e-ISSN:2321-6204
p-ISSN:2347-2359

Research & Reviews: Journal of Food and Dairy Technology

# Functional Properties, Color and Betalain Content in Beetroot-Orange Juice Powder Obtained by Spray Drying

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**Research Article**

Received date: 11/05/2015
Accepted date: 18/05/2015
Published date: 26/05/2015

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**Keywords:** Spray drying, Beetroot, Betalains, Stability, Juices.

**ABSTRACT**

Betalains are water-soluble pigments mainly found in red beetroots. Stability of betalains in processed and storaged products is generally affected by numerous factors such as the matrix constituent, chelating agents, water activity, pH, temperature, oxygen and light. This research paper reports the functional properties, color, and betalain content in beet root-orange juice powder produced by spray drying. A mixture of beet root juice and orange juice (60/40) was spray dried using 3, 5 and 7% (w.b.) of maltodextrin DE 10, three different inlet air temperatures of 130 °C, 140 °C and 150 °C and two feed flow rates (8 and 10 mL/min). The powders obtained were analysed for moisture content, bulk density, hygroscopicity, wettability, solubility, color and total pigments. It was found that processing juice at 140°C with feed flow rate of 8 mL/min and 5% of maltodextrin gave the best functional properties and the conservation of betalains was high.

## INTRODUCTION

Food coloring of fruits and vegetables suffer, in general, color degradation during processing, losing an important quality indicator parameter. The beetroot is a great source of antioxidant components called betalains, which are divided into betacyanins and betaxhantins, the former imparts the purple color to beetroot and the latter gives the yellow-orange color. Red beet has been reported as one of the ten vegetables with most potent antioxidant capacity [1]. Medical studies have demonstrated health related beneficial properties of beetroot such as lowering cholesterol and arterial pressure, and it has being considered a good source of vitamin B complex and folic acid. Stability of betalains is affected by quelating agents, water activity, nitrogen atmosphere, degree of glucosylation or acylation, pH, temperature, light, oxygen and moisture [2-4]. Pedreno and Escribano [5] reported that at pH 3.5 the degradation of betanine was slight, while at pH 8.5 the betanine concentration fell more than 60%, they also reported high antiradical activity at pH 3.5. A pH range between 5 and 7 has been reported to be ideal for the stability of betalains. Von Elbe [6] found that the highest stability of betanins in a model system was between pH 4 and 5, and pointed that pH=5 were most suitable to preserve the betanins in beetroot juice at 100 °C. Stintzing and Carle [7] have reported a pH about 4 as suitable for processing. Addition of antioxidants like ascorbic and isoascorbic acids has been reported to improve betalain stability by oxygen removal. Herbach found better stabilizing effect of betacyanins in pitaya juice heated at 85 °C for 1 hour at pH 4 adjusted with 1.0% ascorbic acid, probably due to that the electrophilic center of betanin was partially neutralized. In order to preserve components of interest in liquid food products, spray drying technique has been widely investigated and used [8-11]. Juices of different vegetable products, contain varied proportions of glucose, fructose, and organic acids, these components have very low glass transition temperature (Tg), high hygroscopicity, low melting point and high solubility resulting in a sticky product when spray dried, leading to different state transitions properties, which are related with collapse, stickiness, agglomeration, crystallization, and caking [12,13]. Dried products are generally in a glassy amorphous form, where the mobility is very limited, but if the temperature is increased above glass transition temperature (Tg) the molecular mobility is accelerated, resulted in an increased rate of physicochemical changes such as sticking, collapse, caking, loss of volatiles, oxidation and aggregation [12]. In order to overcome the above phenomenon it is necessary to add an encapsulating material to obtain a successful powder production, to improve physical properties of powder and to protect the active ingredients in the raw material. In Mexico, it is a common practice to mix beetroot with orange juice, in order to make the former more palatable. Additionally, the orange juice lowers the pH because of its ascorbic acid content. The objective of this study was to establish the effect of processing condition on beetroot-orange juice powder characteristics.

