltodextrin) created. The standard was prepared in a 10% ethanol solution in water, spiked with analytes (myrcene, limonene, nonanal, linalool, terpinen-4-ol, and α-terpineol) to a final concentration of 10 mg kg^{-1}.

Juice powders were subjected to microstructural characterization by means of a scanning electron microscope (Tescam 5130 SB, Prague, Czech Republic), setting a magnification equal to ×600. The diameter of each particle was determined by analysing five photo-micrographs of the powder utilized and was expressed as the average particle size D_{43} (µm).

Spray-drying process parameters were optimized by using the desirability function of response surface methodology (RSM) in order to obtain sour orange juice powders with maximum ascorbic acid retention, maximum volatiles retention, and minimum moisture content. Design-Expert version 7.1.5 (Stat-Ease, Inc., Minneapolis, MN, USA) was utilized to perform optimization. The effect of two independent variables, namely A (inlet air temperature) and B (maltodextrin content), on response variables was evaluated by using the RSM. In the present study, a three-level factorial design was employed to (i) study the principal and combined effects of these factors on response variables; (ii) create empirical models between the variables; and (iii) optimize the factors in terms of the response variables studied within the studied levels of inlet air temperature and maltodextrin concentration. Experiments were randomized in order to minimize the effects of unexplained variability in the actual responses due to extraneous factors. The centre point was repeated four times in order to calculate the repeatability of the method (Montgomery, 2013). The adequacy of response surface models was evaluated by using the F-Fischer test. This test was conducted to compare the experimental values with predicted values. The closeness between the experimental and predicted values was shown by the low residual values. On the other hand, no significant (P>0.05) difference was reported between the experimental and predicted values. Therefore, the experimental values were found to be in agreement with predicted values. This observation verified the adequate fitness of the response equations employed for predicting each response variable as a function of the inlet and exit air temperatures. Graphical and numerical optimizations were carried out to determine optimum factors. Therefore, a three-dimensional response surface was plotted to visualize the relationship between the significant (P<0.05) interactive effects of factors and response variables. Optimum levels of independent variables resulting in maximum response variables were pre-established by superimposing all corresponding response surface plots. Numerical optimization was also carried out by using response optimization to predict the exact optimum level of independent variables (factors) leading to the desirable response goals.

2. Results and discussion

Sour orange juice was initially sprayed into the dryer without any additives. According to observations, a sticky layer was formed on the chamber wall. The minimal amount of additives needed to produce powder without any stickiness was obtained by performing several experiments based on a trial-and-error method.

The application of RSM allowed us to study the main and possible interactive effects between the inlet air temperature and maltodextrin content as important indicators of the process. The responses obtained after conducting the 12 experiments established by the experimental design are shown in Table 1, along with the physical parameters of the different experiments evaluated. Response surface models fitted for the response variables indicated that each response variable was assessed as a function of linear, quadratic, and interactive effects of inlet air temperature (A) and maltodextrin content (B). The estimated regression coefficients of the two coded factors, along with the corresponding R^2, P-values, and lack of fit test for the reduced response surface models are given in Table 2. In general, high coefficients of determination were obtained for all polynomial regression models. Thus, this finding indicated satisfactory adjustment of the reduced response models employed for describing the response variables as a function of inlet air temperature and maltodextrin content. The analysis of variance showed that the second-order response surface equations were found to be significantly (P<0.05) fitted for all response variables studied, with the exception of the model for solubility (Table 2). By means of error analysis, it was shown that there was no significant (P<0.05) lack of fit, which indicated that the regression equations fitted the models more than adequately. The outlier *t*-test showed that any run was consistent with the other runs; therefore, the chosen models hold. The closeness between the experimental and predicted values was exhibited by the low residual values ($t < \pm 3.5$).

2.1. Ascorbic acid retention

The loss of ascorbic acid was in the range of 17–29%, which is most likely due to the high temperature during processing. Indeed, ascorbic acid is considered as one of the most heat sensitive nutrients (Diez et al., 2008). In the comparison of the spray drying technique with several other microencapsulation techniques, the former produced minimal loss of ascorbic acid (Uddin et al., 2010). Similar losses in ascorbic acid content during spray drying of guava puree (Chopda & Barrett, 2001; Patil et al., 2014), for example, were reported at the same range of varying temperature levels.

