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# Comparison of blueberry powder produced via foam-mat freezedrying versus spray-drying: evaluation of foam and powder properties

Sandi Darniadi <sup>a,b</sup>, Peter Ho <sup>a</sup> and Brent S. Murray <sup>a\*</sup>

 <sup>a</sup> Food Colloids and Processing Group, School of Food Science and Nutrition, University of Leeds, Leeds LS2 9JT, England, UK
 <sup>b</sup> Indonesian Centre for Agricultural Postharvest Research and Development (ICAPRD), IAARD-Ministry of Agriculture, Jakarta 12540, Indonesia
 \*Correspondence address: <u>b.s.murray@leeds.ac.uk</u>

# Abstract

BACKGROUND: Blueberry juice powder was developed via foam-mat freeze-drying (FMFD) and spray-drying (SD) via addition of maltodextrin (MD) and whey protein isolate (WPI) at weight ratios of MD/WPI 0.4 to 3.2 (with a fixed solids content of 5 wt.% for FMFD and 10 wt.% for SD). Feed rates of 180 and 360 mL h<sup>-1</sup> were tested in SD. The objective was to evaluate the effect of the drying methods and carrier agents on the physical properties of the corresponding blueberry powders and reconstituted products.

RESULTS: Ratios of MD/WPI = 0.4, 1.0 and 1.6 produced highly stable foams most suitable for FMFD. FMFD gave high yields and low bulk density powders with flake-like particles of large size that were also dark purple with high red values. SD gave low powder recoveries. The powders had higher bulk density and faster rehydration times, consisting of smooth, spherical and smaller particles than in FMFD powders. The SD powders were bright purple but less red than FMFD powders. Solubility was greater than 95% for both FMFD and SD powders.

CONCLUSION: The FMFD method is a feasible method of producing blueberry juice powder and gives products retaining more characteristics of the original juice than SD.

Key words: Foam-mat freeze-drying, foam, spray-drying, blueberry, powder

*Abbreviations*: FMFD: foam-mat freeze-drying, foam-mat freeze-dried; SD: spray drying, spray dried; ACN: anthocyanin; MD: maltodextrin; WPI: whey protein isolate

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#### INTRODUCTION

Berries such as blackberries, blueberries, strawberries, cranberries, and raspberries have received much attention due to their possible positive role in human health and disease prevention.<sup>1,2</sup> Researchers have claimed that their polyphenol content, particularly anthocyanins (ACN), might be responsible for averting various diseases.<sup>2,3</sup> From the published data, we know that the content of ACN in berries varies considerably. According to Wu et al.,<sup>4</sup> the total ACNs of blackberry, blueberry, cranberry, grape, red raspberry and strawberry are 0.25, 0.37, 0.14, 0.03, 0.09 and 0.02 g kg<sup>-1</sup> (of fresh weight), respectively.

Blueberries thus contain a significant amount of ACN compared to similar fruits. According to Routray and Orsat <sup>3</sup>, the ACN content of blueberries varies between 1.43 to 8.22 g kg<sup>-1</sup> (of fresh weight). ACNs are natural pigments in berries from which red, blue and purple colours and have been successfully used as colourant agents in many food products.<sup>5</sup> However, ACNs can easily to deteriorate during blueberry processing.<sup>1</sup>

Fruit juice powders are an interesting form of processed blueberry and have extensive applications in the food industry. These powders are commonly used as reconstituted powders in milk, baby food, cake and other products.<sup>2</sup> The advantages of the powders as opposed to the fresh fruit include longer shelf life, ease of serving, reduced volume or weight and significant reduced cost of transportation.<sup>6</sup>

In order to retain ACNs in the processed blueberry products, a variety of preservation methods have been employed. A common method is spray-drying. Spray-drying is a conventional method extensively used to produce dried food and food ingredients in many industries.<sup>7</sup> To obtain high-quality spray-dried powders, several equipment parameters

and feed solution conditions need to be controlled during the drying process.<sup>7</sup> The physicochemical properties of the final product mainly depend on the feed inlet temperature, air flow rate, feed flow rate, atomiser speed, types of carrier agent and their concentrations.<sup>8</sup> Spray-drying of fruit juice with its high content of low molecular sugars and organic acids is relatively difficult. These conditions result in low values of the glass transition temperature and consequently several problems, such stickiness and caking. <sup>7,9,10</sup> To ease the drying process, it is common to add some carriers to the juice or concentrate. There are many studies on the production of honey powder, watermelon, orange, apple juice and various concentrated fruit juice powders by spray-drying with whey protein, maltodextrin and gum arabic as carriers.<sup>10–13</sup> Another issue regarding the spray-drying of fruit juice, particularly blueberries, is that the utilisation of relatively high drying temperatures (> 100 °C) could increase degradation of ACN.<sup>2</sup>

