

Retention capacity of maltodextrin antioxidants in cranberry juice by spray drying process

Capacidad de retención de antioxidantes de maltodextrina en jugo de arándano en polvo secado por aspersión

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DOI: 10.35429/EJE.2019.10.6.12.20

Received March 18, 2019; Accepted June 30, 2019

Abstract

A group of four maltodextrins with different degrees of dextrose equivalent (DE) was used as carriers for the spray drying of cranberry juice, with the objective of evaluating the effect of drying temperature, DE grade and concentration of maltodextrin on the performance of cranberry juice powder and the ability to retain antioxidants. For this purpose, the conditions of the spray drying process were: inlet air temperature: 170-210° C, maltodextrin type (Mc, M10, M20 and M40) and maltodextrin concentration: 10-30% w/w. The different powders obtained from the spray drying process were analyzed by high performance liquid chromatography (HPLC) for the quantification of antioxidants (resveratrol and quercetin). The analysis of the data was carried out from a design of D-Optimal experiments. The results of the analysis showed that the highest yield of cranberry juice powder is achieved by using 30% w/w of M10 at a drying temperature of 193 °C. Such conditions allowed a retention of antioxidants in the powder of 20% and 30% of resveratrol and quercetin, respectively.

Antioxidants, Cranberry, Spray drying process

Resumen

Un grupo de cuatro maltodextrinas fueron utilizadas como agentes acarreadores para el secado por aspersión de jugo de arándano, con el objetivo de evaluar el efecto de la temperatura de secado y la concentración de maltodextrina sobre el rendimiento de jugo de arándano en polvo y la capacidad de retención de antioxidantes. Para tal efecto las condiciones del proceso de secado por aspersión fueron: temperatura de aire de entrada: 170-210°C y concentración de maltodextrina: 10-30% p/p. Los diferentes polvos obtenidos del secado por aspersión, fueron analizados mediante cromatografía líquida de alta resolución (HPLC) para la cuantificación de dos antioxidantes. El análisis de los datos se llevó a cabo a partir de un diseño de experimentos D-Optimo. Los resultados del análisis realizado mostraron que el mayor rendimiento de jugo de arándano en polvo se logra al utilizar 30% p/p de M10 a una temperatura de secado de 193 °C.

Antioxidantes, Arándano, Polvo secado por aspersión

Citation: SAAVEDRA-LEOS, María Zenaida, SILVA-CÁZARES, Macrina Beatriz, GONZÁLEZ-TREVIZO, Cynthia Lizeth and TERRONES-GURROLA, María Cruz del Rocío. Retention capacity of maltodextrin antioxidants in cranberry juice by spray drying process. ECORFAN Journal-Ecuador. 2019. 6-10: 12-20

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Introduction

Antioxidants are bioactive compounds capable of neutralizing unstable free radicals that cause aging and chronic degenerative diseases, such as cancer, diabetes mellitus, cardio and cerebrovascular diseases, among others (Lee et al., 2015; Masisi et al., 2016; Polanski et al., 2015). From a nutritional point of view, the consumption of foods rich in antioxidants is one of the most effective ways to reduce the risk of chronic diseases.

Cranberries have been recognized for its high content of phenolic and flavonoid compounds, such as quercetin, resveratrol, myricetin and kaempferol (Parada-Caro, 2005). However, the use of this fruit as a source of antioxidants is restricted, as it is a perishable food with a high aqueous content of up to 85% (Hyun-Chun et al., 2012). In 2014, Mexico obtained a production of 5,529 tons of fresh cranberry (Sagarpa, 2014) of which 50% was processed in the form of various products, such as juices, nectars, yogurts, jams and semi-prepared foods, like powders, syrups and reconstitute concentrates (Skrovankova et al., 2015; Waheeda et al., 2015). Unfortunately, during the processing of the fruit, there are losses of the antioxidant compounds, associated with the heat treatments since the phenolic compounds are heat sensitive at temperatures above 60 °C (Wang et al., 2009).

