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Foam-mat freeze-drying of blueberry juice by using trehaloseβ-lactoglobulin and trehalose-bovine serum albumin as matrices

Sandi Darniadi ^{1,3*}, Idolo Ifie ², Prima Luna¹, Peter Ho ³ and Brent S. Murray ³

¹Indonesia Centre for Agricultural Postharvest Research and Development (ICAPRD), IAARD-Ministry of Agriculture, Jakarta 12540, Indonesia

² Department of Food Science and Technology, Delta State University, Abraka, PMB 1 Delta State, Nigeria

³Food Colloids and Bioprocessing Group, School of Food Science and Nutrition, University of Leeds, Leeds LS2 9JT, England, UK

*Correspondence address: sandidarniadi@pertanian.go.id

Abstract

This study aimed to evaluate the effect of pure protein compounds and trehalose incorporated into blueberry juice for foam-mat freeze-drying on the foam and powder properties. Foam-mat freeze-drying (FMFD) of blueberry juice was tested at -55 °C for 24 h. Matrices used were trehalose + β -lactoglobulin (T3BL1) and trehalose + bovine serum albumin (T3A1) and compared to maltodextrin + whey protein isolate (M3W1). Physicochemical properties of foam and powder e.g. foam stability, foam density, moisture, rehydration time, colour, particle morphology, total phenolic, and anthocyanins (total and individuals) were investigated. T3BL1 and T3A1 had more stable foam than M3W1. However, overrun of T3BL1 and T3A1 foamed were inferior to M3W1 sample. M3W1 sample recovered 79% powder (dry weight) and were superior to others. Rehydration time of powdered T3BL1 and T3A1, with bulk densities of 0.55-0.60 g cm⁻³, were the fastest (34-36 s). The blueberry powders of M3W1 showed more irregular particle size and shape, whilst the samples with pure proteins generated particles of more uniform size with obvious pores. T3BL1 and T3A1 showed less redness (a*) values than M3W1 product. All samples were considered as pure red due to hue values < 90. M3W1 was superior in total phenolic content (TPC) and total monomeric anthocyanins (TMA) compared to both samples made with trehalose + β -lactoglobulin and trehalose bovine serum albumin. Delphinidin-3-glucoside (Del3GI) concentration

was found to be higher in M3W1. Also, M3W1 had higher Cyanidin-3-glucoside (Cyn3Gl) and malvidin-3-glucoside (Mal3Gl) concentration. M3W1 also prevented the degradation of these bioactive compounds better than the other FMFD samples. The use of pure proteins and trehalose as matrices in the FMFD process had little advantage compared to maltodextrin/whey protein isolate. Thus, maltodextrin/whey protein isolate seems an ideal matrix for the manufacture of FMFD blueberry.

Key words: Foam-mat freeze-drying, blueberry powder, trehalose, bovine serum albumin, β -lactoglobulin

INTRODUCTION

The foam-mat freeze-drying (FMFD) method is the incorporation of foam-mat drying and freeze-drying to develop dehydrated food from the liquid (Muthukumaran, Ratti, and Raghavan 2008). Freeze-drying can slow down the deterioration of the product by minimization of flavor or aroma loss, maximization of nutrient retention because of the low temperature involved in the process (Ratti 2013), whist the dry powders produced can be relatively porous and rehydrate easily. Conversely, freeze-drying is well known due to its expensive operational cost (Kudra and Ratti 2006; Ratti 2013). This high cost is attributed to the four primary operations, e.g. freezing, vacuum, sublimation, and condensation. Therefore, freeze-drying has conventionally been used only for high-quality and high-value commercial products (Ratti 2013). On the other hand, some researchers have successfully used foaming to decrease processing time during conventional hot-air drying of liquids (Abbasi and Azizpour 2016; Kadam et al. 2012; A. A. Karim and Wai 1999; Thuwapanichayanan, Prachayawarakorn, and Soponronnarit 2008). In foam-mat drying, the product to be dried is converted into a foam before drying. This gives the advantage of increasing the total surface area available for drying thereby improving drying rate as well (Raharitsifa, Genovese, and Ratti 2006; Raharitsifa and Ratti 2010). Foam-mat drying has been extensively used for food products such as Bael (Bag and Srivastav 2011), yam, plantains and cooking

bananas (Falade and Okocha 2012; Falade and Onyeoziri 2012), raspberry (Dachmann et al. 2018) and sour cherry (Abbasi and Azizpour 2016). Notwithstanding, there are few studies that combine foam-mat and freeze-drying for processing of dried products. This combination is rare and warrants further research and detailed investigation to make it feasible and successful (Qadri, Srivastava, and Yousuf 2019). The primary problem of foam-mat drying is the instability of the foam, which can occur both during the foaming and drying steps (Ratti and Kudra 2006), i.e., a collapse of porous structure occurs (Thuwapanichayanan, Prachayawarakorn, and Soponronnarit 2012). Consequently, investigating foam stabilizers and foaming agents, e.g. type and concentration, is critical.