## MATERIALS AND METHODS

### Fresh juice preparation

Beet roots and oranges were purchased from a local supermarket in Durango, Mexico. The respective fruit juice was obtained according to the nature of each product. The juices obtained were vacuum-filtered through a piece of cotton cloth and then transferred into 500 mL PET bottles. The filtered juices were mixed in proportions of 60 beetroot and 40 orange juice by volume to result in a pH of 4.6 which is appropriate for pigment stability.

### Spray drying

A Buchi Mini spray dryer B290 was used for dehydration. The additive used in this work as coating material was Maltodextrin DE 10 obtained from Industrializadora de Maiz (Guadalajara, Mexico) and used in three levels: 3, 5 and 7% (w.b.). These percentages were chosen on the basis of preliminary experiments which yielded what appeared to be an acceptable spray dried powder. The spray drying conditions employed were: inlet air temperature (130,140 and 150ºC) and feed flow rate (8 and 10 mL/min). The powder collected from the cyclone and from the walls of spray dryer was weighed and stored in 200 mL glass jars, tightly closed and stored under darkness at ambient temperature for no more than two days before analysis. The total powder collected was used to establish the performance of the dryer. The experiments were repeated twice.

### Assessment of analytical and functional properties

Analyses of powders were carried out immediately after the spray drying process. Moisture content was determined using the Ohaus MB200 (Pine Brook, New Jersey) moisture balance. The results were expressed as percentage (g/100g). The pH was measured by a pH meter (Hanna instruments, Romania). A total soluble solid (TSS) was analyzed by a portable refractometer (Abbe Superscientific 300003, Tokio, Japan). For determining the bulk density 20 g of powder was weighed into a 100 mL graduated cylinder. The cylinder with sample in it was dropped 10 times onto a rubber mat from a height of 15 cm. The density was then calculated as the ratio of mass and volume. The average time of wettability was taken as the time required to wet 10 g powder when dropped onto the surface of 100 mL water at 25ºC placed in a 17.5 cm glass dish [14]. Powder solubility was determined according to Eastman and Moore [15], modified by Cano-Chauca [16], 1 g of powder was placed into 100 mL of distilled water and shaked manually until complete dissolution. The sample was then transferred to Eppendrof vials and centrifuged (Hettich EBA 12, Tuttlingen, Germany) for 5 min at 5260 rpm. A 25 mL sample was taken from the supernatant and placed in Petri dishes before drying in an oven at 105ºC for 5 h. The solubility was calculated by difference in weight. The powder hygroscopicity was determined according to the method proposed by Al-Kahtani and Hassan [17]. About 5 g of powder was spread evenly on petri dishes (9 cm dia.) to achieve a high surface area between humid air and powder. Samples in the dishes were placed in an environmental chamber (Sanyo Gallenkamp PLC Model HCC 031 CF1.F, Leicester, UK) maintained at 21ºC and 76% RH. The gain in weight of the samples due to moisture absorption was recorded at 15 min intervals. The color parameters were determined using a Color Flex Hunter Lab 45-0, Viginia, EUA. Total betalains content was determined according to the method reported by Castellano-Santiago and Yahia [18] and measurements were performed in triplicate on a spectrometer DR 5000 HACH (Colorado, USA) at 535 nm (betacyanins) and 483 nm (betaxanthins). Betacyanin (BC) or betaxanthin (BX) was calculated as follows:

$$BC,BX[mg/g]=[A(DF)(Mw)Vd/\epsilon LWd)]$$

Where A is the absorption value at the absorption maximum of 535 nm (betacyanins) and 483 (betaxanthins), DF is the dilution factor, Vd is the dried sample solution volume (mL), Wd is the dried sample weight (g), L is the path-length of the cuvette (1 cm), MW is the molecular weight for betacyanin (550 g/mol) and betaxhantin (380 g/mol) and $\epsilon$ is the molar extinction coefficient for betacyanins (60,000 L/mol•cm in water) and betaxanthins (48,000 L/mol•cm in water).