The lack of adequate conservation methods that allow lengthening the shelf life, obtaining minimal losses of volatile components (phenolic and flavonoids) and maintaining the nutritional and organoleptic qualities during the processing and storage of the cranberry, has caused losses of 20% to 50% (Feippe-Fernández, 2013; Cano-Chauca et al., 2005). This leads to the search for more appropriate conservation methods. A viable alternative is spray drying, which has been preferred as an economical, flexible process, easy to operate and control, with high drying rates (Silva et al., 2008; Porras-Saavedra et al., 2015) and very short residence times, allowing heat-sensitive products to be dehydrated at relatively high temperatures, retaining some properties of foods such as taste, color, smell and nutrients (Masters, 1991; Mujumdar, 1998). As a result, a powder product is obtained with a longer shelf life and a reduced volume, a useful feature in terms of storage and transport (Cano-Chauca et al., 2005).

However, the difficulty of applying spray drying in products, such as fruit juices, is due to high water and sugar contents, which have a low glass transition temperature (T_g) (Van Sleuwen et al., 2012; Saavedra-Leos et al., 2012; Collares and Kieckbush, 2002; Vanhal and Blond, 1999; Ruan et al., 1999). This causes low yields, operating problems and difficulty in predicting product quality. A practical solution is the use of high molecular weight additives (adjuvants) as carrier agents to modify the properties of the material in order to reduce the problem of sticking during spray drying (Saavedra-Leos et al., 2015; Ceballos -Peñalosa, 2008). The amount of carrier agent used ranges from 20% to 60% w/v for food dehydration (Masters, 1991).

In spray drying, maltodextrin is used as a carrier agent mainly because of its high molecular weight, low viscosity and glass transition temperature (T_g) in a range of 100 to 188 °C, which depends on its dextrose equivalent (DE). This polysaccharide can have a DE value of 5-40 and depending on it, maltodextrin has different physicochemical and functional properties (Udomrati and Gohtani, 2015; Reineccius, 1991). Based on the above, it can be established that factors such as spray drying temperature and the type and percentage of carrier agent are determinants to improve the performance and quality of powdered juices. It is important to carry out systematic studies based on the use of statistical tools that allow evaluating the effect of these factors, in order to establish the optimal conditions for the processing of fruit juices guaranteeing a good efficiency.

That is why in the present work a design of D-Optimal experiments was used to evaluate four maltodextrins and concentrations of 10 to 30% w/w, as well as drying temperatures of 170 to 210 °C, to obtain cranberry juice powder and evaluate the performance and retention capacity of antioxidants.

Methodology

Spray drying

Dehydration was carried out in a Mini Spray Dryer B290 (Buchi, Switzerland). The samples were placed in the dryer at room temperature and a volumetric flow of hot air of 35 m³/h was used, adjusting the vacuum to 70% capacity.

A constant compressed air pressure of 1.5 bar was used. A total of 25 experiments were performed, where a range of inlet temperatures between 170 °C and 210 °C was handled, while the output was recorded between 70 °C and 90 °C. As the encapsulating agent, maltodextrin (MD) with different equivalents of dextrose (Mc, M10, M20, M40) was used at different concentrations as specified in Table 1.

Random	Run	Factor 1 T (°C)	Factor 2 C (g)	Factor 3 MD*
12	1	181.297271	30	M40
22	2	193.06311	30	M10
14	3	193.06311	30	M10
18	4	190	25.043167	M20
5	5	210	30	MC
11	6	170	10	M20
1	7	170	30	MC
16	8	175.00002	12.5	MC
19	9	209.375	24.1630577	M40
4	10	210	10	M40
21	11	170	10	M40
17	12	190	19.6296296	M10
8	13	170	10	M40
23	14	170	10	M20
20	15	210	20	MC
3	16	170	30	M20
24	17	181.297271	30	M40
2	18	186.541152	10	M10
10	19	170	21.6266586	M10
7	20	210	10	M20
13	21	189.99939	20.0003722	MC
6	22	210	18.3871388	M10
9	23	210	10	MC
25	24	170	21.6266586	M10
15	25	210	30	M20

*MD= maltodextrin

Table 1 Design of experiments for D-Optimal analysis
Source: Prepared by the authors

Design of experiments

A design of D-optimal experiments was used in Minitab 17, to evaluate the effect of the concentration of the carrier agent with different degrees of dextrose and different drying temperatures, in order to establish the optimal conditions for obtaining the best yields of cranberry juice powder. The 3 factors to be analyzed were classified as follows: 1) Temperature (T): 170, 175, 181, 187, 190, 193, 209 and 210 °C; 2) Maltodextrin concentration (C): 10, 12.5, 18, 20, 22, 24, 25 and 30% w/w; and 3) Type of maltodextrin (MD): Mc, M10, M20, M40.