Maltodextrin (MD) and whey protein isolate (WPI) are commonly used as carrier and foaming agent in the drying process due to their film-forming property upon drying for overcoming the surface stickiness of sugar/protein solution (Celli et al. 2016; Fang and Bhandari 2012; Shi, Fang, and Bhandari 2013). Trehalose is an alternative to sucrose in the dehydration of fruit juices as it shows a relatively higher glass transition temperature (T_g) . It is well known that the T_g of trehalose is far greater than that of sucrose and many other disaccharides (Galmarini et al. 2009; Patist and Zoerb 2005) - in the anhydrous state, the T_g of trehalose and sucrose are 105–115 °C and 60–62 °C, respectively. Galmarini et al. (2009) observed that strawberry puree presented better sensory properties when dried with the addition of trehalose in comparison with sucrose and MD. Furthermore, trehalose is preferred because of its supposed additional special properties in acting as a cryoprotective agent (Komes et al. 2005). β lactoglobulin is the main surface active ingredient of WPI (Baeza et al. 2005; Fang, Wang, and Bhandari 2013). β -lactoglobulin, as a natural member of lipocalin family, exhibits a strong binding affinity for various hydrophobic and amphiphilic ligands, including fatty acids, retinol, β -carotene, phospholipids, vitamin D, folic acid, and phenolic compounds (Sahihi, Heidari-Koholi, and Bordbar 2012). Bovine serum albumin (BSA) has been used in other model studies of foaming (Glaser et al. 2007). BSA utilized for foam preparation showed maximum surface viscosity, film elasticity and surface yield stress in the

pH range 5-6. BSA showed maximum foam stability in the pH range 5-6 demonstrating the relationship between film properties and foaming properties (Zayas 1997).

Trehalose is probably always going to be more expensive than MD, but its testing in some products suggests that if it can confer significant advantages then it's use may be practical. Pure milk proteins like β -lactoglobulin and bovine serum albumin are always going to be more expensive than WPI, but the functional properties of WPI can vary widely between different suppliers and even batches, due to different degrees of denaturation during drying and differing levels of salts and lactose. Therefore, it is still interesting to compare the effects of FFD with these pure proteins to see if any trends observed here with WPI + MD were similar, which would therefore suggest that heat denaturation, etc., might not be so important. Furthermore, the use of pure proteins and sugars is more likely to aid an understanding of the protective effects of the proteins and sugars on the color of the anthocyanins of blueberries during drying. To the best of our knowledge, this will be the first such study investigating these protective effects. Consequently, the objectives this present study were: (i) to develop foam-mat freeze-dried blueberry powders with different matrices, including trehalose + pure β -lactoglobulin (T3BL1) and trehalose + bovine serum albumin (T3A1); (ii) to investigate the effect of T3BL1 or T3A1 on the physicochemical properties of the foam, blueberry powders and reconstituted solution.

MATERIALS AND METHODS

Materials

Blueberry juice (concentrated and organic) not containing additional water, sugar, preservatives and additives, according to the label information, was procured from a local store in Leeds, England. After opening, the juice was stored at 4 0 C. The use of trehalose, β -lactoglobulin and bovine serum albumin (BSA) were employed as foam stabiliser and foaming agent, respectively.

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Foam-mat freeze-drying (FMFD) conditions

Foam-mat freeze-drying was conducted according to the method of Darniadi et al. (2017). A ratio of sugar/protein = 4/2.5 was utilized for trehalose + β -lactoglobulin (T3BL1), trehalose + bovine serum albumin (T3A1), and maltodextrin + whey protein isolate (M3W1) as matrices. The foam-mat freeze-drying process was conducted in duplicate for each set of conditions.

Foam density

The method of Sadahira et al. (2016) with slight modifications was used for the measurement of foam density. A measuring cylinder (250 ml) was carefully filled up with the foam and the weight recorded. The foam density (ρ) and overrun were determined using the formula:

$\rho (g \ cm^{-3}) = mf / volume \ of foam$	Eq. 1
<i>Overrun</i> (%) = 100 (<i>mi-mf</i>) / <i>mf</i>	Eq. 2

Where *mi* represents the mass of the initial solution (unwhipped sample), and *mf* is taken as mass of the whipped sample having the same volume of *mi*.

Foam stability: drainage

Foam drainage was analyzed using the method of Raharitsifa et al. (2006) after slight modifications. 50 mL of each foam was introduced to a Buchner funnel filling it to the top. Thereafter, the liquid drained from the foam due to gravity was collected in a 50 mL graduated cylinder and the volume V (mL) measured directly as a function of time over 120 min.

Determination of yield

The product yield was calculated as the ratio of the mass of solid powder obtained at the end of the freeze-drying to the mass of initial substances dried (including MD and WPI) as published by Shi et al. (2013).

Moisture content and water activity

A HB 42-S halogen moisture analyzer (Mettler Toledo, UK) set at 105 ^oC was used to measure the moisture content of FMFD powder. 1 g of powder was placed in the sample pan and the empty sample pan handler and the heating module were closed. The drying time for each sample fell within 2-5 minutes. For measurement of water activity, 0.5 g of sample powders were carefully weighed and analyzed using a Hygrolab C1 water activity meter (Rotronic, UK) (Darniadi et al. 2017).

Solubility

The method published by Ceballos, Giraldo, and Orrego (2012) was used to measure the solubility in sample powders. Water solubility was calculated using the following equation:

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

Rehydration time

Rehydration of blueberry powder was done according to the method described by Islam et al. (2016) after minor modifications. 0.5 g of powder was introduced to a 100 mL glass beaker containing 50 mL distilled water at 26 °C. A Stuart CB-162 magnetic stirrer (Bibby Scientific Ltd, UK) programmed at 900 rpm was used to stir the mixture and the time recorded when no particulate matter was visible to the naked eye.

