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Stickiness and agglomeration of blackberry and raspberry spray dried juices using agave fructans and maltodextrin as carrier agents

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Abstract: The present study shows the effect of agave fructans as a carrier agent compared with maltodextrin to evaluate the particle stability of blackberry and raspberry juices. A pilot spray dryer was used with feed flow of 20 mL h^{-1} and atomization rate of 28 000 rpm. The inlet air temperature of 180 °C and outlet air temperature of 80 °C were used as parameter constants. Only the parameters of the carrier agent concentration of 5, 7.5 to 10% (w/v) were changed. The concentration of 10% agave fructans was high enough to recover the higher yields of 89% only for blackberry, for raspberry the concentration of 7.5% agave fructans was sufficient to recover the yield of 67%. The stability diagrams show the conditions of the particles that should not be exceeded when leaving spray drying, as well as the storage conditions that must be followed to avoid agglomeration.

Keywords: microencapsulation; additive; glass transition temperature; stability diagram; sorption isotherms

The increasing consumer demand for berries in the world is due to their pleasant taste and their beneficial properties to health. One of the main reasons that has increased the popularity of blackberry and raspberry in the human diet is that they are an important and natural source of antioxidants (Pantelidis et al. 2007). Blackberry and raspberry are extensively consumed as fresh fruit or processed into jams, juices, jellies, liqueurs, sauces, purees, ice creams and wine products (Yousefi et al. 2014). However, though blackberry and raspberry have a high potential in the food industry because of their sensory and nutritional qualities, these are highly perishable and have a short post-harvest life, limiting their marketing. Anthocyanins and phenolics are responsible for the antioxidant properties and are generally susceptible to degradation reactions during processing and storage due to their sensitivity to adverse environmental conditions such as high temperatures, light and oxygen (Ferrari et al. 2012). Microencapsulation techniques have been widely used in the food industry to prevent deterioration of antioxidants and functional food components, controlling the rate of release, improving performance, and increasing glass transition temperatures (Nayak & Rastogi 2010). Among these techniques, spray drying is preferred because it produces powders of good quality which facilitate their transportation and storage. Extracts of blackberry and raspberry have a high content

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of low-molecular-weight sugars such as sucrose, glucose and fructose (Xie et al. 2009), which may cause problems of stickiness during spray drying of their respective juices. The stickiness of sugars is mainly due to their low glass transition temperatures (Tg) (Vega et al. 2005), normally an amorphous product exhibits stickiness at 30 °C above the Tg and the outlet air temperature of spray drying is generally between 60 and 100 °C (Bhandari et al. 1997). The state of the particle stickiness can cause cohesion and adhesion of material on the surfaces of the dryer and lump formation in the product particles to adversely affect the free flow property (Fang & Bhandari 2012). This is a problem in the food industry because it causes considerable economic losses (Boonyai et al. 2004). To overcome these problems, the addition of carriers and agents is a common practice. the most commonly used carrier agent is maltodextrin because of its low cost and high water solubility. However, the powder obtained using this polymer as a carrier agent commonly possesses high hygroscopicity which makes difficult its storage and use. Studies by Bhandari et al. (1993) and Righetto & Netto (2005) concluded that concentrations of maltodextrin above 35% help counter the problem of stickiness. However, these large amounts of carrier agents may have problems such as increased cost of drying and altering the original flavour of the product, which can influence consumer acceptance. Different and novel polymers are tested as potential carrier agents. Agave fructans is a generic name for linear or branched polymers consisting mainly of β (2-1) and β (2-6) fructosyl-fructose bonds of various lengths and terminated by a single glucose molecule. Actually, it is commercialized as a powdered product which provides important advantages such as easier manipulation, transportation, and storage. Additionally, it is colourless, flavourless and very soluble in water. All these characteristics make agave fructans a potential new and alternative carrier agent (Moreno et al. 2016). Therefore, the aim of this study was to evaluate the efficiency of the microencapsulation and agglomeration control of spray-dried blackberry and raspberry extracts using agave fructans and maltodextrin as carrier agents.