The response variables were: 1) Yield of dust collected in % w/w; and 2) Retention of antioxidants from cranberry juice in µg/mL.

The design consisted of 25 experimental runs (Table 1), which were randomly selected in order to determine the response variables for each sample. To draw the contour curve graphs, a numerical assignment was made to the type of maltodextrin factor of 0, 10, 20 and 40 for Mc, M10, M20 and M40, respectively. Similarly, an ANOVA analysis was performed to estimate the effect of the factors evaluated on the performance of the powder juice obtained.

Determination of antioxidant content by High Performance Liquid Chromatography (HPLC)

HPLC was carried out with a Waters chromatographic equipment (Waters Assoc. Milford MA, USA) composed of a Binary pump (HPLC model 1525), an autoinjector (model 717) and a double wavelength absorbance detector (model 2487).

The quantification of resveratrol and quercetin was performed under the following operating conditions: room temperature with a mobile phase of acetonitrile: formic acid 0.01% pH 3.0 (70:30 v/v) at a flow of 1 mL/min and a length of 306 nm wave. Chromatographic separation was carried out on a column of the Agilent C18 brand, (75 x 4.6 mm ID 3.5 µm) with an injection volume of 10 µL. All data was processed with the Empower Pro Version 4.0 program.

The calibration curves were made from stock solutions of resveratrol (1000 µg/mL) and quercetin (1000 µg/mL), prepared in HPLC grade methanol. The dilutions used were 0.01, 1, 5, 10 and 20 µg/mL, to prepare the six-point calibration curve. All solutions were prepared the same day as their injection.

Approximately 0.5 g in triplicate were weighed for sample extraction, to which a mixture of 0.01% acetonitrile and phosphoric acid (50:50 v/v) was added. Subsequently, it was vigorously stirred for 5 minutes and left in a dark place for 24 hours. Finally, the solutions were passed through Acrodisc filters of 0.45µm and were injected into the equipment.

Results

Table 2 shows the yields of obtaining dried cranberry juice under the different established conditions.

MD*	MD* Concentration (% weight)	Cranberry juice concentration (% weight)	Drying temperature (°C)	Yield (%)
M40	30	70	181	2.02
M10	30	70	193	9.68
M10	30	70	193	11.87
M20	25	75	190	2.81
Mc	30	70	210	6.74
M20	10	90	170	0
Mc	30	70	170	6.37
Mc	12.5	87.5	175	1.82
M40	24	76	209	7.43
M40	10	90	210	0
M40	10	90	170	0
M10	20	80	190	8.15
M40	10	90	170	0
M20	10	90	170	0
Mc	20	80	210	7.68
M20	30	70	170	4.23
M40	30	70	181	1.25
M10	10	90	187	0.12
M10	22	78	170	9.48
M20	10	90	210	0
Mc	20	80	190	7
M10	18	82	210	5.45
Mc	10	90	210	0
M10	22	78	170	8.37
M20	30	70	210	3.03

*MD= maltodextrin

Table 2 Efficiency and drying conditions of maltodextrins
Source: Prepared by the authors

The highest yield was obtained by using a 20% w/w concentration of maltodextrin at a process temperature of 210 °C, with a weight yield of 7.68%. When using a concentration of 30% w/w at 210 and 170 °C, a lower yield of 6.74% and 6.37%, respectively, was obtained.

In the case of a concentration of 12.5 and 10% w/w of maltodextrin, a collapse of the system associated with problems of bonding of the material in the walls of the drying chamber was observed. These results are similar to those reported by Saavedra et al. (2015) when using a 30% w/w concentration of carrier agent; however, there are no similar reports of system collapse when using 20, 12.5 and 10% w/w of maltodextrin.

