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A new technique for spray drying orange juice concentrate

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ABSTRACT

A new technique for spray drying concentrated orange juice using dehumidified air as drying medium and maltodextrin as drying agent was developed. A pilot-scale spray dryer was employed for the spray drying process. The modification made to the original design consisted in connecting the dryer inlet air intake to an absorption air dryer. 21 DE, 12 DE, and 6 DE maltodextrins were used as drying agents. Concentrated orange juice was spray dried at inlet air temperatures of 110, 120, 130, and 140 °C and (concentrated orange juice solids)/(maltodextrin solids) ratios of 4, 2, 1, and 0.25. Data for the residue remaining on the walls were gathered and the powders were analyzed for moisture content, bulk density, rehydration, hygroscopicity, and degree of caking. The combination of maltodextrin addition and use of dehumidified air was proved to be an effective way of reducing residue formation.

Industrial relevance: Orange juice powder has many benefits and economic potentials over its liquid counterparts and provides a stable, natural, easily dosable ingredient, which generally finds usage in many foods and pharmaceutical products such as flavoring and coloring agents. However, the dehydration of orange juice is not a simple task. Thus, the objective of this study was to develop a new technique for spray drying orange juice using dehumidified air as drying medium and maltodextrin as drying agent.

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1. Introduction

Spray drying is the transformation of feed from a liquid or slurry form to a dry powder. The feed is atomized into a chamber where the resulting spray mixes with hot gas, which evaporates the liquid component of the spray leaving dried particles. One of the main indices of a spray dryer performance is product recovery. Material loss in a spray drying system is due mostly to the adhesion of droplets to the wall of the apparatus (Maa, Nguyen, Sit, & Hsu, 1998; Masters, 1979). Retention of product on the chamber wall over lengthy time is undesirable. It affects product quality, as deposits can become scorched and when dislodged, mix in and contaminate the entire product. Furthermore, it is not cost-effective due to a more frequent shut down of the dryer for cleaning.

The products to be spray dried can be categorized into two major groups: non-sticky and sticky products. Sticky products are generally difficult to spray dry. During the drying process they may remain as syrup or stick on the dryer wall, or form unwanted agglomerates in the dryer chamber and conveying system (Bhandari & Howes, 2005; Hennings, Kockel, & Langrish, 2001). The problem of powder stickiness is mainly due to the low glass transition temperature (Tg) of the low molecular weight sugars present in such products, essentially sucrose, glucose, and fructose (Bhandari, Senoussi, Dumoulin, & Lebert, 1993; Roos, Karel, & Kokini, 1996).

Fruit juice powders have many benefits and economic potentials over their liquid counterparts such as reduced volume or weight, reduced packing, easier handling and transportation, and much longer shelf life. Besides, their physical state provides a stable, natural, and easily dosable ingredient, which generally finds usage in many foods and pharmaceutical products such as flavoring and coloring agents (Shrestha, Ua-arak, Adhikari, Howes, & Bhandari, 2007). However, the dehydration of fruit juices is not a simple task. The low Tg of the main juice components (low molecular weight sugars and organic acids) as well as their high hygroscopicity, low melting point, and high water solubility result in highly sticky product when spray dried.

Various methods capable of producing a free-flowing fruit juice powder have been proposed: addition of drying aids (maltodextrins, glucose, soybean protein, sodium chloride, and skim milk powder) (Adhikari, Howes, Bhandari, & Truong, 2003, 2004; Bhandari et al., 1993; Bhandari, Datta, Crooks, Howes, & Rigby, 1997; Brennan, Herrera, & Jowitt, 1971; Chegini & Ghobadian, 2005; Chegini, Khazaei, Ghobadian, & Goudarzi, 2008; Jaya & Das, 2004; Lazar, Brown, Smith, Wong, & Lindquist, 1956; Papadakis, Gardeli, & Tzia, 2006; Quek, Chok, & Swedlund, 2007; Rao & Gupta, 2002; Roustapour, Hosseinalipour, & Ghobadian, 2006; Shrestha et al., 2007; Tsiouroullis, Flink, & Karel, 1976), scraping of dryer surfaces (Karatas & Esin, 1994),...
cooling of the drying chamber walls (Chegini & Ghobadian, 2005; Chegini et al., 2008; Gransmith, 1971; Jayaraman & Das Gupta, 1995; Spicer, 1974), and admission of atmospheric air near the chamber bottom, allowing transport of the powder to a collector having a low humidity atmosphere (Ponting, Stanley, & Copley, 1973).