MATERIAL AND METHODS

Material and sample preparation

The blackberry and raspberry juices were obtained using a pilot-scale pulper (Jersa DPC-02; Jersa®, Mexico), using fresh fruits of blackberries and raspberries obtained from a local market (Jocotepec, Mexico). The soluble solids in the obtained juices were between 9 and 15% of soluble solids, for this reason the juices were standardized at 10% of soluble solids with added water or sugar and stored (–4 °C). The fractions of fructans used in this study were obtained by tangential filtration performed at the Integral Laboratory of Investigation in Foods (LIIA), in the Technological Institute of Tepic, as reported by Palatnik et al. (2017), obtaining fractions enriched with a profile of fructans with Inulin standard apparent a degree polymerisation (DP) of 4–80 for the High Performance Agave Fructans (HPAF) fraction, with 0.80 g 100 g^{-1} of reducing sugar and DP of 24–80 for High Degree Polymerisation Agave Fructans (HDPAF), with 0.40 g 100 g^{-1} of reducing sugar (Ortiz-Basurto et al. 2017).

Spray drying

A pilot-scale spray dryer (Production mirror, GEA-NIRO®, Denmark) was used for the process. This spray dryer had the drying capacity of 20 L h^{-1}. Three litres of blackberry and raspberry juices were used for the spray drying process of each treatment. The operational conditions of the spray drying process were inlet and outlet drying air temperature of 180–80 °C. In all the experiments the spray dryer feed flow, atomizer speed and airspeed were kept constant at 20 kg h^{-1}, 28 000 rpm and 24 m s^{-1}, respectively. The agave fructans and maltodextrin concentration as carrier agents were added at 5, 7.5 and 10% w/v of fruit extract.

Yield

Spray drying yield was evaluated by the ratio (expressed as percentage) between the weight of the powder after drying and the weight of all solids in the dispersion (solid dissolves of the 10 °Brix juice, plus added solids of the carrier agent) (Goula & Adamopoulos 2010).

Particle morphology

The morphology of the microcapsules was evaluated by using a JEOL JSM-5910LV scanning electron microscope (JEOL, USA) operating at 30 kV and coating the gold samples by sputtering prior to analysis.

Stability diagram

Particle temperature and glass transition temperature. Particle temperature was measured with the aid of an infrared thermometer (Extech 42529; Extech Instruments®, China). Glass transition temperature (Tg) of all spray-dried powders was determined us-

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ing a differential scanning calorimeter DSC 6 (Perkin-Elmer 10299; Perkin-Elmer®, USA). Indium and zinc (Perkin-Elmer® standards) were used for temperature and heat flow calibration. The samples were cooled to desired temperature (–10 °C) by fast cooling to achieve temperature equilibrium. Five to ten mg of blackberry and raspberry powders were scanned in hermetically sealed 50 µL DSC aluminium pans. The tests were conducted from –10 °C to 100 °C with a heating rate of 10 °C min^{-1}. In addition, the model proposed by Gordon and Taylor (Ozmen & Langrish 2002) was used to determine the Tg in food powders (Farias-Cervantes et al. 2016) and T_{sticky} was calculated by adding 30 °C to the Tg (Tg + 30).

Sorption isotherms and stability diagrams. The equilibrium moisture content of the powders of blackberry and raspberry was determined by the gravimetric technique (Goula et al. 2008). Sulphuric acid solutions (50, 60, 70, 80 and 90%) were used to maintain the relative humidity inside the desiccator. The samples were allowed to equilibrate until there was no weight change. Once equilibrium was reached, the water activity and moisture content were measured. The model used for the determination of equilibrium moisture was the GAB (Guggenheim-Anderson-DeBoer) model. The experimental data fit the GAB (Guggenheim-Anderson-DeBoer) equation by linear regression (Shrestha et al. 2007). The determination of moisture content was based on the AOAC method (Association of Official Analytical Chemists 1990). Measurement of water activity (a_w) was carried out using a water activity meter (AquaLab read; AQUALAB®, USA) at room temperature.

Statistical analysis

All experiments were conducted in triplicate. the data were analysed using Infostat software 2008 (InfoStat, Argentina). Means and standard deviations were calculated. Analysis of variance (ANOVA) with $P < 0.05$ and Tukey's multiple range test were performed to find significant differences.