In the case of M10 and based on experiment design, the concentrations handled were: 10, 18, 20, 22 and 30% w/w of carrier. Of the results analyzed in Table 2, the highest yield was obtained using a concentration of 30% w/w maltodextrin at 193 °C, with a yield of 11.87%. From the duplicate under these same conditions, a yield of 9.68% was obtained, with a general average of 10.77 ± 1.5485 . On the other hand, at a concentration of 22% w/w and 170 °C, a yield of 9.48% and 8.37% was obtained in duplicate, giving an average of 8.925 ± 0.7848 .

Likewise, when using a concentration of 20% w/w at 190 °C, a yield of 8.15% was reached. In the case of a concentration of 18% w/w and a process temperature of 210 °C, a yield of 5.45% was obtained. And finally, at 10% w/w of carrier agent the system collapsed, so the yield obtained was only 0.12%. These results are consistent with those reported by Caliskan and Dirim (2013), who studied the feasibility of a spray drying process of sumac extract (*Rhus coriaria* L.) with the addition of maltodextrin DE 10.

They used inlet/outlet temperatures of air of 160/80, 180/90 and 200/100 °C, and adjusted the total solids content of the extract to 10, 15, 20 and 25% w/w with the addition of maltodextrin DE10. The researchers obtained yields of 70.21%, 86.77%, 97.45% and 98.5% for extracts with 10, 15, 20 and 25% maltodextrin (ED 10) respectively, which allowed to conclude that increasing the concentration of carrier agent increased the yield of the powder obtained. On the other hand, Nadeem et al. (2011) obtained similar results to those reported in this work when using M10.

These researchers sprayed mountain tea (*Sideritis stricta*) using maltodextrin DE12 as a carrier at two concentrations (3 and 5% w/w) and three different drying temperatures (145 °C, 155 °C and 165 °C). They found that the increase in product performance was related to the concentration of carrier and not to the temperature process, which may be associated with a higher solids content of the feed solution. In this regard, Ameri and Maa (2006) point out that increasing the total solids content of the feed solution is a way to increase the recovery of powder in spray drying operations.

Regarding M20, concentrations of 10, 25 and 30% w/w of maltodextrin were used. When comparing the results in Table 2, it is observed that the highest yield was obtained by using a concentration of 30% w/w at a process temperature of 170 °C, with a weight yield of 4.23%. At this same concentration and at a temperature of 210 °C, a yield of 3.03% was obtained, while for a concentration of 25% w/w of maltodextrin at a process temperature of 190 °C a yield of 2.81% was obtained. In the case of a 10% w/w concentration of maltodextrin at temperatures of 170 and 210 °C respectively, the samples collapsed.

These results are similar to those reported by Bhusari et al. (2014), who studied the effects of different carrier agents, including DE20 maltodextrin, on the physical and microstructural properties of spray dried tamarind pulp powder. They observed an improvement in product recovery by increasing the concentration of the carrier. This increase in the percentage of powder recovery is attributed to the reduction in the adhesion and deposition of powder particles in the walls of the drying chamber, as well as to the increase in the glass transition temperature resulting from the mixing of the carrier agent and tamarind pulp.

On the other hand, Peng et al., (2013) showed similar results to those obtained in this work; they used maltodextrin DE20 when spray dried purple sweet potato flour with p/w percentages of maltodextrin from 10 to 40%, observing a greater increase with a 30% concentration of maltodextrin. However, they observed that as the percentage of maltodextrin increased from 30 to 40%, there were slight changes in yield, but a significant decrease in the quality of the powder obtained.

Finally, for M40 maltodextrin, concentrations of 10, 24 and 30% w/w were used. According to the results of Table 2, it was observed that the highest yield was obtained by using a concentration of 24% w/w at a process temperature of 209 °C, with a value of 7.43%. For a concentration of 30% w/w and a process temperature of 181 °C, a lower yield of 2.02% and 1.25% was obtained, with a general average of 1.635 ± 0.5444 . In the case of a 10% w/w concentration of maltodextrin at temperatures of 170 and 210 °C respectively, said samples collapsed.

These results are similar to those reported by Saavedra et al., (2015) who analyzed the relationship between the degree of polymerization and the physicochemical properties of 4 samples of maltodextrin with different equivalents of dextrose, including maltodextrin DE40, under a spray drying process. In his research the performance of the product was not studied.