The major components of orange juice such as fructose, glucose, and citric acid have very low Tg of 5.31, and 16 °C, respectively, in a pure, dry state, which decrease drastically when moisture is absorbed. Due to this characteristic, the spray drying of orange juice is complex. Shrestha et al. (2007) produced orange juice powder by spray drying a mixture of juice and maltodextrin with DE of 6 at 160 °C. Tsourou and citric acid have very low concentration. Chegini and Ghobadian (2005) carried out spray drying orange juice using dehumidified air. The major limitations of the use of drying aids are the subsequent change in the product properties and the cost. As far as the cooling of the drying chamber walls is concerned, the cool wall will be favorable to minimize the thermoplastic particles from sticking, as the wall will be cold enough to cool and solidify the outer surface of the thermoplastic particles coming in contact. This method, however, was found to improve the process but not to resolve the problem. The reason is that the cold chamber wall will also cool the surrounding environment and cause an increase in the relative humidity of the air close to the wall surface.

Considering the difficulty in obtaining orange juice powder, the objective of this study was to develop a new technique for spray drying orange juice concentrate using dehumidified air as drying medium and maltodextrin as drying agent.

2. Materials and methods

2.1. Raw materials

Concentrated orange juice with a total solids mass concentration of 62.0±0.3%, containing 43.1±0.5 g/100 mL sugars and 4.3±0.1 g/100 mL citric acid, obtained from a local manufacturer, was used. 21 DE, 12 DE, and 6 DE maltodextrins (Glucidex®, Roquette, France) were used as drying agents. Taking into account the moisture content of orange juice concentrate and of maltodextrin powders (2.5, 3.5, and 4.5%, respectively), solutions with the desired total solids concentration and the desired ratio of (concentrated orange juice solids)/(maltodextrin solids) were prepared. These solutions were subsequently used as feeds to the spray dryer.

2.2. Spray drying

A pilot-scale spray dryer (Buchi, B-191, Buchi Laboratoriums-Technik, Flawil, Switzerland) with concurrent regime and a two-fluid nozzle atomizer was used for the spray drying process. The atomizer had an inside diameter of 0.5 mm and used compressed air with a flow rate that was controlled by a variable area flow meter. Feed was metered into the dryer by means of a peristaltic pump. Inlet drying air, after passing through an electrical heater, flowed concurrently with the spray through the main chamber. The main chamber was made of thick transparent glass and had an inside diameter of 10.5 cm and a total height of 52.5 cm. The distance between the tip of the atomizer and the axis of the side exit tube was 34.9 cm. The bottom of the chamber is cone shaped and makes an angle of 60° with the walls. A cyclone air separator/powder recovery system was used. Dried powder samples were collected from the base of the cyclone. The design of the dryer is such that the outlet air temperature, contrary to the inlet temperature, cannot be set with a temperature regulator, but results from a combination of the inlet temperature, the aspirator setting, the pump setting, as well as the concentration of the feed. The modification made on the original design consisted of connection of the spray dryer inlet air intake nipple with an air drying unit by a flexible plastic air duct. The compressed air was also dehumidified before its supply to the two-fluid nozzle. An Ultrapac 2000 adsorption dryer (Model 00005, Ultrafilter International AG, Haan, Germany) with two desiccant cartridges was used to dry air down to 0.01 g of water per kg of dry air.