A novel drying process, known as a foam-mat freeze-drying includes four primary steps: (a) adding foaming agents/foam stabilisers to a solution followed by whipping to form a stiff foam, (b) freezing a thin layer of foamed liquid, (c) freeze-drying the foam to obtain a dried cake or a dried layer, and (d) grinding the dried cake to obtain free flowing powder.<sup>14,15,16,17</sup> The advantages of the foam are faster drying times and therefore potentially greater retention of heat-labile constituents.<sup>18,19</sup> Protein is commonly needed to generate foam, typically whey protein.<sup>20</sup> Whey protein is a mixture of globular proteins separated out from the caseins during cheese manufacture, typically isolated as a spray dried powder. The principal surface active ingredient (i.e., foam-stabilizing agent) it contains is  $\beta$ -lactoglobulin.<sup>21</sup>. To further stabilise the foam, a thickener is also frequently added, to increase the viscosity. Polysaccharides such as maltodextrin, gum arabic (GA) and CMC are common compounds used.<sup>22</sup> Maltodextrins are commonly used in foods as bulking agents, texture modifiers, fat replacer, the formulation of fruit leathers, and for the encapsulation of wide range of products via various drying methods, where the material desired becomes trapped in the glassy maltodextrin matrix on drying.<sup>23,24</sup> It should be noted that when mixtures of low molecular weight sugars and high molecular weight surface active compounds such as proteins are spray dried, there is a natural tendency for the surface active components to end up more concentrated on the outside of the dried particles, i.e., components segregate to some extent in the particles.<sup>25,26</sup> This can affect the rehydration properties of the powders but also help overcome the stickiness of dried products with high sugar content.<sup>27</sup> Foam-mat freeze-drying has been developed to obtain the apple powder,<sup>14</sup> where the drying time was reduced compared to conventional freeze drying and the powder retained almost all the original phytochemicals, sensory attributes and physical properties when reconstituted in water.<sup>15</sup>

To the best of our knowledge, little is known about production of blueberry powder via foaming prior to freeze drying and comparison with spray-drying. Thus, the objectives of this present study were: (1) to develop blueberry foam-mat freeze-dried and spray-dried powders; and (2) compare the effect of the different drying methods and carrier agents on the physical properties of the blueberry powders and reconstituted products.

#### **MATERIALS AND METHODS**

# Materials

Biona concentrated organic blueberry juice were purchased from a local supermarket in Leeds, England. The blueberry juice was labelled as not containing additional water, sugar, additives and preservatives. The juice was stored at 4 <sup>o</sup>C after opening. Maltodextrin (MD) (Sigma Aldrich, USA) 16.5-19.5 dextrose equivalent (DE) (PubChem

CID: 107526) and whey protein isolate (WPI) (Fonterra, NZ) were utilised as foam stabiliser and foaming agent, respectively.

#### Foam-mat freeze-drying (FMFD) of blueberry juice

Foamed blueberry juice was prepared by whipping blueberry juice + matrices (weight ratio of juice to matrices = 95:5) using a Kenwood KM 330 series mixer (Kenwood, UK) in an 8 L stainless beaker, at maximum speed for 5 min and ambient temperature. The total solids fraction for all foams prepared for freeze-drying was fixed at 50 g kg<sup>-1</sup>. Matrices with MD/WPI ratios of 0.4, 1.0, 1.6, 2.3 and 3.2 were examined. 85 g of the foam produced were spread on to a round Teflon-coated pan (diameter = 180 mm, height = 30 mm) for each formulation. The foams were blast frozen using a Valera BF051ET blast freezer (Valera, Italy) at -30 °C for 6 h and freeze-dried using an Alpha 1-4 LD Plus freeze dryer (Christ Martin, Germany) at -55 °C and a pressure 0.04 mbar, for 24 h. The dried layer obtained was then ground for 1 min using a Kenwood CH 180A mini chopper food processor (Kenwood, UK). The blueberry powders produced were stored in the dark in pre-weighed, air-tight containers in a refrigerator at 5 °C for further analysis. The foammat freeze-drying process was conducted in duplicate for each set of conditions.

#### Spray-drying (SD) of blueberry juice

Feed solutions were prepared with a weight ratio of juice to matrices = 9:1. The total solids fraction of all spray-dried solutions was fixed at 100 g kg<sup>-1</sup>. Matrix weight ratios of MD/WPI were prepared at 0.4, 1.0, 1.6, 2.3 and 3.2, i.e., as for FMFD. Spray-drying was performed in a Buchi B-290 mini spray dryer (Buchi Laborthecnik AG, Switzerland). The drying conditions were kept constant for each run with an inlet temperature of 150  $^{\circ}$ C, outlet air temperature of 101  $^{\circ}$ C, aspirator rate 100 % (35 m<sup>3</sup> h<sup>-1</sup>), air pressure 0.41 bar and nozzle tip diameter 1.5 mm. The feed rates used were 180 and 360 mL h<sup>-1</sup> and spray

drying results at these 2 feed rates are hereafter referred to as SD180 and SD360, respectively. After each drying process, the blueberry powders were collected from the cyclone and stored in the dark in pre-weighed, air-tight containers in a refrigerator at 5 <sup>o</sup>C for further analysis. The spray-drying processes were all performed in duplicate for each set of conditions.