Table 1. Experimental data for response surface analysis

Run	T_I (°C)	MD (%)	Ascorbic acid retention (%)	Moisture content (% w/w)	Volatiles retention (%)
1	120 [–1]	15 [0]	71.7	7.02	62.2
2	140 [0]	15 [0]	81.2	7.07	70.0
3	120 [–1]	10 [–1]	71.2	6.68	60.4
4	160 [+1]	15 [0]	77.1	5.71	74.2
5	160 [+1]	10 [–1]	76.5	6.88	70.1
6	140 [0]	15 [0]	81.4	6.95	69.7
7	140 [0]	15 [0]	80.9	7.00	70.3
8	120 [–1]	20 [+1]	71.4	7.92	63.1
9	160 [+1]	20 [+1]	80.0	5.05	78.4
10	140 [0]	10 [–1]	80.1	7.67	68.2
11	140 [0]	20 [+1]	83.0	6.83	74.1
12	140 [0]	15 [0]	81.6	6.93	69.8

Table 2. Main effects and interactions of the coded response surface models

	Ascorbic acid retention	Moisture content	Volatiles retention
Intercept	81.26	6.99	70.18
A	3.22**	–0.66**	6.17**
B	1.10**	–0.24**	2.82**
AB	0.83*	–0.77**	1.40**
A^2	–6.82**	–0.62**	–2.45**
B^2	0.33	0.27*	0.50
R^2	0.993	0.983	0.994
F model	172.77**	68.95**	217.68**
Lack of fit (F-value)	4.39	8.50	6.87

A: Inlet air temperature; B: maltodextrin content; *: significant at P<0.01;
**: significant at P<0.001

According to the response surface model (Table 2), inlet air temperature (A) was the variable that showed the greatest influence on ascorbic acid retention. The positive coefficient of the first order term indicated that ascorbic acid retention of the sour orange powder increased significantly by increasing inlet air temperature, but the negative effect of the quadratic term was higher than the first order term, so there is a curvature in the contour plot (Fig. 1), with maximal retention in the range 140−150 °C within the experimental ranges studied. Inlet air temperature is directly proportional to the microcapsule drying rate and the final water content. When the inlet air temperature is low, the low evaporation rate causes the formation of microcapsules with high density membranes, high water content, and slow evaporative rate, consequently causing an important loss of ascorbic acid. On the other hand, a high inlet air temperature causes an excessive evaporation resulting cracks in the membrane and inducing subsequent premature degradation of encapsulated ascorbic acid by contact with oxygen. Thus, the inlet air temperature is usually determined by the temperature, which can safely be used without damaging the product (Gharsallaoui et al., 2007).

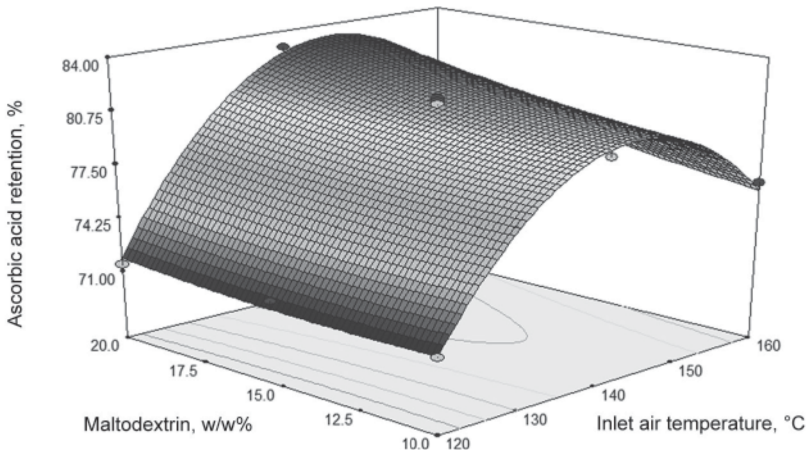

Fig. 1. Effect of inlet air temperature and maltodextrin content on the ascorbic acid retention of sour orange juice

Maltodextrin content (B) significantly affects the ascorbic acid retention of sour orange powder. Increasing the amount of drying agent in the feed mixture produced an increase in ascorbic acid retention. This is probably due to the high encapsulation rate of excess maltodextrin present in the feed mixture, thereby preventing ascorbic acid from degradation. Indeed, similar results were reported, for example, during spray drying of acerola pomace extract (Moreira et al., 2010).

2.2. Moisture content

The moisture content of the powders varied from 5.05 to 7.92% w/w, which is below the target range of spray drying process. As can be seen from the response surface model (Fig. 2), increases in inlet air temperature and maltodextrin content led to lower moisture contents. According to the response surface model (Table 2), inlet air temperature (A) was the variable that demonstrated the greatest influence on moisture content. The greater the temperature difference between the drying medium and the particles, the greater will be the rate of heat transfer into the particles, which provides the driving force for moisture removal. When the drying medium is air, temperature plays the second important role. As water is driven from the particles in the form of water vapour, it must be carried away or the moisture will create a saturated atmosphere at the particle surface. This will slow down the rate of subsequent water removal. The hotter the air, the more moisture it will hold before becoming saturated. The present findings are in agreement with similar results obtained during spray drying of different fruit juices (Rodríguez-Hernández et al., 2005; Goula & Adamopoulos, 2010; Solval et al., 2012).