Forty-eight different experiments were conducted in triplicate. The controlled parameters were the DE of the maltodextrin (DE), the ratio (concentrated orange juice solids)/(maltodextrin solids) (o:m), and the inlet air temperature (Ti). 21 DE, 12 DE, and 6 DE maltodextrins were used as drying agents. Orange juice concentrate was spray dried at inlet air temperatures of 110, 120, 130, and 140 °C (±1°C) and (concentrated orange juice solids)/(maltodextrin solids) ratios of 4, 2, 1, and 0.25. In all experiments the atomizer pressure, the feed rate, the feed solids concentration, and the feed temperature were kept at 5.0±0.1 bar, 1.8±0.1 g/min, 35.0±0.2%, and 32.0±0.5 °C, respectively. Moreover, in a previous work, it was found that the lowest wall deposition rate was achieved at a drying air flow rate of 22.8±0.2 m³/h and a compressed air flow rate of 800±20 L/h (Goula & Adamopoulos, 2005a). These conditions were used for all the experiments reported here.

2.3. Spray dryer performance

The weights of the drying chamber and the receiving vessel for powder were determined before and after spray drying by an electronic balance with an accuracy of 10⁻³ g. Residue yield (nₐ) was determined by dividing the weight of solid mass collected in the chamber with the total amount of solid mass to be spray dried.

2.4. Glass transition temperature measurement

Samples of orange juice powder of about 1 g (±0.01 g), produced by spray drying of orange juice concentrate at 140 °C without maltodextrin, were conditioned at 25 °C using sulphuric acid solutions to maintain the water activity at ten levels between 0.04 and 0.95, according to sorption isotherm methodology (Al-Muhtaseb, McMinn, & Magee, 2004). After equilibrium was reached, samples of about 10 mg were taken for differential scanning calorimetry (DSC) analysis and the remaining material was analyzed for moisture content by drying in a vacuum oven at 70 °C until consecutive measurements were done in triplicate. The glass transition temperature was determined by DSC, with a differential scanning calorimeter (Perkin-Elmer Pyris 1, PerkinElmer Life and Analytical Sciences Inc., Wellesley, MA) supplied with proper software. The rate of thermal scanning was carried out in the following order: 1) isothermal at −80 °C for 1 min; 2) heating at 10 °C/min from −80 °C to a temperature just over the predetermeed apparent Tg; 3) cooling at 50 °C/min to −80 °C; 4) heating at 10 °C/min from −80 to 60 °C. The midpoint of the glass transition was considered as the characteristic temperature of the transition. All measurements were done in triplicate.

The glass transition temperature of a binary solid–water mixture is strongly dependent on the water concentration. Once the moisture content is known, the Tg can be determined using the model proposed by Gordon and Taylor (Ozmen & Langrish, 2002):
2.5. Analysis of powders

Moisture: The moisture content was determined by drying in a vacuum oven at 70 °C until consecutive weighings, made at 2 h intervals, gave less than 0.3% variation. The moisture content was expressed in terms of the percent wet basis (w.b.) (100 × kg water/kg wet material).

Bulk density: 2 g of powder were transferred to a 50 mL graduated cylinder. The bulk density was calculated by dividing the mass of the powder by the volume occupied in the cylinder (Goula, Adamopoulos, & Kazakis, 2004; Goula & Adamopoulos, 2005b; Goula & Adamopoulos, 2008b).

Rehydration: The rehydration of the powder was carried out by adding 2 g of the material to 50 mL of distilled water at 26 °C. The mixture was agitated in a 100 mL low form glass beaker with a Heidolph magnetic stirrer (No 50382, MR 82, Heidolph Instruments GmbH & Co. KG, Schwabach, Germany) at 892 rpm, using a stirring bar with a size of 2 mm × 7 mm. The time required for the material to be completely rehydrated was recorded (Goula & Adamopoulos, 2005b; Goula & Adamopoulos, 2008b).