#### **Foam Capacity**

Foam capacity was measured according to the method of Sadahira et al.<sup>28</sup>, with slight modifications. A 250 mL measuring cylinder was carefully filled up with the foam. The weight of foam was recorded and then the foam density ( $\rho$ ) and overrun were calculated as follows:

$$\rho (g \ cm^{-3}) = mf / volume \ of foam$$
 Eq. 1

$$Overrun (\%) = 100 (mi-mf) / mf$$
 Eq. 2

Where *mi* is the mass of the initial solution (unwhipped sample) and *mf* is the mass of the whipped sample with the same volume of *mi*.

#### Foam stability: drainage

Foam stability was assessed by measuring foam drainage according to the methods of Raharitsifa et al.<sup>29</sup> with slight modifications. A Buchner funnel was filled to the top with 50 mL of each foam. Liquid drained by gravity from the foam was collected in a 50 mL graduated cylinder. The volume V (mL) of liquid drained was measured directly from the graduated cylinder as a function of time over 120 min.

The product yield was defined as by Shi et al.,<sup>13</sup>. The yield was defined as the ratio of the mass of solid powder obtained at the end of the freeze-drying and spray-drying to the mass of initial substances dried, including MD and WPI, i.e.,

Yield (%) = 
$$100 \times (Solids in powder / Total solids in foam or feed solution)$$
 Eq. 3

#### Moisture content and water activity

The moisture content of FMFD and SD powders was measured via a HB 42-S halogen moisture analyzer (Mettler Toledo, UK) at 105 <sup>o</sup>C. Approximately 1 g of powder was placed in the sample pan. The empty sample pan handler and the heating module were closed. The drying time ranged from 2-5 minutes for each sample. The water activity of the approximately 0.5 g of powders was determined by a Hygrolab C1 water activity meter (Rotronic, UK).

#### **Solubility**

Solubility was determined as described by Ceballos et al.<sup>30</sup> The powder samples (1 g) were gently dispersed with 100 ml of distilled water (30 °C) in a beaker and stirred using a Stuart CB-162 magnetic stirrer (Bibby Scientific Ltd, UK) at 400 rpm. After stirring for 5 min, the dispersions were transferred into a 50 mL Falcon tube and centrifuged at 3,000 rpm using a Universal 320 centrifuge (Sartorius, UK), for 10 min. An aliquot of 25 ml of the supernatant was removed to pre-weighed Petri dish and immediately oven dried at 105 °C for 5 h. The supernatant was decanted and determined for water solubility using the following equation:

*Water solubility (%)* = 100 x (weight of dissolved solids in supernatant/weigh of sample) Eq. 4

#### **Rehydration time**

Rehydration of blueberry powder was determined according to the method described by Islam et al.,<sup>31</sup> with slight modifications. Powder (0.5 g) was added to 50 mL distilled water at 26 <sup>o</sup>C in a 100 mL glass beaker. The mixture was agitated using a Stuart CB-162 magnetic stirrer (Bibby Scientific Ltd, UK) at 900 rpm and the time recorded until all particulate matter was not visible to the naked eye.

#### **Bulk density**

The bulk density of the powders was determined by gently adding 1 g of blueberry powder into an empty 10 mL graduated cylinder and holding the cylinder on a FB 15012 top mix vibrator (Fischer Scientific, UK) at 2,000 rpm for 1 min. The DB was calculated by dividing the mass of the powder by the volume occupied in the cylinder.<sup>13</sup>

#### Particle morphology analysis

The microstructural characteristics of powders were analysed by scanning electron microscopy (SEM) using a Quanta 200 F (FEI, Oregon, USA) microscope. The samples were placed on an aluminium support using a double-sided adhesive tape with conductive carbon and then coated with platinum using a Cressington sputter coater 108 (Cressington Scientific Instruments, UK). The images were taken with the detector within the lens, using an acceleration voltage of  $3.00 \text{ kV}.^{32}$ 

#### **Colour characteristics**

Colour values were determined by the method described by Franceschinis et al.<sup>33</sup> and Mahdavee Khazaei et al.<sup>22</sup> The colour of original blueberry juice, and FMFD and SD reconstituted solutions were examined using a Colour-Eye 7000A (Macbeth, Cheshire,

