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Effect of Spray Drying on the Microencapsulation of Blueberry Natural Antioxidants †

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Abstract: Phenolic compounds obtained from blueberries have gained great attention due to their more effective bioactive roles in human health than those of whole berries. However, they are sensitive to environmental conditions and are therefore susceptible to degradation affecting their effectiveness. The microencapsulation of these compounds by spray drying provides a solution to these problems. This work aimed to study the effect of spray drying on the microencapsulation of the blueberry phenolic compounds to optimize the production of a powder rich in stable polyphenols. The phenolic extract from blueberries was spray dried under different conditions of inlet air temperatures (140 and 160 °C) and encapsulating agent concentrations (20 and 30% *w*/*v*), using maltodextrin (14.7 dextrose equivalent). The drying yield, moisture content, water-solubility, total and surface phenolic content, and encapsulation efficiency of total phenolic were investigated. The results obtained showed that the different conditions evaluated influenced the drying yield, moisture content, surface phenolic content, and encapsulation efficiency of phenolic compounds. In this sense, the powders with the best characteristics were obtained with 30% *w*/*v* of maltodextrin at 160 °C inlet temperature. These powders, rich in blueberry polyphenols stabilized by microencapsulation, are easier to handle for application, so they could be used as functional food ingredients.

Keywords: blueberry; polyphenols; spray drying; microencapsulation

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1. Introduction

Consumption and the health-promoting properties of blueberry fruits have attracted considerable attention after a report was published about its their antioxidant activity among 42 fruits and vegetables [1]. Blueberries were found to be a rich source of bioactive compounds such as polyphenols, compounds with proven beneficial effects on human health. Several studies have shown that phenolic extracts from blueberry fruits were found to be more effective in their bioactive roles in human health than whole berries [2–4]. However, polyphenols are chemically labile compounds, and their stability, and therefore effectiveness, may be affected by several factors, such as pH, storage temperature, light, oxygen, solvents, metal ions, etc. Thus, the stabilization of these molecules is the principal focus in recent studies because of their beneficial effects on health.

Microencapsulation is an option that stabilizes polyphenols and controls their release at potential absorption sites [5]. Among microencapsulation techniques, spray drying is a cost-effective and easy operation method that results in the formation of a stable, free-flowing powder. Structurally, the bioactives are trapped in encapsulating agents of polysaccharide or proteinaceous nature. Among the polymers used for polyphenols, maltodextrin is one of the most important polysaccharides, mainly because it forms low viscosity solutions in high concentrations, an important characteristic in the spray drying process [6,7]. Maltodextrin has the additional advantages of a low cost and mild taste [8].

Drying conditions, such as inlet air temperature and concentration of the encapsulating agent, among others, may influence the final characteristics and the production yield of the obtained powder. Therefore, the aim of this work was to evaluate the effect of the drying conditions (inlet air temperatures and maltodextrin concentration) on the properties of the powder obtained from a blueberry phenolic extract in order to optimize the production of a powder rich in stable bioactive polyphenols.

2. Materials and Methods

2.1. Samples

Blueberry fruits were obtained from "Tierra de Arándanos" S.R.L, located in San Miguel de Tucumán, Province of Tucumán, Argentina.

2.2. Obtaining the Phenolic Extract from Blueberries

The whole blueberry fruits, previously selected, destemmed, washed with distilled water, and dried with blotting paper, were crushed in a blender. Their phenolic compounds were extracted with 52.6% (*v*/*v*) ethanol solution (acidified with citric acid at pH 2.0) at a mass solvent to crushed fruits ratio of 4:2. Extraction was performed at room temperature (25 °C) during 5 h using orbital agitation at 200 rpm and protected from light. The mixture was filtered (Whatman N°4) and the supernatant was collected. Then, the ethanol from the extract was eliminated using a rotary evaporator (Büchi, Flawil, Switzerland) at 40 °C. Finally, the extract was reconstituted to its initial volume with distilled water. The extracts were fractionated and stored at −20 °C until spray drying.

2.3. Spray Drying of the Blueberry Phenolic Extracts

The mixture extract-maltodextrin (14.7 dextrose equivalent) was protected from light and homogenized using a magnetic stirrer until the complete dissolution of maltodextrin. Spray drying was carried out in a Buchi mini spray dryer model B-290 (Flawil, Switzerland) using an air flow of 600 L/h. The mixture was fed into the drying chamber with a peristaltic pump at a flow rate of 7.5 mL/min and an aspirator flow rate of 100% (maximum capacity). The variables tested were inlet air temperature, or drying air temperature, (140 and 160 °C), and concentration of the encapsulating agent in the mixture (20 and 30% *w*/*v*). All spray drying experiments were performed in triplicate. Obtained powders were vacuum packed in bags and stored at −20 °C until further analysis.