Hygroscopicity: About 1 g of powder was spread evenly on Petri dishes (9 cm diameter) to allow for a high surface area between humid air and powder. Samples of each powder in the dishes were placed in desiccator under the following conditions: 23 °C and 76% relative humidity using HNO3 solution. A 10 min interval was selected to get the kinetics of moisture sorption. The gain in weight of the samples was considerably lower after 90 min. Although hygroscopicity is based on the equilibrium moisture content, to compare hygroscopicities, the weight increase per gram of powder solids after being subjected to the atmosphere with relative humidity of 76% for 90 min was determined (Goula, Adamopoulos, & Kazakis, 2004; Goula & Adamopoulos, 2008b).

Degree of caking: After the determination of hygroscopicity, the wet sample was placed in a vacuum oven at 70 °C until consecutive weighings, made at 2 h intervals, gave less than 0.3% variation. After cooling, the dried sample was weighed and transferred into a sieve of 500 μm size. The sieve was then shaken for 5 min in a shaking apparatus. The weight of the powder remaining in the sieve was measured. The degree of caking was calculated as (Jaya & Das, 2004): 

\[ CD = \frac{100 \times a}{b} \]  

where CD is the degree of caking (%), a is the amount of the powder remained on the sieve after sieving, and b is the amount of the powder used in sieving.

All analyses were done in triplicate and the averages of these triplicate measurements were recorded. Additional determinations were carried out if the single values from the triplicates deviated by more than ±1.5% from the triplicate mean.

2.6. Statistical analysis

The data were analyzed using the statistical software MINITAB (Release 13.32). Regression analysis was used to fit a full second order polynomial, reduced second order polynomials, and linear models to the data of each of the variables evaluated (response variables). F values for all reduced and linear models with an \( R^2 \geq 0.70 \) were calculated to determine if the models could be used in place of full second order polynomials to predict the response of a variable to the independent variables. The F value was calculated as:

\[ F = \frac{(SSR_r - SSR_f) / (K - q)}{SSE_f / (N - K - 1)} \]  

where SSR is the sum of squares due to regression, SSE is the sum of squares due to error, N is the number of measurements, K is the number of terms of the full model, q is the number of terms of the reduced model, and the subscripts f and t refer to the full and the reduced model, respectively.

The F value indicates no significant difference between the reduced second order polynomial or linear model and its corresponding full model (p < 0.05) if:

\[ F < F_{0.05, K - q, N - K - 1} \]  

where \( F_{0.05, K - q, N - K - 1} \) is the value of the F-distribution with \( K - q \) and \( N - K - 1 \) degrees of freedom.

The reason that a subset of variables rather than a full set would be used is because the subset model may actually estimate the regression coefficients and predict future responses with smaller variance than the full model using all predictors. Typically, \( R^2 \) always increases with the size of the subset. Therefore, \( R^2 \) is most useful when comparing models of the same size. The square root of mean square error (\( S' \)) and the Mallows' Cp statistic (\( Cp \)) can be used as comparison criteria to compare models with different numbers of predictors. A good model should have a high \( R^2 \), a small \( S' \), and a Mallows' Cp statistic close to the number of predictors contained in the model.

3. Results and discussion

3.1. Glass transition temperature

Values of \( T_g \) obtained for the orange juice powders produced by spray drying of orange juice concentrate at 140 °C without maltodextrin and conditioned to maintain the water activity at ten levels between 0.04 and 0.95 are shown in Fig. 1. As can be drawn from Fig. 1, \( T_g \) decreased by increasing moisture content due to the plasticizing effect of water. This plasticizing activity may be based on the weakening of hydrogen bonds and dipole–dipole intra- and inter-macromolecular interactions due to the shielding of these mainly attractive forces by water molecules. The Gordon and Taylor model was fitted to the experimental points using \( T_g = -135 °C \), with the following parameters calculated by non-linear regression: \( T_g = 5.7 °C \) and \( k = 0.82 \), with \( R^2 = 0.99 \). Fig. 1 shows the curve predicted by the Gordon and Taylor model. The calculated k value was similar to those reported for fruits and sugar solutions (Boonyai, Bhandari, & Howes, 2005).
In a previous work, the glass transition temperatures of 21 DE, 12 DE, and 6 DE maltodextrins conditioned at various water activities were determined by differential scanning calorimetry (Goula & Adamopoulos, 2008a). A strong plasticizing effect of water was found with a large reduction of Tg when the moisture content increased. In addition, low-dextrose equivalent maltodextrins were found to give higher glass transition temperatures than high-DE maltrins at the same moisture contents. These data were satisfactory correlated by the Gordon and Taylor model and the obtained values of Tg, and k were 167.9 °C and 0.85, 159.6 °C and 0.84, and 146.4 °C and 0.83 for DE of 6, 12, and 21, respectively.