RESULTS AND DISCUSSION

Yield. The powder yield varied with the carrier agent used and the berry juice (Table 1). The yield of blackberry powder with agave fructans as carrier agent showed values from 52.2 to 89.7%, increasing in the same direction as the carrier agent in the emulsion. However, with maltodextrin, the yield varied from 64.3 to 78.3%. In this case, no clear relation between the carrier agent proportion and powder yield was observed. The powder yields for raspberry juices were lower than for blackberry and did not show any effect of the carrier agent. With agave fructans as a carrier agent the yield values ranged from 33.3 to 67.6% and with maltodextrin they were from 52.2 to 66.7%. In raspberry juice differences in yield between the two carrier agents were observed only in the lower concentration. These results were similar to the results reported by Du et al. (2014) and Ortiz-Basurto et al. (2017). A similar effect of both carrier agents was observed, however the agave fructans got at the highest yields only for blackberry. This behaviour shows the potential use of agave fructans as a carrier agent due to high solids content and better encapsulation that is related to the increase of Tg of the amorphous fractions in mixtures of the extracts that are rich in low molecular sugar. Also, because maltodextrins have a low emulsifying capacity, it is necessary the use of blends with other materials (Silva et al. 2014). The results with maltodextrin were similar to the reported results by Du et al. (2014) for the spray drying of kaki pulp using maltodextrin and different carrier agents. However, it was also observed that there is a small difference between agave fructans and maltodextrin as carrier agents in raspberry powder, even when adding 5% of maltodextrin, the yield is higher than in agave fructans (Table 1).

Particle morphology. The examination of the SEM (scanning electron microscope) micrographs of particles of polydisperse powder sample showed the particle size of blackberry microencapsulated with agave fructans ranging from about 5 to 50 µm, blackberry microencapsulated with maltodextrin with the particle size from 5 to 60 µm, the particle size of raspberry microencapsulated with agave fructans ranging from 10 to 50 µm, and raspberry microencapsulated with maltodextrin from 15 to 50 µm (Figure 1). These particles have the form of a sphere with smooth walls and structure agglomerations. Some authors have suggested that agglomeration occurs due to static electricity effects and Van der Waals force. The agglomeration also occurs because the short-chain polysaccharides are more hygroscopic than long-chain polysaccharides as they contain more hydrophilic groups. So, the DE (dextrose equivalent) maltodextrin and branches of agave fructans can expose these hydrophilic groups and cause agglomeration (Cai & Corke 2000).

Stability diagrams. The moisture content of microencapsulated blackberry juice with agave fructans ranged from 6.4 to 6.9%; with maltodextrin the values ranged from 6.5 to 7.9%. For raspberry juice with

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Table 1. Possible stickiness of particles during and after spray drying of blackberry and raspberry juices with different carrier agents maltodextrin and agave fructans

Juice	Carrier agent	Carrier agent concentration (%)	Yield* (%)	Final moisture* (%)	Final water activity (a_w)*	Tg–T_{sticky} (°C)	Final particle temperature (°C)	Sticky in spray drying chamber	Sticky in storage
Blackberry	agave fructans	5	52.2 ± 0.5^{f}	6.9 ± 0.03^{b}	0.19 ± 0.01^{f}	41.94–71.94	44^{a}	no agglomeration	possible
		7.5	59.1 ± 0.5^{e}	6.4 ± 0.04^{cb}	0.28 ± 0.08^{c}	46.22–76.22	44^{a}	no agglomeration	possible
		10	89.7 ± 0.5^{a}	6.9 ± 0.01^{b}	0.24 ± 0.08^{d}	35.47–65.47	33^{c}	no agglomeration	no agglomeration
	maltodextrin	5	78.3 ± 0.5^{b}	6.8 ± 0.56^{b}	0.38 ± 0.01^{b}	46.98–76.98	45^{a}	possible	possible
		7.5	64.3 ± 0.5^{d}	7.9 ± 0.57^{a}	0.37 ± 0.01^{b}	46.01–76.01	44^{a}	possible	possible
		10	71.9 ± 0.5^{c}	6.5 ± 0.51^{c}	0.38 ± 0.01^{b}	49.81–79.81	39^{b}	no agglomeration	no agglomeration
Raspberry	agave fructans	5	33.3 ± 0.5^{g}	7.7 ± 0.29^{a}	0.23 ± 0.04^{d}	55.98–85.98	44^{a}	no agglomeration	no agglomeration
		7.5	67.6 ± 0.5^{d}	7.7 ± 0.23^{a}	0.23 ± 0.03^{d}	53.63–83.63	43^{a}	no agglomeration	no agglomeration
		10	65.8 ± 0.5^{d}	7.0 ± 0.12^{a}	0.29 ± 0.04^{c}	41.81–71.81	44^{a}	no agglomeration	no agglomeration
	maltodextrin	5	52.2 ± 0.5^{f}	7.7 ± 0.46^{a}	0.21 ± 0.01^{e}	51.34–81.34	42^{ba}	no agglomeration	no agglomeration
		7.5	66.7 ± 0.5^{d}	6.3 ± 0.43^{b}	0.42 ± 0.01^{a}	48.45–78.45	40^{b}	no agglomeration	no agglomeration
		10	65.8 ± 0.5^{d}	6.1 ± 0.47^{b}	0.45 ± 0.01^{a}	45.53–75.53	43^{a}	possible	no agglomeration