Bulk density

1 g of blueberry powder was transferred to an empty 10 mL graduated cylinder and placed on an FB 15012 top mix vibrator (Fischer Scientific, UK) set at 2,000 rpm for 1 min. The bulk density was calculated by dividing the mass of the powder by the volume occupied in the cylinder (Shi et al. 2013).

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 kV0 (Darniadi et al. 2017; Franceschinis et al. 2014).

Color properties

The Lightness (L*), redness (a*), and yellowness (b*) values of powders were determined according to the method described by Franceschinis et al. (2015) and Mahdavee Khazaei et al. (2014).

Determination of total monomeric anthocyanin

Reconstituted FMFD powders were analysed for total monomeric anthocyanin using the pH differential method (Cliff, King, and Schlosser 2007; Eisele et al. 2005). Two portions of test sample were diluted with pH 1.0 and pH 4.5 buffers respectively and the absorbance measured at both 520 nm and 700 nm against a blank cell filled with distilled water using a 6715 UV /VIS spectrophotometer (Jenway, UK). Anthocyanin pigment concentration was then calculated and expressed as cyanidin-3-glucoside equivalents, as follows:

$$TMA (mg/L) = \frac{(A \times MW \times DF \times 1000)}{\varepsilon \times 1} Eq. 5$$

where A = (absorbance at 520 nm – absorbance at 700nm)_{pH 1.0} – (absorbance at 520 nm – absorbance at 700nm)_{pH 4.5}; MW (molecular weight) = 449.2 g/mol for cyanidin-3-glucoside (Cyn3Gl); DF =

dilution factor; l = path length in cm; $\mathcal{E} = 26\ 900\ molar$ extinction coefficient, in L/mol x cm, for cyd-3-glu; and 1000 = factor for conversion from g to mg.

Determination of total phenolic content

The total phenolic content (TPC) in reconstituted blueberry powders was determined using Folin-Ciocalteau's method (Ifie et al. 2016). The assay contained 1 mL of sample (concentrated blueberry juice or reconstituted blueberry powder) diluted (1:10) with 80 % methanol solution, 5 mL of diluted Folin-Ciocalteau's phenol reagent (1:10), and 4 mL of 75 g/L sodium carbonate solution. The mixture was then incubated in a water bath at 25 $^{\circ}$ C and the absorbance measured after 2 h with a spectrophotometer at 765 nm. The estimation of phenolic content was done using gallic acid as standard.

HPLC-PAD (HPLC coupled with photodiode array) individual ACNs

HPLC analysis was conducted according to the method described by Darniadi et al. (2019). A UFLCXR system (Shimadzu) comprising of a binary pump, a photodiode array with multiple wavelengths (SPD-20A), a Solvent Delivery Module (LC-20AD) coupled with an online unit degasser (DGU-20A3/A5) and a thermostat auto sampler/injector unit (SIL-20A). The photodiode array detector was set to measure at a wavelength of 520 nm.

Identification of anthocyanins in the reconstituted powders was achieved by comparing the retention time with external standards of phenolic compounds run under similar conditions, UV-visible spectrum and spiking of the sample with the corresponding standard phenolic compound. A standard curve was developed using cyanidin-3-glucoside (Cyn3Gl) to express total monomeric anthocyanin (TMA) as mg/g Cyn3Gl solids, while quantification of individual anthocyanins was done by external standards: cyanidin-3-glucoside (Cyn3Gl), delphinidin-3-glucoside (Del3Gl) and malvidin-3-glucoside (Mal3Gl).

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

Foam capacity and foam stability

Figure 1A presents the foam stability of three different carrier agents. The foam stability was determined by measuring the liquid that drained from the foam. The carrier agent types had a significant (p < 0.05) effect on the foam stability (drain volume). It was observed that M3W1 had higher liquid drain (less stable foam) compared to those samples prepared with pure proteins (more stable foam). This occurrence can possibly be attributed to the type of proteins used, where β -lactoglobulin and bovine serum albumin form a stiffer foam with more air entrapped (therefore lower density). As a result, the low-density foam traps more water (less liquid drainage) than the high-density foam (A.A. Karim and Wai 1999). Moreover, Dachmann et al. (2018) reported the stability of foamed (aged for 30 min) raspberry puree, on the addition of potato protein isolate (5% w/w) and maltodextrin (5% w/w), was 50%. This foam stability is inferior compared to the blueberry foams (aged for 30 min) in our study, which were 54%, 76%, and 82% for M3W1, T3BL1 and T3A1 respectively. This can possibly attributed to their higher viscosity and water-binding capacities.

Foam overrun of the blueberry foam prepared by three carrier agents are shown in Figure 1B. ANOVA of observed data showed there were a significant (p < 0.05) differences among all foams. The overrun of M3W1 was recorded as 762% and higher in comparison with T3BL1 (608%) and T3A1 (654%). Zayas (1997) reported that foam overrun values ranged from 753% for WPI 5% w/w. The lower overrun

of T3BL1 is possibly due to the fact that β -lactoglobulin requires a slightly higher temperature for optimum unfolding and adsorbed film formation (Zayas 1997). The overruns from our study were also higher values than the foamed raspberry puree prepared with 5% (w/w) maltodextrin and 5% (w/w) potato protein isolate (Dachmann et al. (2018)). (The overrun of the raspberry puree was about 580%). This highlights how the choice of foaming agent affects foam stability and overrun during whipping (Thuwapanichayanan et al. 2012).