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UK) with illuminant D65 and  $10^{\circ}$  observer angle. 0.5 of powder was dissolved completely in 50 mL distilled water. The results were expressed as CIE colour values L\*, a\*, b\* where L\* was used to denote brightness, a\* redness and greenness and b\* yellowness and blueness. The indices of:

| Hue angle:                | $\mathrm{H}^{0} = tan^{-1}(b^{*}/a^{*})$   | Eq. 4 |
|---------------------------|--|-------|
| Chroma:                   | $C = a^* + b^*$  | Eq. 5 |
| Total colour differences: | $\Delta \mathbf{E} = \left[ (L_0 - L)^2 + (a_0 + a)^2 + (b_0 - b)^2 \right]^{1/2}$ | Eq. 6 |

: where L<sub>0</sub>, a<sub>0</sub> and b<sub>0</sub> were the L\*, a\* and b\* of the standard and L, a, and b were the corresponding values for the dissolved powder, were calculated and the mean of three replicates are reported.

#### **Statistical Analysis**

The processing treatments were duplicated, and the means of the results were reported. Analysis of variance (ANOVA) was done to establish the presence or absence of significant differences between means. Multiple comparisons were performed using the Tukey test and significance level was set at p < 0.05. All statistical analyses were carried out using Minitab 17.0.

#### **RESULTS AND DISCUSSION**

#### Physicochemical characteristics of blueberry juice prior to drying

The physicochemical properties of blueberry juice were examined prior to dehydration. The blueberry juice had  $89.78 \pm 0.08$  % moisture content,  $10.23 \pm 0.8$  g kg<sup>-1</sup> total soluble solids,  $2.36 \pm 0.01$  pH and  $1.031 \pm 0.0$  g cm<sup>-3</sup> density. The parameter colour of blueberry juice was L\* (brightness) = 0.08, a\* (redness) = 0.45 and b\* (yellowness) = 0.06. The freeze-drying and spray-drying conditions then applied to this juice are summarized in Table 1.

### Foam capacity

Figure 1 shows the effect of carrier agents on the density ( $\rho$ ) and overrun of foamed blueberry. As the MD/WPI ratio was increased the overrun increased significantly, i.e., the volume fraction of air was higher<sup>28,34</sup> and the density was lower. In these experiments the MD/WPI ratio was increased from 0.4 to as high as 9.0, to find the optimum foaming conditions at the fixed overall added solids concentration of 5 wt.%. The overrun was a maximum at the MD/WPI ratio = 1.6 - the overrun was 825% and foam density 0.14 g cm<sup>-3</sup>. Higher additions of MD seemed to produce the opposite effect: beyond MD/WPI = 6 there was a significant decrease in overrun, i.e., decrease in foam stability. This result agrees with Karim and Wai<sup>34</sup> who found that the overrun of foamed starfruit was reduced beyond a methylcellulose concentration of greater than 40 g kg<sup>-1</sup>. Such reductions are undoubtedly due to increased viscosity of the mixture, as well as a corresponding reduction in the concentration of surface active WPI.

Figure 2 shows the volume of liquid drained from the foams over 120 min for the same range of MD/WPI ratios as in Figure 1. The inset shows the result of a cut through these smooth curves at 60 min: volume drained after 60 min is plotted as function of MD concentration [MD]. From [MD] = 1.3 to 3.1 wt.% (MD/WPI ratio 0.4 to 1.6) the drainage volume decreases slightly, indicating greater foam stability, i.e., water holding capacity /WPI ratio, but beyond these concentrations the drainage starts to increase again. This is in agreement with the overrun results: at the higher MD/WPI ratios foam stability decreases again. Although the aqueous phase may be more viscous at higher MD/WPI

ratios, which would be expected to decrease drainage rates, the lower overrun means that the average film thickness between the bubbles will be thicker and the overall film surface area lower, so drainage is faster. For these reasons subsequent FMFD experiments were therefore not conducted for MD/WPI ratios > 3.2.

#### **Powder yield**

Figure 3A shows the yields of blueberry powders produced by FMFD and SD (SD180 and SD360). In general, FMFD yields decreased with higher MD/WPI ratios (up to 3.2) whilst SD yields increased, although FMFD gave statistically higher yields (p < 0.05) than SD at all MD/WPI ratios. Spray-drying involves atomisation and air circulation causing particle loss during the process.<sup>35</sup> The powder particles stick to the drying chamber and cyclone walls. In this study, SD powder was collected from collection vessel only and any particles deposited in drying chamber were discarded.

In case of FMFD, various MD/WPI ratios gave significantly different yields. The ratio 1.6 gave the highest yield. MD/WPI ratios greater than this resulted in a fall of the yield to approximately 72 %. For SD powders, SD180 gave a yield of 61 % but this increased to 67.1 % at MD/WPI = 3.2. Regarding the SD360 process, the lowest yield (62.9 %) was with the MD/WPI ratio 0.4, whilst 3.2 increased this to a maximum of 71 % and 1.0 gave a slight increase to 64.6 %. These results generally agree with those of with Fang and Bhandari <sup>35</sup>.