2.4. Drying Yield or Powder Production Yield

Yield was expressed as the percentage of the mass recovered after drying in relation to the total solids before drying.

2.5. Moisture Content

The percent moisture content of the powders was determined in a halogen moisture analyzer (HB43-S Mettler Toledo).

2.6. Water Solubility

This parameter was determined according to Paini et al., 2015 [9].

2.7. Estimation of Phenolic Compounds in Powders

2.7.1. Phenolic Compounds Extraction

The total phenolic compounds and surface phenolic compounds from the powders were extracted according to Tolun et al., 2016 [10].

2.7.2. Determination of Total Phenolic Content (TPC), Surface Phenolic Content (SPC), and Encapsulation Efficiency of Phenolic Compounds (EE)

The total phenolic content (TPC) and surface phenolic content (SPC) were measured by the Folin–Ciocalteu method according to Tolun et al., 2016 [10]. Results were expressed in mg of gallic acid equivalents (GAE) per 1 g of powder. The encapsulation efficiency of phenolic compounds (EE) was calculated as follows:

$$EE = (TPC - SPC)/TPC \times 100. \tag{1}$$

2.8. Statistical Analysis

Results are expressed as mean ± SD and were analyzed using the Infostat software package [11]. Analysis of Variance (ANOVA) was performed and in the case of significance ($p < 0.05$), a DGC [12] comparison test was performed to reveal paired differences between means.

3. Results and Discussion

3.1. Drying Yield or Powder Production Yield

The drying yield, or powder production yield, under different process conditions is shown in Figure 1. Conditions that result in sufficient drying of the particles enhance the product yield. In our study, all spray drying conditions exceeded the satisfactory level of 50% for a laboratory scale spray dryer [13], with results ranging from 81.3% to 93.4%. We observed significant differences among treatments. In this sense, results showed that, with reduced inlet air temperature (140 °C) and less concentration of the encapsulating agent (20%), there was a decrease in the drying yield or powder production yield; and therefore, the conditions with higher powder production yield were 30%MD 160 °C, 30%MD 140 °C, and 20%MD 160 °C, all of them showing yields higher than 90%.

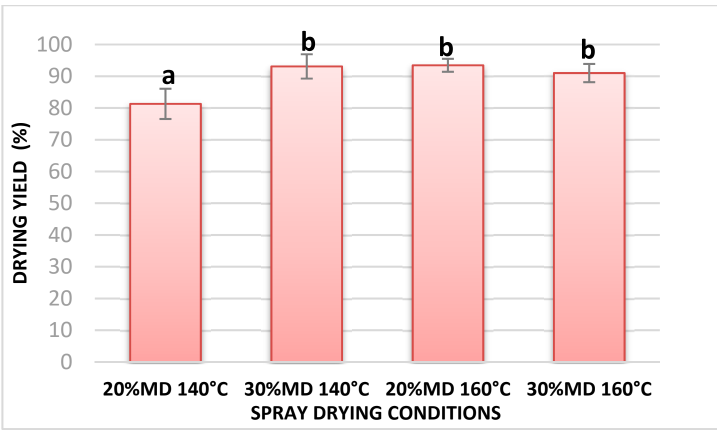

Figure 1. Drying yield (%) under different spray drying conditions. Different letters indicate statistically significant differences ($p < 0.05$) among conditions.

3.2. Moisture Content and Water Solubility of Powders

The moisture content in powders under different process conditions are shown in Figure 2. Spray drying must be sufficiently efficient to yield a powder with a residual moisture content below 4–5%, which is required to ensure powder stability. Our results showed that the moisture content in powders ranged from 3.4% to 4.6% among the spray drying conditions evaluated, with the 30%MD 160 °C treatment showing the lowest value with respect to all the others. According to our results, a decreased moisture content was observed with the increase in drying air temperature due to the high temperatures, leading to greater energy transferred to the mixture and the evaporation of greater amounts of water. This variable, together with an increase in MD concentration, gave the lowest moisture (30%MD 160 °C). The fact that the increase in MD concentration is accompanied by an increase in the solid matter in the feed solution and a decrease in the moisture to be evaporated may result in a decrease in the moisture content of the end powder. Our results are consistent with Tolun et al., 2016. [10].