Powder yield, moisture content and water activity (a_w)

The yield of foam-mat freeze-dried powder prepared with M3W1, T3BL1, and T3A1 is presented in Figure 2A. ANOVA of yield showed that M3W1 had statistically (p < 0.05) higher yield compared to those prepared with pure proteins. The yield of M1W3 samples reached 79%, whereas T3BL1 and T3A1 recovered 66 and 67%, respectively. In a different experiment, the yield was also higher in freeze-dried blueberry juice prepared with β -cyclodextrin (15% w/w); it was calculated as 78% (Wilkowska et al. 2016).

Foam-mat freeze-dried powders achieved variable moisture content and a_w as shown in Figure 2B. There was a significant (p < 0.05) effect of carrier agents on the moisture content. The moisture content of M3W1 was higher (3.5%) compared to T3BL1 and T3A1. It was observed that between T3BL1 (1.8%) and T3A1 (1.3%) there was no statistical difference (p > 0.05) in moisture content. The T3BL1 and T3A1 gave lower moisture content than M3W1 possibly due to the more stable foams of T3BL1 and T3A1. Therefore, the stable foam led to increase in the porosity of the structure, increase in the drying rate, and consequently reduced the drying time (Abbasi and Azizpour 2016). In the case of water activity, ANOVA of a_w showed that the foam-mat freeze-dried sample prepared with trehalose and pure proteins had statistically (p < 0.05) lower a_w compared to M3W1 sample. T3BL1 and T3A1 obtained a_w of 0.18 and 0.25 respectively, while a_w of M3W1 was 0.27. The low water activity values are probably

related to the moisture loss in the samples because of their more porous structure (Azizpour, Mohebbi, and Khodaparast 2016).

Solubility, rehydration time, and bulk density

The solubility of foam-mat freeze-dried powders prepared with T3BL1, T3A1, and M3W1 is shown in Figure 3A. Type of carrier agents had no significant effect (p > 0.05) on the solubility of foam-mat freeze-dried samples. The solubility was 98.3, 96.7, and 96.5%, for T3BL1, T3A1, and M3W1 respectively. These results seem to be higher compared to the foam-mat dried of sour cherry powder (solubility 43-48%) using egg white and methylcellulose as foaming agents (Abbasi and Azizpour 2016). Different types of proteins and polysaccharides employed in foam-mat drying cause variations in foam structural stability. More stable foams result in more bubbles being retained during drying. Thus, these bubbles increase the porosity of the powder and its solubility (Abbasi and Azizpour 2016). Figure 3B shows rehydration time of the foam-mat freeze-dried powders made with T3BL1, T3A1, and M3W1. ANOVA of rehydration time revealed that there were significant (p < 0.05) differences among all the samples. The shortest rehydration time recorded was 34 s (T3BL1), while M3W1 gave the longest rehydration time, i.e. 90 s. The bulk density of foam-mat freeze-dried powders is also presented in Figure 3B. There was a significant effect of carrier agent type on the bulk density. M3W1 had a lower bulk density (0.32 g/cm^3) compared to the foam-mat freeze dried samples made with trehalose and pure proteins, i.e. 0.55 and 0.6 g/cm³. The high bulk density caused by the presence of water is considerably denser than the dry solids (Chegini and Ghobadian 2005; Fazaeli et al. 2012.). As a consequence, the particles are wetted easily and result in a reduction of rehydration time. However, the less dense powder tends to be less wettable, due to the entrapped air amongst particles, thus causing an increase in rehydration time.

Particles morphology

Figure 4 presents SEM micrographs of foam-mat freeze dried powders made with T3BL1, T3A1 and M3W1. The particles of three foam-mat freeze-dried powders had irregular shapes. However, the first two foam-mat freeze-dried powders made with trehalose and pure proteins generated pores and ordered structures, while the M3W1 powder had broken glass-like structures. These results were attributed to the foam density and stability: the T3BL1 and T3A1 both had lower foam density and higher foam stability compared to M3W1 samples. Thus, T3BL1 and T3A1 generated more small bubbles. Furthermore, when the water was sublimed from the foam, these bubbles retained a similar shape and size compared to before the freeze-drying stage. These results are in agreement with the study carried out by Thuwapanichayanan et al. (2008). In the case of M3W1, the foam drained quickly and could not hold the structure due to foam instability. Consequently, once the foams were dried and crushed, they showed flake-like particles. Franceschinis et al. (2014) reported a similar phenomenon in producing blueberry powder using the freeze-drying technique.