### Statistical analysis

The analyses were carried out in triplicate and the results are presented as mean values with standard deviations. The mean values were analyzed by ANOVA procedure with SATISTICA version 7.0 (2004).

## RESULTS AND DISCUSSION

### Analysis of beetroot-orange juice

The composition of beetroot-orange juice studied in this work is given in **Table 1**. It can be observed that the concentration of total pigments is similar to the one reported by Azeredo [9], when carried out the extraction of betacyanin at pH 3.6, but is lower than the amount found by Pitalua [10], who worked at pH 6.5. In both cases, the authors worked with pure beetroot juice. Therefore, the less amount found in this work is in part due to the addition of the orange juice.

**Table 1.** Beetroot-orange juice (60/40) composition.

| Analytical properties | Values |
|---|---|
| **Total solids** | 11.77% |
| **Soluble solids** | 10.58°Bx |
| **pH** | 4.6 |
| **Total pigments** | 10.3 mg/g |

## Physical properties of the powders produced

### Moisture content

Results of the physical properties of the powders are presented in Tables 2-4. The moisture content for the powders produced ranged between 3.5 and 5.6 %. As the amount of coating material was increased, the moisture content of samples tended to decrease independently of the feed flow rate used. This can be explained by the fact that the addition of microencapsulating material leads to higher amount of solids in the feed and less water to be evaporated [19]. Similar results have been found by several authors [20-21] who have reported lower moisture content by increasing the amount of maltodextrin in spray drying of concentrated raisin, watermelon and pomegranate juice. This might happen because the coating material formed a barrier that reduced transfer of moisture from juice droplet. As expected, it also was observed that as the temperature was increased, the moisture content decreased.

**Table 2.** Physical properties of the microencapsulated powders at 130°C.

| Maltodextrin | Moisture | Bulk density | Hygroscopicity (g water/ kg dry solids/min) | Wettability | Solubility |
|---|---|---|---|---|---|
| (%) | (g/100g) | (g/cm$^3$) | | (s) | (%) |
| 8 mL/min | | | | | |
| 3 | 5.0 ± 0.57a | 0.724 ± 0.01a | 0.615 ± 0.02a | 149.3 ± 1.26a | 86.5 ± 0.12a |
| 5 | 5.0 ± 0.10a | 0.701 ± 0.0b | 0.579 ± 0.01a | 154.4 ± 12.6a | 86.0 ± 0.00a |
| 7 | 4.7 ± 0.30b | 0.600 ± 0.0c | 0.526 ± 0.01b | 176.2 ± 10.3b | 90.4 ± 0.18bc |
| 10 mL/min | | | | | |
| 3 | 5.6 ± 0.17a | 0.725 ± 0.0a,b | 0.618 ± 0.06a | 109.4 ± 14.21a | 76.37 ± 0.25d |
| 5 | 5.4 ± 0.17a,b | 0.702 ± 0.01a | 0.624 ± 0.03a,b | 110.8 ± 16.0a | 84.81 ± 0.24b |
| 7 | 4.1 ± 0.05c,d | 0.632 ± 0.0c,d | 0.603 ± 0.08b | 133.46 ± 4.70b | 91.84 ± 3.7c |

The values in the same column followed by different superscripts were significantly different ($p<0.05$)