*Values are measured in mean ± standards deviation (SD); values with different letters (a, b, c) imply significant differences ($P < 0.05$); Tg – transition temperature; T_{sticky} – calculated by adding 30 °C to the Tg (Tg + 30)

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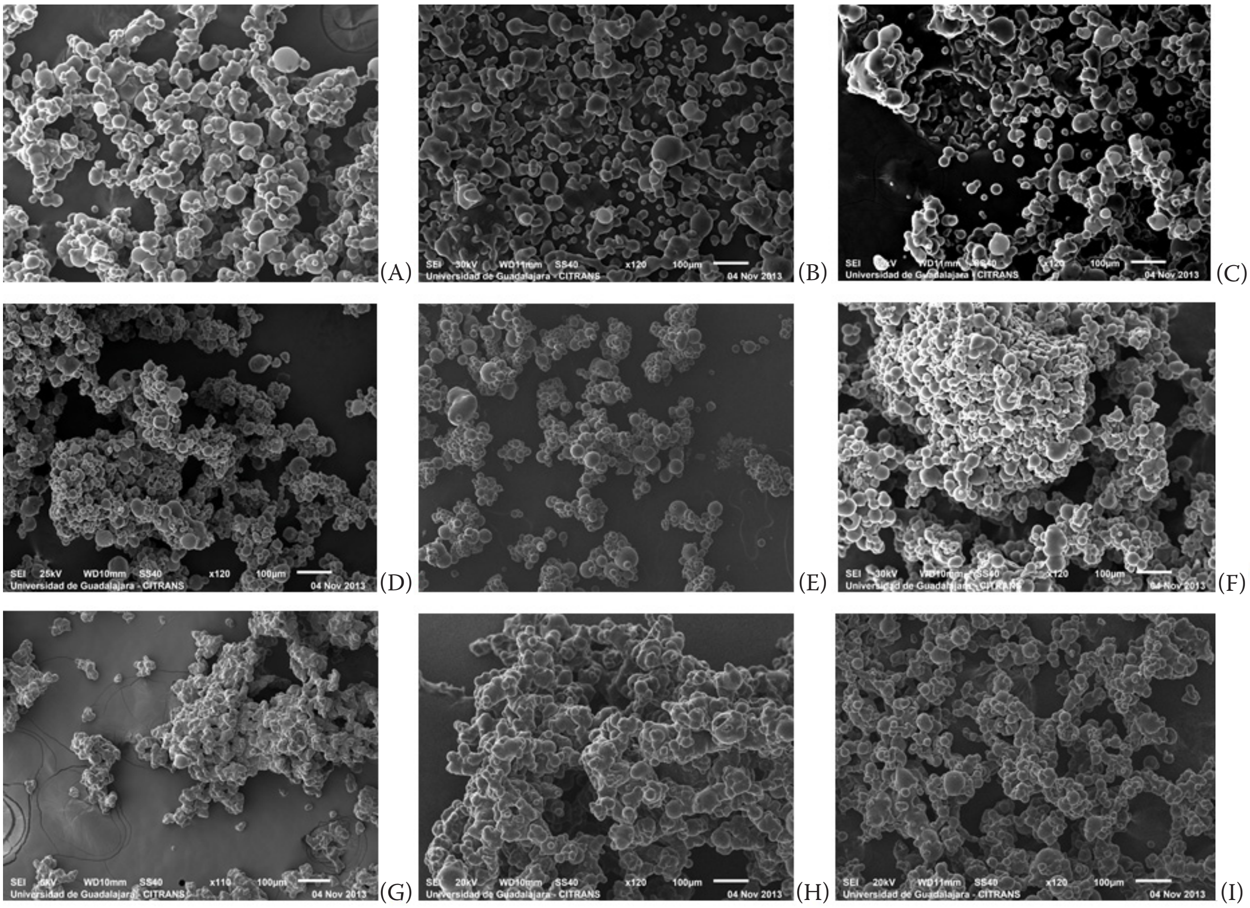