Color properties

Color is one of the important properties of fruit powders, which has a great influence on their desirability and final price (Azizpour et al. 2016). Lightness (L*), redness (a*), and yellowness (b*) values of foammat freeze-dried reconstituted powder are shown in Figure 5A. The L* value of reconstituted powder made with trehalose and pure proteins was statistically (p < 0.05) lower than the sample made with M3W1. This phenomenon indicates that T3BL1 and T3A1 had little influence on the brightness of the reconstituted solutions. The a* value of M3W1 was significantly higher (p < 0.05) than the other foammat freeze-dried samples. The a* value is related to the anthocyanins content of foam-mat freeze-dried powders. The high a* value of M3W1 sample can be attributed to high total monomeric anthocyanins (TMA) content. In the case of b*, M3W1 also recorded the highest reading, while T3BL1 and T3A1 gave lower (p < 0.05) b* values compared to M3W1. The study conducted by Franceschinis et al. (2014) and Duangmal, Saicheua, and Sueeprasan (2008) showed that there was an increase in a* and b* value by addition maltodextrin and/or trehalose during production of blackberry powder and Roselle anthocyanin obtained by freeze-drying. Total colour density (TCD), Chroma (C*), and Hue (H⁰) of foam-mat freeze-dried reconstituted powders are presented in Figure 5B. M3W1 sample was found to be higher in total colour density (TCD) and C* values than those of T3BL1 and T3A1. TCD of M3W1 was recorded as 78, while T3BL1 and T3A1 were 48 and 6, respectively. This high TCD can be correlated with the high level of a* reading and the total monomeric anthocyanins content in M3WI samples. The C* value of M3W1 was calculated as 67, whereas both powders made with trehalose and pure proteins gave C* < 50. In the case of Hue, the T3BL1 were slightly higher (H⁰ = 29) compared to the M3W1 sample (H⁰ = 23). H⁰ of the three foam-mat freeze-dried samples were found to be < 90, which were considered as pure red.

Total monomeric anthocyanin (TMA) and total phenolic content (TPC)

Figure 6A shows the total phenolic content (TPC) and total monomeric anthocyanins (TMA) of foammat freeze-dried made with three different carrier agents. It was observed that M3W1 was superior (p < 0.05) in TPC and TMA compared to both samples made with trehalose + β -lactoglobulin and trehalose + BSA. The M3W1 had 31.5 mg GAE g⁻¹ solids and 8.5 mg Cyn3Gl g⁻¹ solids for the TPC and TMA, respectively. The TPC of T3BL1 and T3A1 obtained were 17.7 ± 0.08 and 14.5 ± 0.3 mg GAE g⁻¹ solids, respectively. In the case of TMA content, T3BL1 and T3A1 had 5.9 ± 0.33 and 5.1 ± 0.09 mg Cyn3Gl g⁻¹ solids, respectively. This phenomenon could be related to maltodextrin used in M3W1 sample, which was better at preserving phenolic compounds and anthocyanins compared to trehaloseprotein-treated samples as demonstrated by other researchers (Duangmal, Saicheua, and Sueeprasan 2008; Franceschinis et al. 2014). Therefore, the M3W1 powder had a high retention of TPC and TMA (Figure 6B) which were 73 and 95% for TPC and TMA, i.e. < 60%. Franceschinis et al.(2014) reported a retention of 73 and 75% for TPC and TMA retention of freeze-dried blackberry powder made with maltodextrin. However, the M3W1 sample showed lower TPC retention when compared to the freezedried blueberry powder (TPC retention: 95%) reported in elsewhere (Turan, Cengiz, and Kahyaoglu 2015). This may be related to the blueberry cultivar used in the experiment. Similarly, anthocyanin retention (50-80%) reported elsewhere (Ozcelik et al. 2019) in freeze-dried raspberry puree using maltodextrin and potato protein was less superior to values observed in this study.

Individual anthocyanins

The data on individual anthocyanins of foam-mat freeze-dried produced with T3BL1, T3A1, and M3W1 are presented in Figure 7. Del3Gl concentration was found to be higher in M3W1. Also, M3W1 had the highest Cyn3Gl and Mal3Gl concentration (Figure 7A). The concentration of Del3Gl, Cyn3Gl, and Mal3Gl in M3W1 sample was recorded as 1.17., 1.38, and 0.85 mg g⁻¹ solids, respectively. Retention of individual anthocyanins is shown in Figure 7B. M3W1 prevented the degradation of individual anthocyanins better than other foam-mat freeze-dried samples. Del3Gl retention of M3W1 was calculated as 85%, while 46 and 48% was recorded from T3BL1 and T3A1 powders (Figure 7B). In the case of Cyn3Gl retention, M3W1 recovered > 95 %, whereas T3BL1 and T3A1 recovered 64 and 69%, respectively. Again, M3W1 had a high retention of Mal3Gl (85%), while both powder samples made with pure protein recovered 52-53%. In a recent study, processing of grape juice into powders by foam mat drying using maltodextrin showed a retention of 71-79%, 79-100% and 77.5-87.5% for Del3Gl, Cyn3Gl and Mal3Gl respectively (Maria et al. 2019).