Utilisation of MD and WPI for freeze drying and spray-drying has been studied in other food products. Shi et al.<sup>13</sup> obtained spray-dried honey powder recoveries > 50 % with addition of MD and WPI, but no powder was recovered when pure honey was spray-dried. Another study revealed that MD + WPI gave excellent HCA (hydroxycitric acid) recovery of microencapsulated *Garcinia* fruit powder, typically 90%.<sup>36</sup>

#### Moisture content and water activity

The final moisture content and water activities of blueberry FMFD and SD powders are shown in Figure 3B and 3C, respectively. All FMFD samples had no significant (p > 0.05) differences in moisture content compared to SD samples of similar composition. However, there were two powders which had the notably highest and lowest moisture content, namely FMFD with MD/WPI = 0.4 and SD360 with MD/WPI = 1.6, respectively. Numerous studies have compared the moisture contents of freeze-dried and spray-dried powders. Generally, they suggest that spray-drying gives a lower moisture content than freeze-drying due to the higher temperature in the spray-drying process. <sup>32,36</sup>

Water activity is a measure of the availability of free water in food that is responsible for biochemical reactions and is an important index to determine microbial stability of food. In this study, the water activity value of all the powders produced was less than 0.40, which is acceptable for such powders in terms of inhibition of microbial growth and biochemical degradation.<sup>13</sup> However, the statistical analysis confirmed that the water activity was significantly (p < 0.05) affected by the drying methods and carrier agents. The highest water activity was the FMFD powder produced with a MD/WPI ratio = 0.4. In contrast, the SD180 powder with the lowest aw was produced with the highest ratio of MD/WPI = 3.2. This powder also had the lowest moisture content.

# Solubility

The solubility of FMFD and SD powders is shown in Figure 4A. Drying methods and carrier agents had no significantly different effects (p > 0.05) on the solubility of the powders: the solubility was > 95% for all powders. For FMFD powder, MD/WPI 1.6 gave highest solubility (99%). These results seem to be consistent with those of Franceschinis et al.<sup>32</sup> who reported no statistical differences in the solubility of freeze-

dried and spray-dried blueberry powders, either made with maltodextrin alone or with trehalose and maltodextrin. The solubility of their freeze-dried powders was in range 99-99.5%, while 98.5-100% was obtained for spray-dried samples.

# **Rehydration Time**

Figure 4B indicates that SD360 powders had significantly (p < 0.05) shorter rehydration times than FMFD and SD180 powders. Rehydration times of FMFD powders were in range 74-78 s, whilst the SD360 powders had rehydration times in range 65-70 s. The rehydration time decreased with increasing MD/WPI ratio, probably due to the high solubility of MD in water <sup>37</sup>, in an agreement with Islam et al.<sup>31</sup>

#### **Bulk Density**

Bulk density is an important property of food powders and spray-dried products. Bulk density targets have to be met to provide consistent weight during packaging, warehousing and transportation. FMFD powders had significantly lower (p < 0.05) bulk densities than SD powders (Figure 4C). Since spray-dried and freeze-dried powders had very similar moisture content (Figure 3B), this suggest a more open and porous structure of the freeze-dried powders (see later). However, this result contrasts significantly with a study conducted by Turan et al.<sup>38</sup> for blueberry powders produced via an ultrasonic spray dryer and freeze dryer. Another study also found that blackberry freeze-dried powder had higher bulk density than spray-dried powder.<sup>32</sup>

#### **Particle Morphology**

The morphological characteristics of FMFD and SD powders are presented in Figure 5 and Figure 6, respectively. In general, the surface morphology of FMFD powders was irregular particles of larger size than those observed for SD powders. Most of FMFD particles resembled broken glass or flake-like structures, probably due to their origin as bubble surface films, broken up via the food processor. The FMFD powders produced with MD/WPI ratios 0.4 (Figure 5 A-B) had similar appearances to those produced with 3.2 ratios (Figure 5 C-D). This flake-like structure is similar to that observed in FD soursop fruit pulp<sup>30</sup> and blackberry.<sup>32</sup> In contrast, the blueberry SD powders appeared as spherical particles, typical of SD powders. Similar behaviour was observed by Tonon et al.<sup>39</sup> for spray-dried Acai juice and Ersus and Yurdagel <sup>40</sup> for black carrot juice. The shape of the particles in SD powders suggested shrunken spheres at low MD (e.g., MD/WPI = 0.4) (Figure 6 A-B), whereas at higher MD (e.g., MD/WPI = 3.2) spheres appeared smooth (Figure 6 C-D). This may be because the higher MD produced a soft and porous crust.