Figure 1. SEM micrographs, particle shapes: (A), (B) and (C) powders of blackberry juice with 5, 7.5 and 10% of maltodextrin concentration as a carrier agent, respectively; (D), (E) and (F) powders of blackberry juice with 5, 7.5 and 10% of agave fructans concentration as a carrier agent, respectively; (G) powders of raspberry juice with 7.5% concentration of maltodextrin as a carrier agent; (H) and (I) powders of raspberry juice with 7.5 and 10% of agave fructans concentration as a carrier agent, respectively

SEM – the scanning electron microscope

agave fructans it ranged from 7.02 to 7.73%, with maltodextrin the moisture varied from 6.1 to 7.7%. No significant differences in the moisture content of dried juices were observed (Table 1). Grabowski et al. (2006) observed that higher concentrations of solids in the feeding emulsion corresponded to low moisture content in powders of mashed potatoes. Comparing the results obtained in this study with reports by Syamaladevi et al. (2012) and Du et al. (2014), in the spray drying of persimmon pulp, acai pulp and red raspberry moisture values above those reported were shown. However, the results are similar to those reported by the authors like Goula & Adamopoulos (2010), León et al. (2010) and Fang et al. (2012) for the spray drying of milk protein concentrate, orange juice and cactus mucilage, respectively. The results for water activity are shown in Table 1, where it can be observed that water activity values of blackberry juice with agave fructans ranged between 0.18 and 0.29 and with maltodextrin from 0.37 to 0.38; in raspberry the values were 0.23 to 0.29 when agave fructans were used and with maltodextrin the values ranged from 0.21 to 0.45. Agave fructans as a carrier agent for juices of blackberry and raspberry showed lower water activity than maltodextrin when used as a carrier agent. These results are due to chemical structural differences in maltodextrin, which allow having different binding capacities with water (Du et al. 2014). When comparing the results between raspberry and blackberry, it was noted that raspberry powder presented the highest water activity (0.45) with maltodextrin as a carrier agent, indicating that the microencapsulated raspberry has a high content of water available for microbial growth and biochemical reactions. This result is probably due to that the amount

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of maltodextrin added to the juice was not high enough to generate a stable powder, so it is recommended increasing the concentration of two carrier agents. The results of water activities were like those reported by other authors (Tonon et al. 2009; Ferrari et al. 2013), except for the treatment of raspberry with maltodextrin. Determining the glass transition temperature (Tg) and the sorption isotherms of microencapsulated powders are very important to evaluate whether a drop/particle is susceptible to adhesion and cohesion during and after processing. Stability diagrams predict and define the region of stickiness offering information for the controlling operating conditions of spray drying process and preventing storage agglomeration. The stability diagram for blackberry powder is shown in Figure 2, Figure 3 and Table 1, where the temperatures of the particles of the treatments of blackberry powder with agave fructans and maltodextrin were between 33 to 44 °C and 39 to 35 °C, respectively. During spray drying, the stickiness region corresponds to water activity values between 0.32 and 0.68. This shows that the storage temperature of blackberry powder should be lower than 52 °C. Therefore, in Table 1 agglomerated treatments during and after spray drying and non-agglomerated ones are shown, and increasing the concentration of two carrier agents is recommended. The stability diagram for raspberry powder is shown in Figure 4, Figure 5 and Table 1, where one can see that the region of stickiness for the powders with particle temperatures between 40 and 44 °C is located to water activity values between 0.5 and 0.78. This shows that the storage temperature of the raspberry powder should be less than 52 °C. The temperatures of the particles of all blackberry powders with agave fructans and maltodextrin fell outside the region of stickiness demonstrating that the high yields were achieved and showing that the losses obtained were not adhering to the walls of the dryer but the effect