CONCLUSIONS

Foam-mat freeze-dried blueberry powders were successfully made with different matrices of trehalose and pure proteins. These samples were then compared to maltodextrin + whey protein (M3W1)-treated samples. T3BL1 and T3A1 had lower drained liquid volumes compared to M3W1 foams, which means they were stiffer and more stable. However, the overruns of T3BL1 and T3A1 foams were inferior compared to M3W1 samples. M3W1-treated samples gave a powder recovery of 79% (dry weight), while pure protein-treated samples gave 66-67% (dry weight). There were no significant differences in solubility of the reconstituted product amongst all samples. Rehydration times of T3BL1 and T3A1 were 34 and 36 s respectively, while M3W1 sample was 90 s. This longer time may be due to the higher density of powder and lower moisture content of T3BL1- and T3A1-treated powders. The blueberry powders of M3W1 consisted of irregular particles, while pure protein-treated samples gave powders with a more uniform particle size that also contained pores of regular size. T3BL1and T3A1 recorded less redness (a*) values compared to M3W1 product. All samples were considered as pure red since they all had hue values < 90. M3W1 was superior in the TPC and TMA compared to both samples made with trehalose + β -lactoglobulin and trehalose bovine serum albumin. Del3G1 concentration was found to be higher in M3W1, as well as Cyn3G1 and Mal3G1 concentrations. M3W1 prevented the degradation of these bioactive compounds (TPC AND TMA) better than other foam-mat freeze-dried samples.

Overall, the foam-mat freeze-dying process was found to be a promising alternative to conventional freeze-drying, as it offers better physical properties with good protection of bioactive compounds and there appears to be no particular advantage in using the more pure (and expensive) matrix materials trehalose, β -lactoglobulin or bovine serum albumin under the FMFD conditions investigated.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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REFERENCES

- Abbasi, Elnaz and Mehran Azizpour. 2016. "Evaluation of Physicochemical Properties of Foam Mat Dried Sour Cherry Powder." *LWT - Food Science and Technology* 68:105–10.
- Azizpour, Mehran, Mohebbat Mohebbi, and Mohammad Hosein Haddad Khodaparast. 2016. "Effects of Foam-Mat Drying Temperature on Physico-Chemical and Microstructural Properties of Shrimp Powder." *Innovative Food Science and Emerging Technologies* 34:122–26.
- Baeza, Rosa, Cecilio Carrera Sanchez, Ana M. R. Pilosof, and Juan M. Rodríguez Patino. 2005.
 "Interactions of Polysaccharides with β-Lactoglobulin Adsorbed Films at the Air-Water Interface."
 Food Hydrocolloids 19(2):239–48.
- Bag, Subrata Kumar and Prem P. Srivastav. 2011. "Optimization of Process Parameters for Foaming of Bael (Aegle Marmelos L.) Fruit Pulp." *Food and Bioprocess Technology* 4:1450–58.
- Ceballos, Adela Maria, Gloria Ines Giraldo, and Carlos Eduardo Orrego. 2012. "Effect of Freezing Rate on Quality Parameters of Freeze Dried Soursop Fruit Pulp." *Journal of Food Engineering* 111(2):360–65.
- Celli, Giovana, Rojin Dibazar, Amyl Ghanem, and Su-Ling Brooks Marianne. 2016. "Degradation Kinetics of Anthocyanins in Freeze-Dried Microencapsulates from Lowbush Blueberries (Vaccinium Angustifolium Aiton) and Prediction of Shelf-Life." *Drying Technology* 34(10):1175– 84.
- Chegini, G. R. and B. Ghobadian. 2005. "Effect of Spray-Drying Conditions on Physical Properties of Orange Juice Powder." *Drying Technology* 23(June 2015):657–68.
- Cliff, Margaret a., Marjoire C. King, and Jimmy Schlosser. 2007. "Anthocyanin, Phenolic Composition, Colour Measurement and Sensory Analysis of BC Commercial Red Wines." *Food Research International* 40(1):92–100.

Dachmann, E., C. Hengst, M. Ozcelik, U. Kulozik, and J. Dombrowski. 2018. "Impact of Hydrocolloids

and Homogenization Treatment on the Foaming Properties of Raspberry Fruit Puree." *Food and Bioprocess Technology*.

- Darniadi, S., P. Ho, and B. S. Murray. 2017. "Comparison of Blueberry Powder Produced via Foam-Mat Freeze-Drying versus Spray-Drying: Evaluation of Foam and Powder Properties." *Journal of the Science of Food and Agriculture* 2002–10.
- Darniadi, Sandi, Idolo Ifie, Peter Ho, and B. S. Murray. 2019. "Evaluation of Total Monomeric Anthocyanin, Total Phenolic Content and Individual Anthocyanins of Foam-Mat Freeze-Dried and Spray-Dried Blueberry Powder." *Food Measurement and Characterization*.
- Duangmal, Kiattisak, Busararat Saicheua, and Suchitra Sueeprasan. 2008. "Colour Evaluation of Freeze-Dried Roselle Extract as a Natural Food Colorant in a Model System of a Drink." *LWT Food Science and Technology* 41(8):1437–45.
- Eisele, T., M. M. Giusti, H. Hofsommer, S. Koswig, D. A. Krueger, S. Kupina, S. K. Martin, B. K. Martinsen, T. C. Miller, F. Paquette, A. Ryabkova, G. Skrede, U. Trenn, and J. D. Wightman. 2005. "Determination of Total Monomeric Anthocyanin Pigment Content of Fruit Juices, Beverages, Natural Colorants, and Wines by the PH Differential Method: Collaborative Study." *Journal of AOAC International* 88:1269–78.
- Falade, Kolawole O. and Joy O. Okocha. 2012. "Foam-Mat Drying of Plantain and Cooking Banana (Musa Spp.)." Food and Bioprocess Technology 5:1173–80.
- Falade, Kolawole O. and Ngozi F. Onyeoziri. 2012. "Effects of Cultivar and Drying Method on Color, Pasting and Sensory Attributes of Instant Yam (Dioscorea Rotundata) Flours." Food and Bioprocess Technology 5:879–87.
- Fang, Z. and B. Bhandari. 2012. "Comparing the Efficiency of Protein and Maltodextrin on Spray Drying of Bayberry Juice." *Food Research International* 48(2):478–83.
- Fang, Zhongxiang, Ruobing Wang, and Bhesh Bhandari. 2013. "Effects of Type and Concentration of Proteins on the Recovery of Spray-Dried Sucrose Powder." *Drying Technology* 31(13–14):1643– 52.