The moisture content reveals a decrease with an increase in maltodextrin content. These findings could be accounted for the fact that additional concentrations of drying aid resulted in an increase in feed solids and a reduction in total moisture for evaporation. Many other authors reported a reduction of moisture content with increasing maltodextrin concentration

(FAZAELI et al., 2012; HORUZ et al., 2012). However, in the spray drying of concentrated orange juice an increase in moisture content with an increase in maltodextrin concentration was reported (GOULA & ADAMOPOULOS, 2010). These authors concluded the presence of larger maltodextrin molecules made it difficult for water molecules to diffuse, but they employed high concentrations of maltodextrin (25−400%) for producing powder and this increasing carrier concentration resulted in an increase in moisture content.

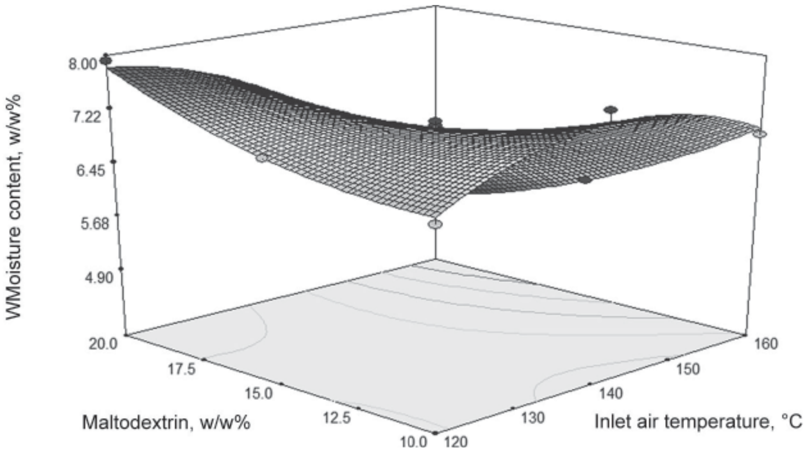

Fig. 2. Effect of inlet air temperature and maltodextrin content on the moisture content of sour orange juice

2.3. Volatiles retention

Although flavour is an important sensory characteristic for the overall acceptability of any food product, quantitative study on the flavour volatiles of spray-dried fruit juice powders is limited. It is expected that the higher the retention of volatile compounds is, the higher the flavour quality will be. The loss of volatile compounds was in the range of 22–40%, possibly due to the use of high temperature during processing. Since the volatiles evaporated from the droplet surface at a faster rate than water molecules during the constant stage in drying, the loss of a certain amount of volatiles was inevitable. However, the volatile profiles of the samples were similar and the residual amount of volatiles in sour orange juice powder was similar to the level of volatiles retained in spray dried fruit products such as pear (KOMES et al., 2007) and durian juice (CHIN et al., 2010).

According to the response surface model (Table 2), inlet air temperature (A) was the variable that showed the greatest influence on volatiles retention (Fig. 3). The positive coefficient of the first order term indicated that this factor increased significantly by increasing inlet air temperature. These findings could be accounted for the fact that increment of inlet air temperature improved the volatiles retention by enhancing the rate of droplet film formation (REINECCIUS et al., 1982).

Increasing the amount of drying agent (B) in the feed mixture caused an increase in volatiles retention. This may be due to the high encapsulation rate of excess maltodextrin present in the feed mixture, preventing volatiles release during drying (REINECCIUS et al., 1982).

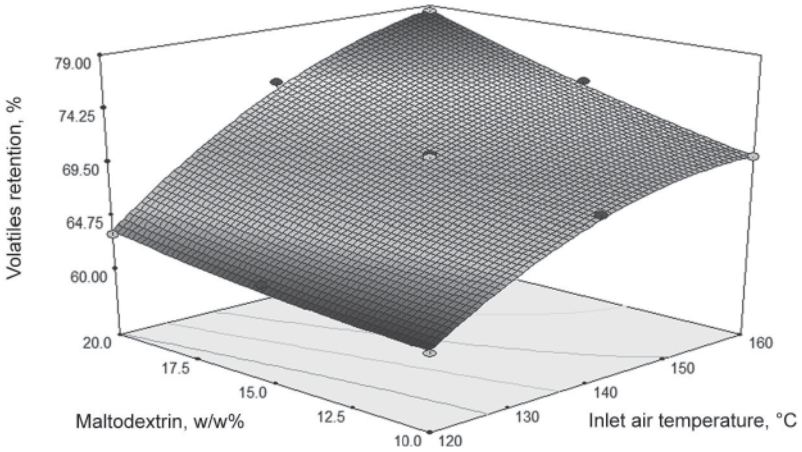

Fig. 3. Effect of inlet air temperature and maltodextrin content on the volatiles retention of sour orange juice

2.4. Optimization

Optimum conditions for sour orange juice powders were determined to obtain maximum ascorbic acid and volatiles retention and minimum moisture content within the studied ranges of inlet air temperature and maltodextrin concentration. To determine optimum spray-drying process conditions, desirability function was used for numeric and graphic optimization (Fig. 4). From the optimization, an inlet air temperature of 156 °C and a 20% w/w maltodextrin were suggested to be optimal for spray drying of sour orange juice. The desirability function at this point was 0.89, close to the maximum of 1. Under such conditions, ascorbic acid retention was estimated to be 81.5%, 5.5% w/w moisture content, and volatiles retention 78%.