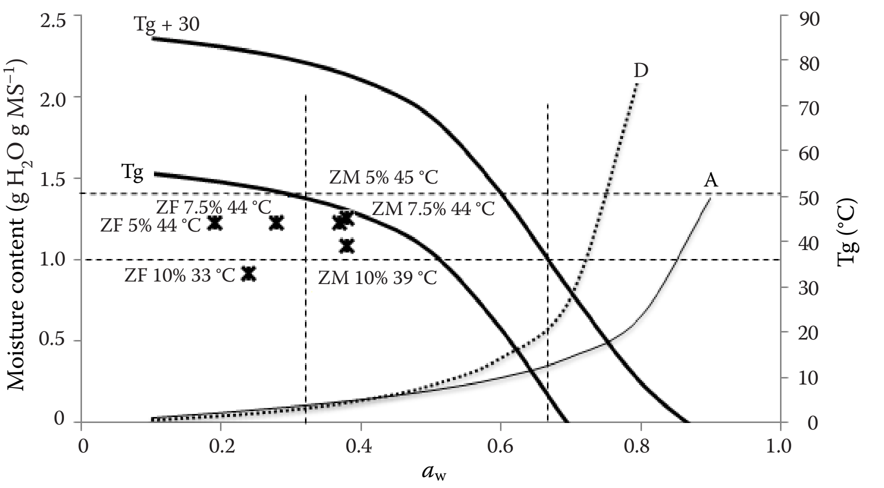

Figure 2. Stability diagrams representing the water activity (a_w), moisture content (g H_2O g MS^{-1}), glass transition temperature (Tg), particle temperature and stickiness point during spray drying of blackberry with maltodextrin (ZM) and agave fructans (ZF)

D – desorption isotherm; A – adsorption isotherm; Tg – line that represents the Gordon-Taylor model; Tg + 30 – the line that represents the Gordon-Taylor model with the sum of 30 °C; dotted lines limit the stickiness point

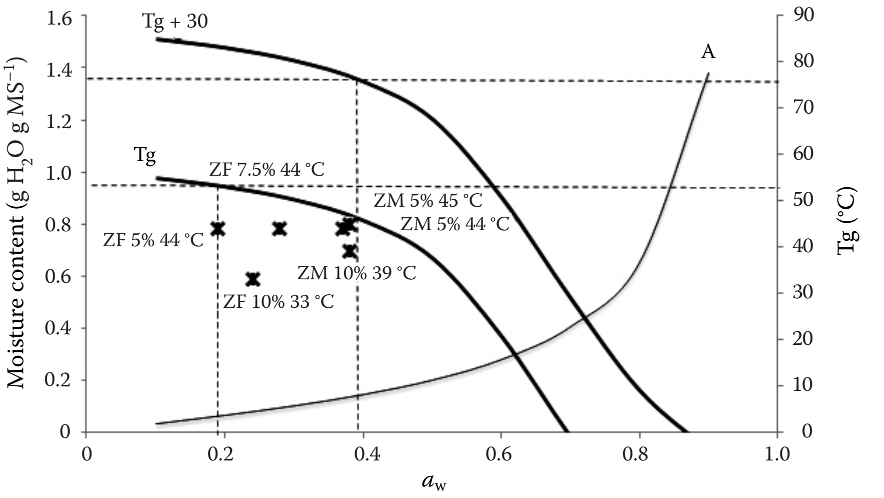

Figure 3. Stability diagrams representing the better conditions for storage of blackberry with maltodextrin (ZM) and agave fructans (ZF) as carrier agents

A – adsorption isotherm; Tg – line that represents the Gordon-Taylor model; Tg + 30 – the line that represents the Gordon-Taylor model with the sum of 30 °C; dotted lines limit the better conditions for storage