However, they mention that the lowest glass transition (T_g) was presented by maltodextrin with a greater degree of dextrose equivalents (M40), so it can be assumed that the lowest yields were obtained when using M40, since the primary effect of the addition of adjuvants such as maltodextrins lies mainly in increasing the T_g of the system to avoid problems of sticking and collapse in the dryer chamber.

It can be observed in Table 2 that the highest percentage was achieved by using 30% w/w of M10 at a process temperature of 193 °C. With these results, it can be perceived that the use of the M10 carrier agent in the spray drying process of cranberry juice favors the obtention of higher yields. These results are consistent with those obtained by Saavedra et al., (2015) who proposes the use of M10 as a carrier agent in spray drying of sugar-rich systems such as fruit juices, due to their physical ($T_g = 100$ °C and 90 °C, $T_d =$ greater than 190 °C) and chemical properties (experimental DE = 1 and Degree of polymerization = 16).

D-Optimal Analysis

Of the treatments proposed in the D-optimal design, a yield range of cranberry juice from 0 to 11% was obtained. From the combination of the three factors evaluated, we prepared the graphs corresponding to the contour curves (Figure 1). To identify the regions associated with the best yields, yield curves greater than 10% were analyzed, which are presented in an intense green color in Figure 1.

For the drying temperature-concentration ratio of maltodextrin (Figure 1a), there are two optimal regions, the first between 20-23% maltodextrin and 170-175 °C, and the second at concentrations of 28-30% maltodextrin in an interval from 190-200 °C. In Figure 1b where the contour curves of the drying temperature-type of maltodextrin ratio are shown, the optimal region for M10 is set at temperatures of 170 ° and 190-195 °C.

Finally, for the concentration-type of maltodextrin ratio (Figure 1c) the optimal region corresponds to M10 at a concentration of 30% maltodextrin. Based on the results obtained from the contour curves, it can be established that M10 maltodextrin is the carrier agent that allows obtaining the best yields at a concentration of 30% and temperatures between 190-195 °C.

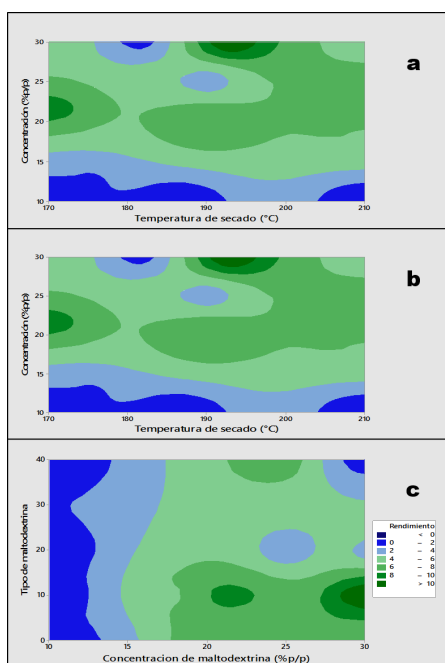


Figure 1 Contour graphs for the relations a) Drying temperature-concentration of maltodextrin, b) Drying temperature-type of maltodextrin, c) Concentration of maltodextrin-type of maltodextrin

Source: Prepared by the authors

Table 3 shows the results of the analysis of variance ($\alpha = 0.05$), where the independent effect is determined. It shows that the concentration and type of MD are the only factors that have a significant effect on the yield of the powdered juice, while the temperature showed no significant effect. Based on these results, it can be concluded that to obtain the best yields the determining factors to consider are the concentration and type of MD.

Source	GL	SC	MC	F	p
Temperature	8	119.7412	14.9676	5.854	0.248
Concentration	6	190.0694	31.6782	1.303	0.011
Type of MD	5	9.1718	1.8344	2.578	0.000
Error	3	0.6160	0.2053		
Total	22	319.5985			

Table 3 Analysis of variance for the performance of cranberry juice powder.

Source: Prepared by the authors

Antioxidant retention

The results corresponding to the retention capacity of antioxidants in cranberry juice are shown in Table 3. It is observed that the sample AJ-M10 at 30% (w/w) and 193 °C was the only sample that managed to retain antioxidants. Regarding resveratrol, this varied from 0.662 $\mu\text{g}/\text{mL}$ in cranberry juice to 0.140 $\mu\text{g}/\text{g}$ powder with maltodextrin (AJ-Mc).