- Fazaeli, Mahboubeh, Zahra Emam-Djomeh, Ahmad Kalbasi Ashtari, and Mahmoud Omid. 2012.
 "Effect of Spray Drying Conditions and Feed Composition on the Physical Properties of Black Mulberry Juice Powder." *Food and Bioproducts Processing* 90(4):667–75.
- Franceschinis, Lorena, Daniela M. Salvatori, Natalia Sosa, and Carolina Schebor. 2014. "Physical and Functional Properties of Blackberry Freeze- and Spray-Dried Powders." *Drying Technology* 32(2):197–207.
- Franceschinis, Lorena, Paula Sette, Carolina Schebor, and Daniela Salvatori. 2015. "Color and Bioactive Compounds Characteristics on Dehydrated Sweet Cherry Products." *Food and Bioprocess Technology* 8(8):1716–29.
- Galmarini, Mara V., Carolina Schebor, María C. Zamora, and Jorge Chirife. 2009. "The Effect of Trehalose, Sucrose and Maltodextrin Addition on Physicochemical and Sensory Aspects of Freeze - Dried Strawberry Puree." *International Journal of Food Science and Technology* 44(10):1869– 76.
- Glaser, L. A., A. T. Paulson, R. A. Speers, R. Y. Yada, and D. Rousseau. 2007. "Foaming Behavior of Mixed Bovine Serum Albumin-Protamine Systems." *Food Hydrocolloids* 21(4):495–506.
- Ifie, Idolo, Lisa J. Marshall, Peter Ho, and Gary Williamson. 2016. "Hibiscus Sabdariffa (Roselle) Extracts and Wine: Phytochemical Profile, Physicochemical Properties, and Carbohydrase Inhibition." *Journal of Agricultural and Food Chemistry* 64:4921–31.
- Islam, M. Z., Yutaka Kitamura, Yoshitsugu Yamano, and Mai Kitamura. 2016. "Effect of Vacuum Spray Drying on the Physicochemical Properties, Water Sorption and Glass Transition Phenomenon of Orange Juice Powder." *Journal of Food Engineering* 169:131–40.
- Kadam, D. M., R. A. Wilson, Kaur Varinder, S. Chadha, Kaushik Pratibha, Kaur Sumandeep, R. T.
 Patil, and D. R. Rai. 2012. "Physicochemical and Microbial Quality Evaluation of Foam-Mat-Dried Pineapple Powder." *International Journal of Food Science & Technology* 47(8):1654–59.
- Karim, A.A. and C. Chee Wai. 1999. "Characteristics of Foam Prepared from Starfruit (Averrhoa Carambola L .) Puree by Using Methyl Cellulose." *Food Hydrocolloids* 13:203–10.

- Karim, A. A. and C. Chee Wai. 1999. "Foam-Mat Drying of Starfruit (Averrhoa Carambola L.) Puree. Stability and Air Drying Characteristics." *Food Chemistry* 64(3):337–43.
- Komes, Draženka, Tomislav Lovrić, K. K. Ganić, J. G. Kljusurić, and Mara Banović. 2005. "Trehalose Improves Flavour Retention in Dehydrated Apricot Puree." *International Journal of Food Science* and Technology 40(4):425–35.
- Kudra, T. and C. Ratti. 2006. "Foam-Mat Drying: Energy and Cost Analyses." *Canadian Biosystem Engineering* 48:27–32.
- Mahdavee Khazaei, K., S. M. Jafari, M. Ghorbani, and a Hemmati Kakhki. 2014. "Application of Maltodextrin and Gum Arabic in Microencapsulation of Saffron Petal's Anthocyanins and Evaluating Their Storage Stability and Color." *Carbohydrate Polymers* 105:57–62.
- Maria, Iasnaia, De Carvalho Tavares, Maurício Bonatto, Machado De Castilhos, Maria Aparecida, Afonso Mota, Reginaldo Teodoro, De Souza, Sergio Gómez-alonso, Eleni Gomes, Roberto Dasilva, Isidro Hermosín-gutiérrez, and Ellen Silva Lago-vanzela. 2019. "BRS Violeta (BRS Rúbea × IAC 1398-21) Grape Juice Powder Produced by Foam Mat Drying . Part I : Effect of Drying Temperature on Phenolic Compounds and Antioxidant Activity." *Food Chemistry* 298(May):124971.
- Muthukumaran, Arun, Cristina Ratti, and Vijaya G. S. Raghavan. 2008. "Foam-Mat Freeze Drying of Egg White and Mathematical Modeling Part II Freeze Drying and Modeling." *Drying Technology* 26(4):508–12.
- Ozcelik, M., A. Heigl, U. Kulozik, and S. Ambros. 2019. "Effect of Hydrocolloid Addition and Microwave-Assisted Freeze Drying on the Characteristics of Foamed Raspberry Puree." *Innovative Food Science and Emerging Technologies* 56(January):102183.
- Patist, Alex and Hans Zoerb. 2005. "Preservation Mechanisms of Trehalose in Food and Biosystems." *Colloids and Surfaces B: Biointerfaces* 40(2):107–13.
- Qadri, Ovais Shafiq, Abhaya Kumar Srivastava, and Basharat Yousuf. 2019. "Trends in Foam Mat Drying of Foods : Special Emphasis on Hybrid Foam Mat Drying Technology." *Critical Reviews*

in Food Science and Nutrition 0(0):1–10.