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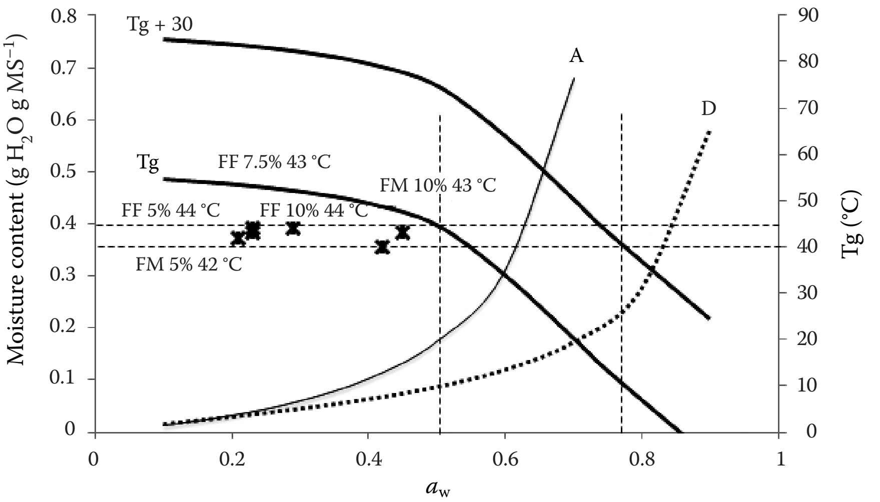

Figure 4. Stability diagrams representing the water activity (a_w), moisture content (gH_2O gMS^{-1}) glass transition temperature (Tg), particle temperature and stickiness point during spray drying of raspberry with maltodextrin (FM) and agave fructans (FF)

D – desorption isotherm; A – adsorption isotherm; Tg – line that represents the Gordon-Taylor model; Tg + 30 – the line that represents the Gordon-Taylor model with the sum of 30 °C; dotted lines limit the stickiness point

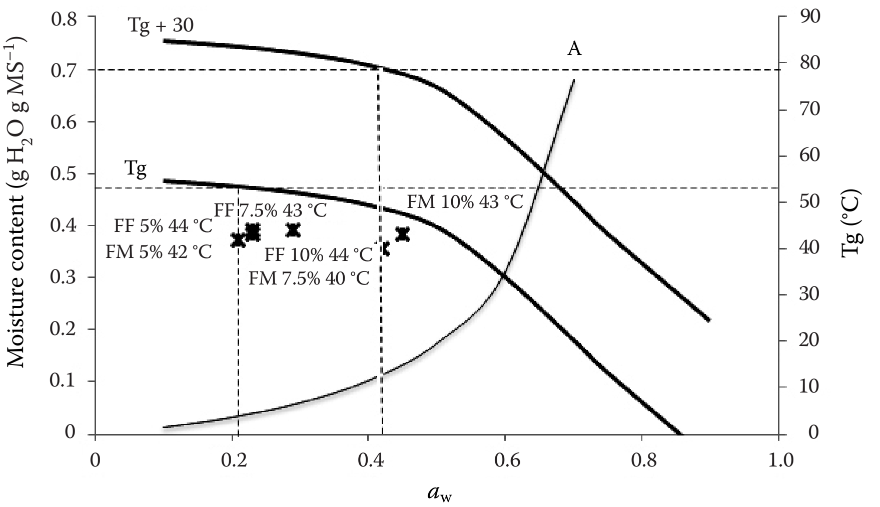

Figure 5. Stability diagrams representing the better conditions for storage of raspberry with maltodextrin (FM) and agave fructans (FF) as carrier agents

A – adsorption isotherm; Tg – line that represents the Gordon-Taylor model; Tg + 30 – the line that represents the Gordon-Taylor model with the sum of 30 °C; dotted lines limit the better conditions for storage

of other operating conditions. However, some blackberry powders with agave fructans and maltodextrin have a high possibility of agglomeration during storage if they are not stored appropriately.

CONCLUSION

The use of the carrier agents did not show any significant differences in the stability of the particles obtained in blackberry and raspberry powders. Using agave fructans as a carrier agent, the highest yield of 89.7% and 67% was obtained for blackberry and raspberry, respectively. Micrographs show a more stable powder for blackberry than raspberry with maltodextrin as a carrier agent, because with agave fructans it presented more agglomeration. Stability diagrams allow observing and evaluating the efficiency of the spray drying process. During the spray drying operation the particle temperature should not exceed 38 °C and 40 °C for blackberry and raspberry, respectively, to obtain more stable powders. For better preservation of the powders, they should be kept between 0.2 and 0.38 water activity (a_w) at a maximum temperature of 52 °C according to the stability diagrams obtained. It is suggested to continue with shelf life studies of the powders.

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