While for quercetin 3-D-galactoside the content ranged from 0.326 $\mu\text{g}/\text{mL}$ in cranberry juice, to 0.093 $\mu\text{g}/\text{g}$ powder in the AJ-M10 sample at 30% (w/w) at 193 °C. These results coincide with those reported by Robert et al., (2010), who encapsulated bioactive compounds of pomegranate juice and ethanolic extracts with maltodextrin and soy protein isolate by spray drying.

Bakowska-Barczak and Kolodziejczyk (2011) found that inulin was less effective in the encapsulation of blackcurrant polyphenols (*Ribes nigrum* L.) than maltodextrin. While Santiago-Adame et al. (2015) managed to retain about 60% of polyphenols by spray drying cinnamon infusions, using maltodextrin as an encapsulating material.

González et al. (2011) evaluated a series of treatments to conserve volatile watermelon juice compounds with spray drying, where they obtained a greater conservation of these compounds by using a mixture of maltodextrin DE10 and gum arabic 1:1 p/p and less conservation when using only maltodextrin or only gum arabic as carrier. Likewise, Quirino-Lacerda et al. (2016) mention in their work that when performing spray drying of jussara pulp, a higher anthocyanin content was obtained by using mixtures of maltodextrin and inulin as the wall material.

MD	MD Concentration (%)	Cranberry juice concentration (%)	Drying temperature (°C)	Efficiency (%)	Antioxidants $\mu\text{g}/\text{mL}$	
					Resveratrol	Quercetin
AJ-M40	30	70	181	2.02		
AJ-M10	30	70	193	9.68		
AJ-M10	30	70	193	11.87	0.140	0.093
AJ-M20	25	75	190	2.81		
AJ-Mc	30	70	210	6.74		
AJ-M20	10	90	170	0		
AJ-Mc	30	70	170	6.37		
AJ-Mc	12.5	87.5	175	1.82		
AJ-M40	24	76	209	7.43		
AJ-M40	10	90	210	0		
AJ-M40	10	90	170	0		
AJ-M10	20	80	190	8.15		
AJ-M40	10	90	170	0		
AJ-M20	10	90	170	0		
AJ-Mc	20	80	210	7.68		
AJ-M20	30	70	170	4.23		
AJ-M40	30	70	181	1.25		
AJ-M10	10	90	187	0.12		
AJ-M10	22	78	170	9.48		
AJ-M20	10	90	210	0		
AJ-Mc	20	80	190	7		
AJ-M10	18	82	210	5.45		
AJ-Mc	10	90	210	0		
AJ-M10	22	78	170	8.37		
AJ-M20	30	70	210	3.03		
					0.662	0.326

Table 4 Resveratrol and quercetin antioxidant concentration of cranberry juice powder

Source: Prepared by the authors

Although maltodextrin has been one of the most used carrier agents for drying fruit juices, it is evident that it has a limited capacity to retain the antioxidants present in cranberry juice.

So it would be interesting and a better alternative to use mixtures of maltodextrin with other carrier agents to improve the retention of antioxidants.

Conclusions

Cranberry juice powder was obtained by using Mc, M10, M20 and M40 at concentrations of 30, 20, 25, 24, 22 and 18 w/w and a process temperature of: 210, 209, 193, 190, 181 and 175, 170 °C.

The Mc, M10, M20 and M40 systems collapsed when using a concentration of 12.5, 10 w/w at a process temperature of 210, 175 and 170 °C.

The highest percentage of juice powder was obtained at 30% w/w of M10 at a process temperature of 193 °C, which favored higher yields.

Two optimal process regions are established: a) maltodextrin concentrations at 20-23 w/w and a range of 170-175 °C, b) concentrations of 28-30% maltodextrin in a range of 190-200 °C.

The optimal region is established for M10 at temperatures of 170 ° and 190-195 °C.

For the concentration-type of maltodextrin ratio it was determined that the optimal region corresponds to M10 at a concentration of 30% maltodextrin.

Based on the results obtained from the contour curves, it can be established that M10 maltodextrin is the carrier agent that obtains the best yields, it was also the only MD that presented resveratrol and quercetin antioxidant retention in an approximate percentage of 20% and 30%, respectively.

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