- Raharitsifa, N., D. B. Genovese, and C. Ratti. 2006. "Characterization of Apple Juice Foams for Foam-Mat Drying Prepared with Egg White Protein and Methylcellulose." *Journal of Food Engineering and Physical Properties* 71(3):E142–51.
- Raharitsifa, Narindra and Cristina Ratti. 2010. "Foam-Mat Freeze-Drying of Apple Juice Part 2: Stability of Dry Products During Storage." *Journal of Food Process Engineering* 33(2010):341–64.
- Ratti, C. and T. Kudra. 2006. "Drying of Foamed Biological Materials: Opportunities and Challenges." *Drying Technology* 24(9):1101–8.
- Ratti, Cristina. 2013. "Freeze Drying for Food Powder Production." Pp. 57–84 in *Handbook of Food Powder*, edited by and P. S. Bhes Bandari, Nidhi Bansal, Min Zhang. Woodhead Publishing.
- Sadahira, Mitie S., Maria I. Rodrigues, Mahmood Akhtar, Brent S. Murray, and Flavia M. Netto. 2016.
 "Effect of Egg White Protein-Pectin Electrostatic Interactions in a High Sugar Content System on Foaming and Foam Rheological Properties." *Food Hydrocolloids* 58:1–10.
- Sahihi, Mehdi, Zobeideh Heidari-Koholi, and Abdol-Khalgh Bordbar. 2012. "The Interaction of Polyphenol Flavonoids with β-Lactoglobulin: Molecular Docking and Molecular Dynamics Simulation Studies." *Journal of Macromolecular Science, Part B: Physics Journal of Macromolecular Science R*, Part B: Physics 51:2311–23.
- Shi, Qilong, Zhongxiang Fang, and Bhesh Bhandari. 2013. "Effect of Addition of Whey Protein Isolate on Spray-Drying Behavior of Honey with Maltodextrin as a Carrier Material." *Drying Technology* 31(13–14):1681–92.
- Thuwapanichayanan, Ratiya, Somkiat Prachayawarakorn, and Somchart Soponronnarit. 2008. "Drying Characteristics and Quality of Banana Foam Mat." *Journal of Food Engineering* 86(4):573–83.
- Thuwapanichayanan, Ratiya, Somkiat Prachayawarakorn, and Somchart Soponronnarit. 2012. "Effects of Foaming Agents and Foam Density on Drying Characteristics and Textural Property of Banana Foams." *LWT - Food Science and Technology* 47(2):348–57.

- Turan, F. T., A. Cengiz, and T. Kahyaoglu. 2015. "Evaluation of Ultrasonic Nozzle with Spray-Drying as a Novel Method for the Microencapsulation of Blueberry's Bioactive Compounds." *Innovative Food Science and Emerging Technologies* 32:136–45.
- Wilkowska, Agnieszka, Wojciech Ambroziak, Agata Czyzowska, and Janusz Adamiec. 2016. "Effect of Microencapsulation by Spray-Drying and Freeze-Drying Technique on the Antioxidant Properties of Blueberry (Vaccinium Myrtillus) Juice Polyphenolic Compounds." *Polish Journal* of Food and Nutrition Sciences 66(1):11–16.
- Zayas, Joseph F. 1997. "Foaming Properties of Proteins." Pp. 260–309 in *Functionality of Proteins in Food*. Berlin: Springer-Verlag.



Figure 1 A: drain volume of blueberry juice-foam with M3W1 (\blacktriangle), T3BL1 (\blacksquare), and T3A1 (\bigcirc). B: Overrun of blueberry juice-foam prepared by M3W1, T3BL1, and T3A1.



Figure 2 A: Yield of FMFD powders. B: Moisture content (\blacksquare) and water activity/ $a_w(\Box)$



Figure 3 A: Solubility of FMFD powders. B: Rehydration (\blacksquare) and bulk density (\Box) of FMFD powders.



Figure 4 SEM micrographs of foam-mat freeze-dried powders made with T3BL1, T3A1, and M3W1, magnification 5,000 x



Figure 5 A: L*, a*, and b* values of FMFD powders. B: Hue/ H^0 , Chroma/C*, and total colour difference/TCD of FMFD reconstituted powders.



Figure 6 A: Total phenolic content/TPC and total monomeric anthocyanins/TMA of FMFD powders. B: Retention of TPC and TMA of FMFD powders.



Figure 7 A: Concentration of Del3Gl, Cyn3Gl, and Mal3Gl of FMFD powders. B: Retention of Del3Gl, Cyn3Gl, and Mal3Gl of FMFD powders. Results are expressed as means ± range of duplicate determinations