#### **Colour characteristics**

The parameter colour of original blueberry juice was  $L^* = 0.08$ ,  $a^* = 0.45$  and  $b^* = 0.06$ . The brightness, redness and yellowness of original blueberry juice were significantly (p < 0.05) different from both FMFD and SD reconstituted solutions, as shown in Table 2. The L\* of blueberry juice was very dark. In contrast, the L\* of FMFD and SD reconstituted solutions were brighter, which can attributed to the addition of MD, which is a white flour in the dry state. In addition, the SD solutions contained higher matrix proportions (e.g. 100 g kg<sup>-1</sup> dried matter) compared to FMFD solutions (e.g. 50 g kg<sup>-1</sup> dried matter) corresponding to higher L\* value.

Regarding the a\* values, FMFD reconstituted solutions had greater a\* than those from SD180 and SD360 reconstituted solutions (Table 2). The FMFD reconstituted solutions had a deeper red colour compared to SD solutions. In contrast, Franceschinis et al.<sup>32</sup> found

that the redness value was not affected by the drying method. They found that SD blackberry powder produced with MD had the same a\* value as FD powder produced with MD. However, they claimed that utilisation of other types of matrices (e.g., trehalose) had impact on the redness. Trehalose-treated powders had lower a\* than MD-treated powders, using freeze drying and spray-drying processes. In the case of FMFD, increasing MD resulted vary a\* value.

The FMFD reconstituted solutions showed higher b\* (p < 0.05) than SD180 and SD360 reconstituted solutions (Table 2). The SD360 method resulted in the lowest yellowness value ( $b^* = 6.94$ ). In contrast,  $b^* = 26.96$  was obtained from FMFD powder sample. The same behaviour of b\* values was founded for FMFD and SD blackberry powders.<sup>32</sup>

Regarding the colour intensity (Chroma), FMFD reconstituted solutions produced with MD/WPI = 3.2 had significantly (p < 0.05) higher values amongst the FMFD treatments (Table 3). The Chroma value of FMFD powders were also to be found higher according to SD180 and SD360 samples. The higher Chroma values indicate the a\* (redness) and b\* (yellowness) of samples were greater.<sup>33,41</sup> Hue angle (h<sup>0</sup>) of the blueberry juice was 7.05 and this presented the colour in pure red. The FMFD, SD180 and SD360 reconstituted solutions gave h<sup>0</sup> values lower than 24. These values indicate pure red for all samples due to the values lower than 90, which were considered as pure yellow.<sup>41</sup> ANOVA of H<sup>0</sup> showed that FMFD reconstituted solutions produced greater values in comparison with SD180 and SD360 reconstituted solutions.

In order to compare the colour difference ( $\Delta E$ ) obtained between FMFD and SD samples, the powders were reconstituted in water. Higher  $\Delta E$  means more colour changes correspond to changes of L\*, a\* and b\*. Low values of total colour differences ( $\Delta E$ ) are desirable since it is indicative of colour stability of the powder.<sup>38</sup> As seen in Table 3, the re-constituted solution of FMFD with MD/WPI = 1.0 presented higher  $\Delta E$  than SD180 and 360. FMFD solutions produced with MD/WPI = 3.2 showed the highest  $\Delta E$ . With SD180 and 360 powders  $\Delta E$  was significantly changed with increasing MD.

#### CONCLUSIONS

Addition of 50 g of MD + WPI per kg of blueberry juice at ratios MD/WPI = 0.4, 1.0 and 1.6 produced highly stable foamed blueberry solutions. Other ratios (2.3 and 3.2) gave lower stability than 1.6. Blueberry powders were successfully formed via foam-mat freeze-drying (FMFD) and spray-drying (SD) in the presence of MD and WPI. FMFD powders had high yield (> 72 %) with moisture contents = 2.6 to 4.0 %, rehydration times between 74 and 78 s and bulk densities between 0.18 and 0.23 g cm<sup>-3</sup>. The FMFD process also gave dark purple powders and flake-like shaped of particles. In contrast, SD180 and SD360 gave lower yields (e.g. 61% and 62.5%), slightly lower moisture contents (1.7 to 3.2 %), higher bulk densities (0.33 to 0.40 g cm<sup>-3</sup>) and slightly shorter rehydration times (65 to 70 s). The SD method also gave bright pink powders with less red, i.e., less like the original blueberry colour. The powder particles were smooth, spherical and smaller sized than the particles formed via FMFD.