According to Adhikari et al. (2004), the Tg of multi-component solid mixtures, such as orange juice concentrate–maltodextrin mixture, is determined using a mass weighted mean rule. The multi-component mixture is assumed to be composed of n individual binary solid–water mixtures, where n is the number of solid components. Firstly, the moisture dependence of Tg for each binary solid–water mixture is determined. Finally, the solids are assumed to be perfectly mixed and the Tg of the multi-component mixture is computed as a mass weighted mean on a water free basis:

\[ T_{g_m} = \sum_{i=1}^{n} \left( T_{g_i} \times x_i \right) \]  

where \( T_{g_m} \) represents the Tg for the multi-component mixture including water, \( T_{g_i} \) is the Tg of binary solid–water mixtures, such as maltodextrin–water, determined by Eq. (1), and \( x_i \) is the mass fraction of an individual solid component on a water free solids basis.

Fig. 2 shows the calculated values of Tg against moisture content and (concentrated orange juice solids)/(maltodextrin solids) ratio for the concentrated orange juice solids–maltodextrin 6 DE–water mixture. Shrestha et al. (2007), who spray dried orange juice concentrate with maltodextrin, reported a measured Tg value of anhydrous orange juice:maltodextrin 6 DE (50:50) powder equal to 66.4 °C whereas an increase in maltodextrin level from 50 to 60 parts resulted in a Tg value of 86.4 °C. The lower values of Tg obtained in the work of Shrestha et al. (2007) may be due to the higher sugars and citric acid content of the orange juice concentrate, about 48–51% and 5.25–6.25%, respectively, compared to 43.1% and 4.3% in the concentrate used in this work. Glass transition temperature is known to decrease with decreasing molecular weight (Kasapis, 2005), in such a way that incorporated sugars and citric acid could have caused the observed decrease in Tg. In addition, this difference in the Tg values does not seem particularly significant if one considers the uncertainties of the prediction (Eqs. (1) and (5)) and of the experimental measurements. According to Werner, Jones and Paterson (2007), the glass transition occurs over a 10–20 °C range, but usually only a single temperature is quoted as Tg. In addition, sample preparation can lead to discrepancies in Tg data and high-molecular weight polymers also have wide spanning thermal peaks leading to difficulties in pinpointing the Tg.

### 3.2. Residue formation

Residue data were gathered as an indication of the process performance. Fig. 3 shows the achieved values of residue yield, ranging from 5 to 14%, against inlet air temperature and (concentrated orange juice solids)/(maltodextrin solids) ratio for each level of maltodextrin dextrose equivalent. Each data point in the figure represents average values of three replications. The repeatability for \( n_i \) expressed as the average standard deviation of the three replications was 0.2%.

During drying of orange juice concentrate–maltodextrin 6 DE with a o:m value of 4 at 110 °C, the residue yield was about 12%, whereas an increase in maltodextrin level from 0.25 to 0.50 parts resulted in a significant decrease in residue yield of about 17%. Further increase in maltodextrin level from 0.50 to 1 parts and from 1 to 4 parts led to reductions of approximately 20 and 27%, respectively. During drying at 120 °C, a o:m value of 4 led to a residue yield of 12% and increases of maltodextrin level from 0.25 to 0.50, from 0.50 to 1, and from 1 to 4 parts resulted in \( n_i \) reductions of about 16, 20, and 24%, respectively. Finally, during drying at 130 and 140 °C, these percentages were 14, 21, and 24% and 12, 20, and 26%, respectively. Thus, the residue yield decreases with increasing maltodextrin concentration; because the lower the o:m value, the higher the elevation of the Tg of the orange juice concentrate–maltodextrin mixture, as it can be concluded from Fig. 2. However, this decrease is not linear.