**Table 3.** Physical properties of the microencapsulated powders at 140°C.

| Maltodextrin | Moisture | Bulk density | Hygroscopicity (g water/kg dry solids/min) | Wettability | Solubility |
|---|---|---|---|---|---|
| (%) | (g/100g) | (g/cm$^3$) | | (s) | (%) |
| 8 mL/min | | | | | |
| 3 | 4.6 ± 0.11b | 0.725 ± 0.0a | 0.429 ± 0.02c | 155.8 ± 13.2a | 88.7 ± 0.81ab |
| 5 | 4.5 ± 0.36b | 0.666 ± 0.0a,e | 0.362 ± 0.00d | 160.9 ± 15.01a | 90.7 ± 3.23bc |
| 7 | 3.5 ± 0.73c | 0.604 ± 0.0e | 0.330 ± 0.02d,e | 185.6 ± 8.24.b | 90.8 ± 0.04bc |
| 10 mL/min | | | | | |
| 3 | 5.0 ± 0.23b,c | 0.712 ± 0.0b | 0.639 ± 0.03a | 118.9 ± 5.7a | 76.5 ± 0.50d |
| 5 | 4.7 ± 0.20b,c | 0.711 ± 0.0b | 0.590 ± 0.01a,b | 120.3 ± 4.03a | 89.98 ± 1.15b |
| 7 | 4.6 ± 0.13c,d | 0.612 ± 0.0c | 0.570 ± 0.02b | 142.9 ± 3.81b | 96.45 ± 0.03c |

The values in the same column followed by different superscripts were significantly different ($p<0.05$)

### Bulk density

The bulk density in powders is an important parameter for packaging and storage. Lower bulk density implies greater volume for packaging and reduces the shelf life because as the more occluded air exist the greater is the possibility for oxidation. Values for the bulk density property can be observed in **Tables 2-4**. From these Tables, is possible to see that increasing the amount of coating material the value of density diminished. This could be related to the moisture content of samples, that particles with less moisture content are lighter. This effect was reported by Chegini and Ghobadian [8], they found heavier particles at higher moisture content because the water is heavier than the dry solid. Ferrari [22] who observed higher bulk density at higher moisture content in spray dried blackberry powders, pointed out that heavier material accommodates easier into the spaces between the particles, resulting in higher bulk density values. They reported values from 0.409 to 0.443 g/cm3 lower than in this work. It has been reported that bulk density increases while increasing feed flow rate and decrease with increasing inlet air temperature [23].

In this study, bulk density increased with increased feed flow rate. In general, it was found, that the bulk density decreased when the temperature was higher, similar behavior was reported by Cai and Corke [24].

**Table 4.** Physical properties of the microencapsulated powders at 150°C.

| Maltodextrin | Moisture | Bulk density | Hygroscopicity (g water/kg dry solids/min) | Wettability | Solubility |
|---|---|---|---|---|---|
| (%) | (g/100g) | (g/cm³) | | (s) | (%) |
| 8 mL/min | | | | | |
| 3 | 4.3 ± 0.50b | 0.682 ± 0.0d | 0.365 ± 0.03d | 162.83 ± 13.8a | 92.18 ± 1.93c |
| 5 | 3.8 ± 0.20d | 0.643 ± 0.0c | 0.351 ± 0.11d | 179.2 ± 6.3a | 94.5 ± 0.00ce |
| 7 | 3.4 ± 0.20c | 0.587 ± 0.0f | 0.323 ± 0.02d | 192.7 ± 11.8c | 94.7 ± 0.45ce |
| 10 mL/min | | | | | |
| 3 | 4.5 ± 0.23d | 0.711 ± 0.0a | 0.580 ± 0.03c | 122.7 ± 5.6a | 78.41 ± 0.04a |
| 5 | 4.0 ± 0.11e | 0.661 ± 0.0c | 0.581 ± 0.01c | 124.1 ± 7.0a | 96.4 ± 0.2e |
| 7 | 3.8 ± 0.60e | 0.591 ± 0.0d | 0.560 ± 0.07c | 192.59 ± 11.5c | 98.21 ± 0.22e |