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| Freeze drying          | Value     | Spray drying     | Value                                    |
|------------------------|-----------|------------------|--|
| parameters             |           | parameters       |  |
| Blast freezing         | -30 °C    | Inlet air        | 150 °C                                   |
| temperature            |           | temperature      |  |
| Freezing time          | 6 h       | Outlet air       | 101 <sup>0</sup> C                       |
|                        |           | temperature      |  |
| Condenser pressure     | 0.04 mbar | Feed temperature | 30 °C                                    |
| Condenser temperature  | -55 °C    | Feed rates       | 180 mL h <sup>-1</sup>                   |
|                        |           |                  | 360 mL h <sup>-1</sup>                   |
| Main drying time       | 24 h      | Aspirator rate   | $35 \text{ m}^3 \text{ h}^{-1} (100 \%)$ |
| Desorption temperature | -55 °C    | Nozzle cleaner   | 1 per min                                |
| Desorption time        | 1 h       | Air pressure     | 0.41 bar                                 |

Table 1. Foam-mat freeze-drying (FMFD) and spray-drying (SD) conditions used

| Sample          | MD/WPI | L*                            | a*                            | b*                            |
|-----------------|--------|-------------------------------|-------------------------------|-------------------------------|
| Blueberry juice | 0      | $0.08\pm0.04\ ^{m}$           | $0.45\pm0.03^{\rm l}$         | $0.06 \pm 0.0^{-1}$           |
|                 |        |                               |                               |                               |
| FMFD            | 0.4    | $44.70 \pm 0.24$ <sup>h</sup> | $60.07 \pm 0.15$ <sup>b</sup> | $18.25 \pm 0.27$ <sup>d</sup> |
|                 | 1.0    | $41.51 \pm 0.07^{i}$          | $61.94 \pm 0.01$ <sup>a</sup> | $18.25 \pm 0.27$ <sup>d</sup> |
|                 | 1.6    | $41.20 \pm 0.05$ <sup>j</sup> | $62.02 \pm 0.01$ a            | $24.92 \pm 0.11$ °            |
|                 | 2.3    | $40.54 \pm 0.06$ k            | $62.06 \pm 0.01$ a            | $26.28 \pm 0.09$ <sup>b</sup> |
|                 | 3.2    | $40.24 \pm 0.07^{1}$          | $61.97 \pm 0.01$ a            | $26.96 \pm 0.09$ <sup>a</sup> |
|                 |        |                               |                               |                               |
| SD180           | 0.4    | $52.24 \pm 0.07$ <sup>e</sup> | $56.18 \pm 0.07$ <sup>d</sup> | $12.56 \pm 0.04$ f            |
|                 | 1.0    | $51.90 \pm 0.05$ f            | $47.37 \pm 0.06^{j}$          | $10.19 \pm 0.01^{i}$          |
|                 | 1.6    | $53.16 \pm 0.05$ °            | $52.99 \pm 0.06$ <sup>h</sup> | $10.95 \pm 0.03$ h            |
|                 | 2.3    | $52.53 \pm 0.05$ <sup>d</sup> | $54.68 \pm 0.05$ f            | $12.32 \pm 0.02$ f            |
|                 | 3.2    | $51.90 \pm 0.04$ f            | $56.32 \pm 0.03$ <sup>d</sup> | $13.05 \pm 0.03$ <sup>e</sup> |
|                 |        |                               |                               |                               |
| SD360           | 0.4    | $60.34 \pm 0.04$ <sup>a</sup> | $42.42 \pm 0.06$ k            | $6.94 \pm 0.01$ k             |
|                 | 1.0    | $53.58 \pm 0.04$ <sup>b</sup> | $49.36 \pm 0.06^{i}$          | $8.80 \pm 0.02^{\text{ j}}$   |
|                 | 1.6    | $51.58 \pm 0.06$ g            | $53.27 \pm 0.06$ g            | $11.22 \pm 0.03$ gh           |
|                 | 2.3    | $52.22 \pm 0.05^{\text{e}}$   | $54.93 \pm 0.05$ °            | $11.52 \pm 0.03$ g            |
|                 | 3.2    | $51.82 \pm 0.04^{\rm \; f}$   | $56.96 \pm 0.05$ °            | $13.13 \pm 0.03$ <sup>e</sup> |

**Table 2**. Brightness (L\*), redness (a\*) and yellowness (b\*) values of blueberry juice and reconstituted solutions from FMFD and SD powders containing different weight ratios of maltodextrin to whey protein isolate (MD/WPI).

Mean values  $\pm$  range of duplicate determinations, followed by different single letter in each column if significantly different (p < 0.05, Tukey's test), whereas ab, for example, indicates values that are not significantly different from the corresponding a and b values.