This observation is similar to that reported by Shrestha et al. (2007), who mentioned that when orange juice:maltodextrin ratio was 50:50, the residue yield was 78%, much higher than this obtained in this work, whereas an increase in maltodextrin level from 50 to 60 parts resulted in a significant reduction (22%), and a further increase only slightly decreased the wall deposits. In general, several authors mentioned that a increase in the maltodextrin content results in an increase of the recovery of feed solids in the product (Bhandari, Datta, Crooks et al., 1997; Papadakis et al., 2006; Roustapour et al., 2006). However, in this work, the achieved values of residue yield are much lower than those reported, although those authors used similar operating conditions and maltodextrin contents and the products they used have Tg of dry solids similar to this of orange juice. In addition, Chegini and Ghobadian (2005) and Chegini et al. (2008), who spray dried orange juice concentrate:maltodextrin mixtures cooling the drying chamber walls, reported higher values of residue yield when the additive concentrations were similar to these used in this work, whereas only higher drying aids concentrations led to \( n_i \) values close to these obtained with the combination of maltodextrin addition and use of dehumidified air. However, a minimum quantity of drying additives is desirable, as additives change product properties and may increase product cost (Bhandari, Datta, Crooks et al., 1997).

The lower values of \( n_i \) obtained in this work may be due to the lower air temperatures in the drying chamber when dehumidified air is used as the drying medium instead of dehumidified air (Goula and Adamopoulos, 2005a). The extent of stickiness or the consequence on structural change of the powder depends on the difference between the temperature of the product, which is generally approaching the outlet air temperature, and the glass transition.

![Fig. 2. Relationship between glass transition temperature (Tg) and moisture content (X) of concentrated orange juice–maltodextrin 6 DE mixture for various (concentrated orange juice solids)/(maltodextrin solids) (o:m) ratios.](image-url)
as a drying medium enhancing the rapid removal of water contributes to the reduction of residue accumulation.

As it can be seen in Fig. 3, the wall deposits increased by increasing the inlet air temperature. However, the lower the proportion of maltodextrin, the lower the significance of this effect is. Bhandari et al. (1993) and Chegini and Ghobadian (2007) also reported that besides the amount of drying aid, the recovery of a spray dried sugar-rich product is largely affected by the inlet air temperature and the wall depositions increase by increasing the inlet air temperature.

The residue yield also decreases with decreasing the maltodextrin dextrose equivalent, because the lower the maltodextrin DE, the higher its glass transition temperature and, as a consequence, the higher the elevation of the T_g of the orange juice concentrate-maltodextrin mixture. Werner et al. (2007), who used a probe tack test to map the level of stickiness of droplets containing 20 and 40% of maltodextrins 5 DE, 10 DE, and 18 DE, reported that low-DE maltodextrins develop stickiness faster and reach a state of non-adhesion faster than high-DE maltodextrins. For this reason, lower DE maltodextrins are favored as wall deposit-reducing agents during drying.

The best model predicting response of the variable n_r to the process variables was the following:

\[ n_r = 1.94 + 0.0749 \cdot DE + 0.0249 \cdot Ti + 3.42 \cdot (o : m) - 0.408 \cdot (o : m)^2 - 0.00692 \cdot DE \cdot (o : m). \]  

(6)

Eq. (6) has a R^2 value of 0.99, and C_p and S^2 equal to 4.8 and 0.146, respectively.