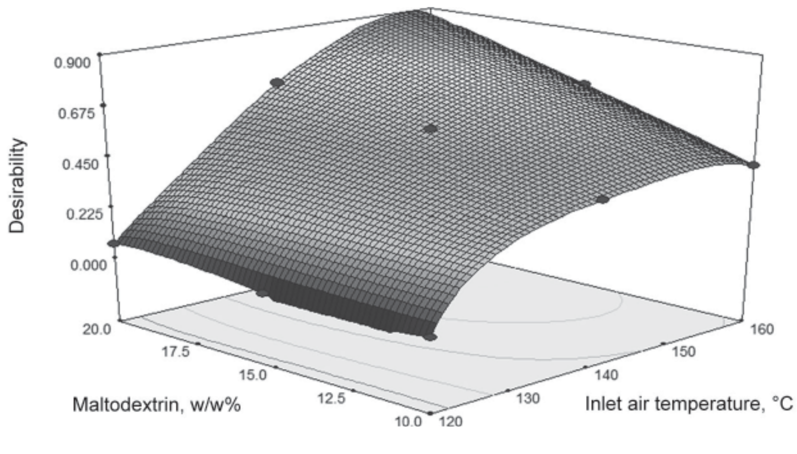

Fig. 4. Response surface plot for optimization of inlet air temperature and maltodextrin content on the spray drying of sour orange juice

Regarding the microstructure of the powder of the treatment obtained by spray dryer using the optimized parameters (Fig. 5), it was noted that the obtained particles demonstrated as a general characteristic a higher degree of uniformity regarding shape and good distribution of the particles (smooth and intact surfaces). However, they show several sizes and disfigured particles that are rarely seen. A certain percentage of particles with higher magnitude show adherence of smaller particles on their surfaces. However, it can be affirmed that particles of powder in this system revealed amorphous surfaces. The average particle size D_{43} was 18.41±0.37 µm. When compared with other spray dried juices (CHEGINI & GHOBADIAN, 2007; TONON et al., 2008), the sour orange juice powder obtained with the optimized parameters was more homogeneous and presented a smaller mean diameter, which is a desired result since, in general, water solubility is enhanced as particle size is reduced.

The water activity (a_w) of the powder obtained by spray dryer using the optimized parameters was 0.302±0.009. Generally, foods with a_w<0.6 are considered to be microbiologically more stable (QUEK et al., 2007).

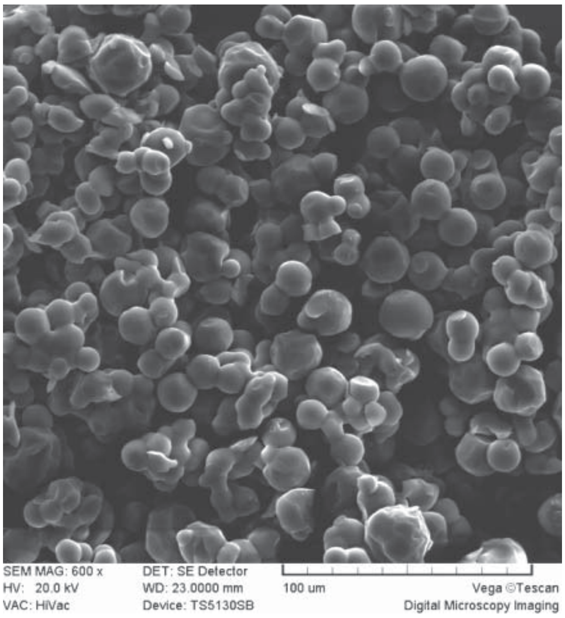

Fig. 5. SEM micrograph of sour orange juice spray dried with the optimized parameters

3. Conclusions

Within the experimental ranges studied, the predicted conditions to simultaneously provide the maximum possible retention of ascorbic acid and volatiles were: inlet temperature 156 °C and maltodextrin content 20% w/w. Under such conditions, ascorbic acid retention was estimated to be 81.5%, 5.5% w/w moisture content, volatiles retention 78%, and water activity of 0.302. The product with these specifications is acceptable from a consumer perspective and potentially commercially viable.

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