| Sample          | MD/WPI | С                             | h <sup>0</sup>                | ΔΕ                            |
|-----------------|--------|-------------------------------|-------------------------------|-------------------------------|
| Blueberry juice | 0      | $0.45 \pm 0.03$ <sup>n</sup>  | $7.05 \pm 0.59^{j}$           | **                            |
|                 |        |                               |                               |                               |
| FMFD            | 0.4    | $62.78 \pm 0.22$ d            | $16.89 \pm 0.20$ <sup>d</sup> | $76.66 \pm 0.03 \ {\rm f}$    |
|                 | 1.0    | $64.58 \pm 0.09$ °            | $16.42 \pm 0.23$ d            | $76.35 \pm 0.02$ h            |
|                 | 1.6    | $66.84 \pm 0.04$ <sup>b</sup> | $21.89 \pm 0.09$ <sup>c</sup> | $78.10 \pm 0.02$ <sup>b</sup> |
|                 | 2.3    | $67.39 \pm 0.05$ a            | $22.94 \pm 0.07$ <sup>b</sup> | $78.23 \pm 0.02$ a            |
|                 | 3.2    | $67.58 \pm 0.05$ <sup>a</sup> | $23.51 \pm 0.06$ <sup>a</sup> | $78.24 \pm 0.01$ a            |
|                 |        |                               |                               |                               |
| SD180           | 0.4    | $57.57 \pm 0.08 \ {\rm f}$    | $12.60 \pm 0.03$ ef           | $77.35 \pm 0.01$ <sup>d</sup> |
|                 | 1.0    | $48.45 \pm 0.06^{-1}$         | $12.14 \pm 0.00$ fg           | $70.64 \pm 0.01$ <sup>n</sup> |
|                 | 1.6    | $54.11 \pm 0.06^{j}$          | $11.68 \pm 0.02$ g            | $75.48 \pm 0.01^{\text{ j}}$  |
|                 | 2.3    | $56.05 \pm 0.05$ h            | $12.70 \pm 0.01$ e            | $76.44 \pm 0.01$ g            |
|                 | 3.2    | $57.81 \pm 0.04$ f            | $13.04 \pm 0.02$ <sup>e</sup> | $77.30 \pm 0.01$ <sup>e</sup> |
|                 |        |                               |                               |                               |
| SD360           | 0.4    | $42.98 \pm 0.06$ m            | $9.30 \pm 0.01^{i}$           | $73.76 \pm 0.01^{1}$          |
|                 | 1.0    | $50.14 \pm 0.06^{k}$          | $10.11 \pm 0.01$ <sup>h</sup> | $73.01 \pm 0.0$ <sup>m</sup>  |
|                 | 1.6    | $54.43 \pm 0.07^{i}$          | $11.89 \pm 0.01$ g            | $74.61 \pm 0.0^{\text{ k}}$   |
|                 | 2.3    | $56.13 \pm 0.05$ <sup>h</sup> | $11.85 \pm 0.02$ g            | $76.28 \pm 0.01^{i}$          |
|                 | 3.2    | $58.48 \pm 0.05$ <sup>e</sup> | $12.97 \pm 0.02$ <sup>e</sup> | $77.74 \pm 0.01$ °            |

**Table 3**. Chroma (C), hue angle ( $h^0$ ) and  $\Delta E$  of blueberry juice and reconstituted solutions from FMFD and SD powders containing different weight ratios of maltodextrin to whey protein isolate (MD/WPI).

Mean values  $\pm$ range of duplicate determinations, followed by different single letter in each column if significantly different (p < 0.05, Tukey's test), whereas ab, for example, indicates values that are not significantly different from the corresponding a and b values.

\*\* Blueberry juice was used as a control against powder

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Figure 1. Foam density,  $\rho$  ( $\blacksquare$ ), overrun ( $\Delta$ ) as a function of mass ratio of maltodextrin to whey protein isolate (MD/WPI).

Figure 2. Drained liquid volume versus time of foamed blueberry juice at different mass ratios of maltodextrin to whey protein isolate: 9.0 ( $\bullet$ ), 7.3 ( $\bigcirc$ ), 5.3 ( $\checkmark$ ), 3.2 ( $\bigtriangledown$ ), 2.8 ( $\blacksquare$ ), 2.3 ( $\Box$ ), 1.6 ( $\blacklozenge$ ), 1.0 ( $\diamondsuit$ ), 0.4 ( $\blacktriangle$ ). The inset shows a cut of the drained volume after 60 min as a function of % maltodextrin (MD).

Figure 3. Yield (A), % moisture content (B) and a<sub>w</sub> (C) of: FMFD (■), SD 180 (O) and SD 360
(▲) powders as a function of mass ratio of maltodextrin to whey protein isolate (MD/WPI).

Figure 4. Solubility (A), Rehydration time (B) and bulk density (C) of: FMFD (■), SD 180 (O) and SD 360 (▲) powders as a function of mass ratio of maltodextrin to whey protein isolate (MD/WPI).

Figure 5. SEM Micrographs (5000 x magnification) of blueberry FMFD powder produced with MD/WPI = 0.4 (A, B) and 3.2 (C, D).

Figure 6. SEM Micrographs of blueberry SD powder produced with ratio with MD/WPI = 0.4 (A, B) and 3.2 (C, D) at magnifications of 5000 x and 15000 x, respectively.





Figure 2



Figure 3A





















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# Figure 6