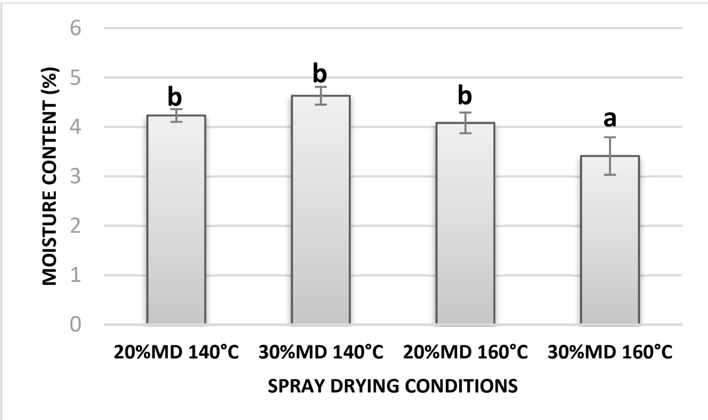

Figure 2. Moisture content (%) under different spray drying conditions. Different letters indicate statistically significant differences ($p < 0.05$) among conditions.

Regarding the water solubility of the powders, at lower values there is minor degradation of the powder constituents, and such condition leads to fewer numbers of soluble molecules, so they are desirable high values for this parameter. In our study, the obtained values were higher than 98% without differences among treatments (data not shown). The presence of maltodextrin increases the solubility of atomized samples due to this encapsulating agent being highly soluble in water, making it one of the main materials used in spray drying. Our results are consistent with de Souza et al., 2015 [14].

3.3. Total Phenolic Content (TPC), Surface Phenolic Content (SPC), and Encapsulation Efficiency of Phenolic Compounds (EE)

The total phenolic content (TPC), surface phenolic content (SPC), and encapsulation efficiency of phenolic compounds (EE) in powders under different process conditions are shown in Figure 3. The TPC in powders ranged from 1.17 to 1.70 mg GAE/g powder according to Fang et al., 2011 [15]. According to our results, it was found that the inlet air temperature and encapsulating agent concentration do not have an effect on TPC ($p < 0.05$). However, we observed significant differences in the SPC. In this sense, we observed the lower SPC at higher inlet air temperature and higher encapsulating agent concentration (30%MD 160 °C). SPC has a direct relationship with the EE of the microcapsules produced. The EE ranged from 90.6% to 97.8% with significant differences among treatments. The higher values were obtained for 20%MD 140 °C and 30%MD 160 °C.

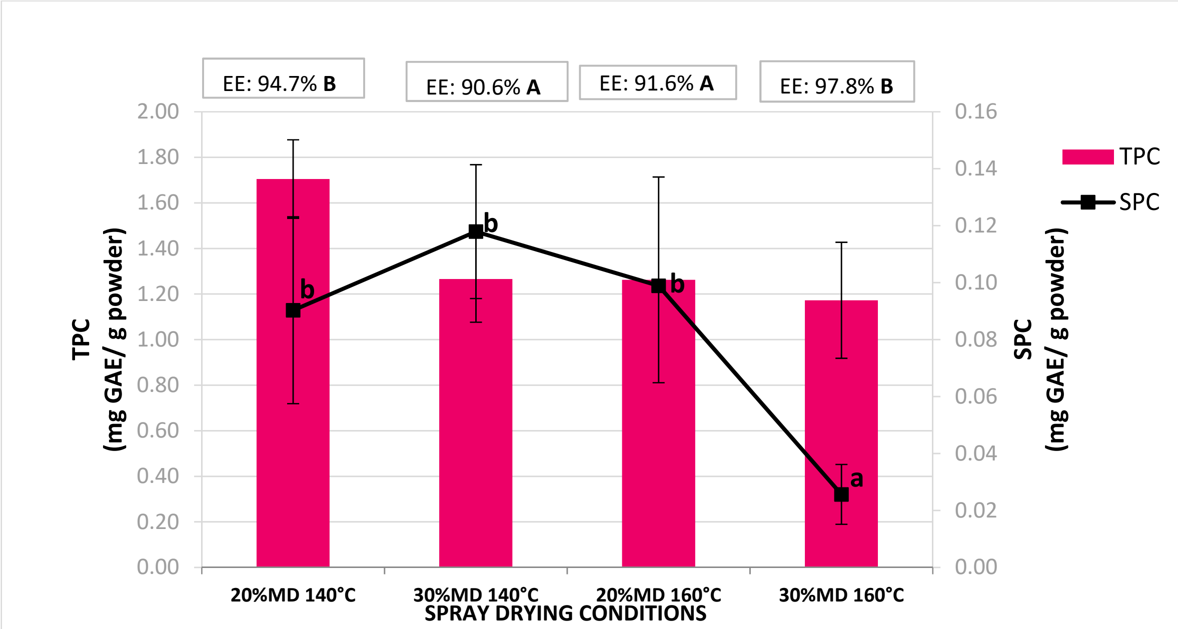

Figure 3. Total phenolic content (TPC), surface phenolic content (SPC), and encapsulation efficiency of phenolic compounds (EE) under different spray drying conditions. Different letters indicate statistically significant differences ($p < 0.05$) among conditions: lower case for SPC and upper case for EE.