### Hygroscopicity

The addition of coating material as drying aid in fruit juices is used in order to improve the flowability and reduce the hygroscopicity because the adsorbed water increases the liquid bridges and capillary forces acting on the particles, Fitzpatrick [25]. In this investigation, hygroscopicity values varied from 0.330 to 0.639 (g water/Kg dry solids/min). From Tables 2-4, it can be observed that increasing the amount of MD the hygroscopicity was reduced, this behavior was also observed by Osman and Endut [19], who investigated on spray drying of roselle-pineapple juice. In comparison with other authors our values are high. Vardin and Yasar [11], worked with spray dried pomegranate juice and found values that varied from 0.132 to 0.249 g $H_2O$ per Kg dry solids min-1. Araujo [26] reported hygroscopicity values in powder from anacardo juice in the range of 0.38 to 0.48 (g water/Kg powder/min), they found lower values when the anacardo gum was used instead of maltodextrin, pointed out that the behavior of anacardo gum is similar to the arabic gum. Papadakis reported values for hygroscopicity of concentrated raisin juice between 0.14 and 0.22 (g water/Kg powder/min) using maltodextrin DE 21 and DE 12. These differences compared to our research could be due to the nature of the raw material, the amount and kind of simple sugars in each product, the interaction of the carrier agent with sugars and the degree of hydrolysis of the MD used in each case. All the investigations carried out on spray dried fruit powders have reported a positive effect on hygroscopicity and as a consequence avoiding caking and improving rehydration properties.

### Wettability

Wettability is a functional property of powders and represents the ability of a particle powder to adsorb water on its surface. The values of wettability are presented in **Tables 2-4**. It was observed that as the concentration of MD increased the time of wettability also increased. Probably because of the functional properties of the MD, this has lower dissolution capacity than low molecular sugars present in the beetroot powder. Powders with higher moisture content presented less time of wettability, this is because the moisture tend to form agglomerations which help to the reconstitution process [27]. Ferrari [22] established significant differences in wettability values when used 7% of MD and 7% of Gum Arabic as encapsulating agents, they found 82.20 and 134.20 s respectively. It is well known the good solubility capacity of maltodextrin compared to Arabic gum. While observing the effect of feed flow rate on the wettability time, it was found that at lower feed flow rate the wettability time was higher and the moisture content and bulk density was lower. This corroborates the statement that in the absence of non-agglomerated particles, the starting of reconstitution process takes more time. Gong [28] reported a value of 120 s for bayberry juice powder, using DE 12 maltodextrin and inlet air temperature 150-155 °C, when the powder was subjected to an agglomeration process, the value of 120 s was reduced to only 15 s, showing with this how important is the agglomeration for reconstitution properties in powders.

### Solubility

Solubility is one of the main attributes in powder products. In this investigation this parameter was found between 76.5 and 98.21%. It was observed that the solubility improved when the amount of MD was increased, mainly with feed flow rate of 10 mL/min. Similar results have been reported by several authors. Abadio [29] found that when the amount of Maltodextrin was increased the solubility in pineapple juice powder also increased. Cano-Chauca [16] reported that when maltodextrin was used, the solubility significantly was increased, however when Arabic gum was used as encapsulating material the solubility of mango juice powder decreased. The maltodextrins are some of the main materials used in spray drying of fruit juices, due to its functional properties as the high solubility in water.

### Color

**Tables 5 and 6** presents the values for parameters L, a*, b* and ΔE in beetroot-orange juice powders. It can be observed that when the feed flow rate was lower the parameter L* was higher than the control in every case. The addition of encapsulating material provoked higher values of lightness, this was expected, because the color and quantity of maltodextrin influenced the final color of the powder. Kha [23] reported lower lightness in powder from pitaya obtained by spray dryer when the amount of maltodextrin was increased, they argued that the white color of the maltodextrin contributed to higher values of lightness. Sousa [30], found high L* values in tomate powder obtained at high temperaturas, indicating that the dark color of the powder was lost due to oxidation of pigments. On the contrary, Quek [21], observed low value of lightness in watermelon powder, they explained that the sugars in the watermelons undergo darkness reactions as the inlet temperature was increased. The value of the parameter a* was increasing in all the treatments, this indicated a change of color from purple to red-orange. This also was reported by Herbach [4], who said that the thermal treatment provoked degradation products originating red-orange betanins, this enhance the increase in the parameter a*.

**Table 5.** Results for color parameters in powders obtained with feed flow rate of 8 mL/min.

| Sample | L* | a* | b* | ∆ E |
|---|---|---|---|---|
| Control | 32.23 ± 0.38 | 35.53 ± 0.10 | 4.11 ± 0.22 | |
| 130°C, 3% | 33.79 ± 0.15a | 40.85 ± 0.12a | 5.17 ± 0.14a | 7.41 |
| 130°C, 5% | 41.91 ± 0.22b | 44.55 ± 0.23b | 0.41 ± 0.10b | 11.08 |
| 130°C, 7% | 40.75 ± 0.03c, e | 42.34 ± 0.07c | 1.91 ± 0.05 c | 10.13 |
| 140°C, 3% | 35.17 ± 0.46d | 43.39± 0.04d, g | 6.96 ± 0.06d | 7.9 |
| 140°C, 5% | 40.09 ± 0.36 c,e | 41.15 ± 0.03e | 1.90 ± 0.08c | 9.96 |
| 140°C, 7% | 40.97± 0.11c | 43.29 ± 0.02 f | 1.50 ± 0.07e | 11.26 |
| 150°C, 3% | 33.09 ± 0.08f | 47.65 ± 0.13f | 0.81 ± 0.05f | 12.59 |
| 150°C, 5% | 39.13 ± 0.15g | 40.82 ± 0.06h | 1.87 ± 0.04c | 8.97 |
| 150°C, 7% | 39.15 ± 0.63g | 43.67 ± 0.43g | 1.17 ± 0.05g | 11.05 |

The values in the same column followed by different superscripts were significantly different ($p<0.05$)

**Table 6.** Results for color parameters in powders obtained with feed flow rate of 10 mL/min.

| Sample | L* | a* | b* | ∆ E |
|---|---|---|---|---|
| Control | 32.23 ± 0.38 | 35.53 ± 0.10 | 4.11 ± 0.16 | |
| 130°C, 3% | 39.08 ± 0.09a | 40.85 ± 0.126a | 2.68 ± 0.14a | 8.76 |
| 130°C, 5% | 32.72 ± 1.45b | 44.55 ± 0.23b | 1.27 ± 0.31b, e | 9.24 |
| 130°C, 7% | 46.95 ± 0.12c | 42.34 ± 0.07c | 3.37 ± 0.16c | 16.23 |
| 140°C, 3% | 35.52 ± 0.02d | 45.32 ± 0.00d | 4.24 ± 0.00d | 4.53 |
| 140°C, 5% | 36.64 ± 0.02e | 46.23 ± 0.06e | 0.87 ± 0.07b | 11.71 |
| 140°C, 7% | 42.33± 0.04f | 40.34 ± 0.26 f | 1.56 ± 0.13e | 11.46 |
| 150°C, 3% | 36.8 ± 0.02b | 44.16 ± 0.04b | 3.39 ± 0.086c | 9.76 |
| 150°C, 5% | 38.54 ± 0.01g | 47.00 ± 0.01g | 0.17 ± 0.08f | 13.66 |
| 150°C, 7% | 28.67 ± 0.40h | 43.32 ± 0.21i | 1.21 ± 0.18b, e | 9.05 |

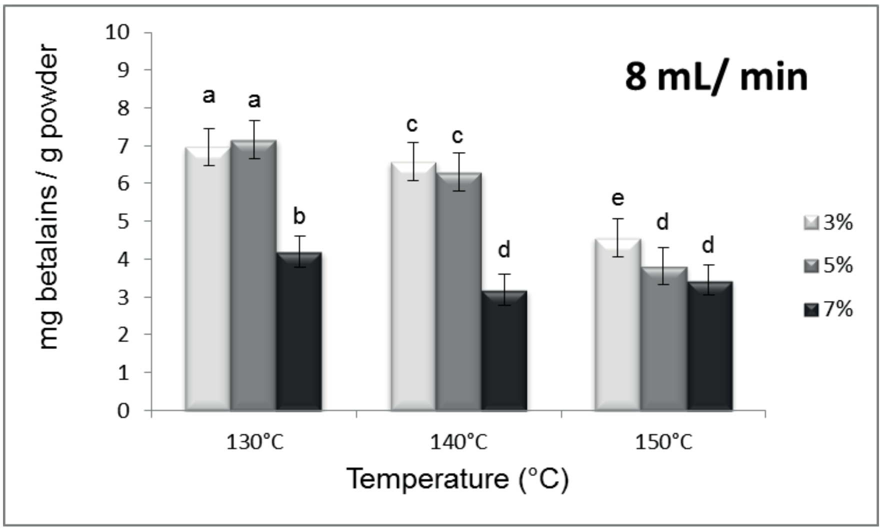
The values in the same column followed by different superscripts were significantly different ($p<0.05$)