4. Conclusions

The drying air temperature and the maltodextrin concentration as encapsulating agent influenced the drying yield, moisture content, surface phenolic content, and encapsulation efficiency of phenolic compounds from blueberry extracts. In this sense, the powders with the best characteristics were obtained with 30% *w*/*v* of maltodextrin at 160 °C inlet temperature. The powders rich in blueberry polyphenols stabilized by microencapsulation produced by utilizing these optimum conditions have the potential to be used as functional food ingredients.

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Conflicts of Interest: The authors declare no conflict of interest.

References

1. Lee, J.; Wrolstad, R.E. Extraction of anthocyanins and polyphenolics from blueberry processing waste. *J. Food Sci.* **2004**, *69*, C564–C573.
2. Prior, R.L.; Wilkes, S.E.; Rogers, T.R.; Khanal, R.C.; Wu, X.; Howard, L.R. Purified blueberry anthocyanins and blueberry juice alter development of obesity in mice fed an obesogenic high-fat diet. *J. Agric. Food Chem.* **2010**, *58*, 3970–3976.

3. Prior, R.L.; Wu, X.L.; Gu, L.W.; Hager, T.J.; Hager, A.; Howard, L.R. Whole berries versus berry anthocyanins: Interactions with dietary fat levels in the C57BL/6J mouse model of obesity. *J. Agric. Food Chem*. **2008**, *56*, 647–653.
4. Prior, R.L.; Wu, X.L.; Gu, L.W.; Hager, T.; Hager, A.; Wilkes, S.; Howard, L. Purified berry anthocyanins but not whole berries normalize lipid parameters in mice fed an obesogenic high fat diet. *Mol. Nut. Food Res.* **2009**, *53*, 1406–1418.
5. Betz, M.; Kulozik, U. Whey protein gels for the entrapment of bioactive anthocyanins from bilberry extract. *Int. Dairy J*. **2011**, *21*, 703–710.
6. Berg, S.; Bretz, M.; Hubbermann, E.M.; Schwarz, K. Influence of different pectins on powder characteristics of microencapsulated anthocyanin and their impact on drug retention of shellac coated granulate. *J. Food Eng*. **2012**, *108*, 158–165.
7. Tonon, R.V.; Brabet, C.; Hubinger, M.D. Anthocyanin stability and antioxidant activity of spray-dried açai (Euterpe oleracea Mart.) juice produced with different carrier agents. *Food Res. Int.* **2010**, *43*, 907–914.
8. Rocha, G.A.; Trindade, M.A.; Netto, F.M.; Favaro-Trindade, C.S. Microcapsules of a casein hydrolysate: Production, characterization, and application in protein bars. *Food Sci. Technol. Int.* **2009**, *15*, 407–413.
9. Paini, M.; Aliakbarian, B.; Casazza, A.A.; Lagazzo, A.; Botter, R.; Perego, P. Microencapsulation of phenolic compounds from olive pomace using spray drying: A study of operative parameters. *LWT-Food Sci. Technol.* **2015**, *62*, 177–186.
10. Tolun, A.; Altintas, Z.; Artik, N. Microencapsulation of grape polyphenols using maltodextrin and gum arabic as two alternative coating materials: Development and characterization. *J. Biotechnol.* **2016**, *239*, 23–33.
11. Di Rienzo, J.A.; Casanoves, F.; Balzarini, M.G.; González, L.; Tablada, M.; Robledo, C.W. *InfoStat, Versión 2008*; Grupo InfoStat, FCA, Universidad Nacional de Córdoba: Córdoba, Argentina, 2008.
12. Di Rienzo, J.A.; Guzmán, A.W.; Casanoves, F. A Multiple-Comparisons Method Based on the Distribution of the Root Node Distance of a Binary Tree. *J. Agric. Biol. Environ.* **2002**, *7*, 129–142.
13. Selvamuthukumaran, M. *Handbook on Spray Drying Applications for Food Industries*; CRC Press: Boca Raton, FL, USA, 2019.
14. de Souza, V.B.; Thomazini, M.; de Carvalho Balieiro, J.C.; Fávaro-Trindade, C.S. Effect of spray drying on the physicochemical properties and color stability of the powdered pigment obtained from vinification byproducts of the Bordo grape (Vitis labrusca). *Food Bioprod. Process* **2015**, *93*, 39–50.
15. Fang, Z.; Bhandari, B. Effect of spray drying and storage on the stability of bayberry polyphenols. *Food Chem.* **2011**, *129*, 1139–1147.