**Betalains content**

With regard to the concentration of betalains (**Figure 1**), it was observed retention of 85.4% in the powder (8.8 mg/g) compared with the fresh beetroot-orange juice (10.3 mg/g), under the conditions of flow rate of 10 mL/min, 3% of maltodextrin and 140°C. From **Figure 1**, it can be observed that the concentration of total betalains is lower as the concentration of encapsulating material is increased, this could be due to a dilution of the betalains. Azeredo [9] and Pitalua [10] have reported a retention of 90% and 80% respectively of betalains in spray dried beetroot juice powder. In this research work, the betalain content was higher than that found by Azeredo who reported a value of 6.19 mg/g in the fresh extract of beetroot juice. In our study, it seems that 3% of maltodextrin is enough to preserve the higher amount of betalains when the flow rate was 10 mL/min. However, the parameters such as moisture content, bulk density and hygroscopicity of powders showed lower values at feed flow rate of 8 ml/min, than powders obtained at 10 mL/min, which infers longer shelf life. Additionally, these powders (obtained with 8mL/min) showed better sensorial properties like color, flavor and general acceptance (data not shown).

## CONCLUSION

The properties of beetroot powder obtained under different conditions of spray drying were established. From the results, it can be concluded that the conditions for obtaining the better preservation for total betalains were in general at the lower temperature (130°C) using 3 and 5% of maltodextrin DE10. However, these conditions produced a powder with poor functional properties. A as result it was necessary to select conditions were the powder had good functional properties and good content of total betalains. These conditions were: Inlet air temperature,140ºC; feed flow rate, 8 mL/min and 5% of maltodextrin.



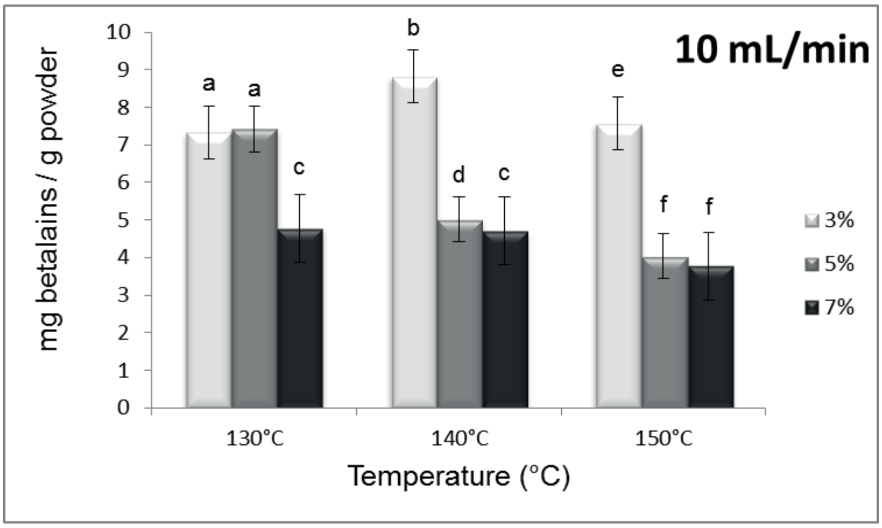




**Figure 1.** Betalains content in microencapsulated powders under different processing conditions. Different letter indicate significant differences ($p < 0.05$).

## ACKNOWLEDGEMENT

The author Vázquez-Juárez had a scholarship for her MSc studies from the National Council of Science and Technology (CONACYT), México.

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