Various studies have shown that the surface stickiness of an amorphous powder is closely related with the glass transition temperature (Downton, Flores-Luna, & King, 1982; Shrestha et al., 2007). Attempts have also been made to develop a direct relationship between glass transition and stickiness during spray drying process (Bhandari & Howes, 1999). Fig. 4 shows a good linear correlation (Eq. (7)) between the residue yield and the T_g of the powder at the exit of the dryer chamber (T_gp), which is presented in Table 1, but with a different line to the various DE and Ti levels (Eqs. (8) and (9)).

\[ n_r = A \cdot T_gp + B \]  

(7)

\[ A = -0.315 - 2.63 \cdot 10^{-6} \cdot DE + 4.05 \cdot 10^{-3} \cdot Ti - 0.10 \cdot 10^{-6} \cdot DE^2 - 0.16 \cdot 10^{-6} \cdot Ti^2 \]  

(8)

\[ B = 27.60 + 0.0391 \cdot DE - 0.25 \cdot Ti + 0.00113 \cdot Ti^2. \]  

(9)

Eqs. (8) and (9) have a R^2 value of 0.91 and 0.95, respectively, and C_p and S^2 equal to 4.0 and 0.002 and 2.4 and 0.122, respectively.

Thus, it may be possible to use the glass transition temperatures directly to determine the amount of drying aids required. To do this, one needs to calculate the T_g of a multicomponent mixture based on its composition (Eq. (5)). However, according to Bhandari, Datta, Crooks et al. (1997), the behavior of the material (such as T_g) is strongly dependent on its previous history. This questions whether the behavior of the product can be correctly characterized only on the basis of T_g, particularly in the case of rapid dehydration such as in a dynamic spray drying, where number of physical parameters change simultaneously.

Some researchers have proposed, and in some cases shown, that glass transition related flow changes (sticking, crystallization, and collapse) occur at a rate determined by the amount that the T_g has been exceeded by (Paterson, Brooks, Bronlund, & Foster, 2005; Roos & Karel, 1991), whereas others have proposed that these flow changes occur at a particular T_g (Hennings et al., 2001; Ozmen & Langrish, 2002). According to Adhikari et al. (2003), during spray drying, the
A droplet is sticky if its $T_g$ is lower than the droplet temperature ($T_d$) and becomes completely non-sticky when $T_g \geq T_d + 10 \, ^\circ C$. Bhandari, Datta and Howes (1997) reported that during spray drying, product temperature generally approaches the outlet air temperature ($T_o$) and there is a narrow range of outlet air temperature for a successful drying operation. The values of the measured outlet air temperature for each experiment, along with those of the powder moisture content ($X_p$), are listed in Table 1. In Table 1, cells with the superscript a show the experiments where $T_g \geq T_d + 10 \, ^\circ C$ and, thus, the droplets are completely non-sticky and the residue formation is not due to the sticky nature of the product. According to Masters (1979), product formation on the walls falls into two categories. Sticky deposits caused by the nature of the product at the dryer temperature and semi-wet deposits caused by droplets that are not sufficiently dry before hitting the wall. As it can be seen in Table 1, in experiments with $T_g \geq T_d + 10 \, ^\circ C$ and, thus, is difficult for water molecules to diffuse past the larger maltodextrin molecules. This results in less dry droplets hitting the chamber walls.

Fig. 4 shows a good correlation, described by Eq. (10), between the residue yield ($n_r$) and $T_g$ of the powder at the exit of the dryer chamber ($T_{gp}$).

\[ n_r = 0.10 \cdot (T_o - T_{gp}) + 9.31 \quad (R^2 = 0.97). \]  

This observation is similar to that reported by Foster, Bronlund and Paterson (2006), who mentioned that the rate of stickiness development in amorphous maltose and glucose/lactose, galactose/lactose, and fructose/lactose mixtures are proportional to the $T - T_g$ and not the individual conditions used to obtain the $T - T_g$.

### 3.3. Powder properties

The moisture content of the orange juice powders varied from 1.9 to 7.0% w.b. Fig. 6 shows the achieved values against inlet air temperature and ($\text{concentrated orange juice solids}/\text{maltodextrin solids}$) ratio for different maltodextrin dextrose equivalents. As shown in Fig. 6, an increase in air inlet temperature leads to a
The greater the temperature difference between the drying medium and the particles, the greater will be the rate of heat transfer into the particles, which provides the driving force for moisture removal. When the drying medium is air, temperature plays a second important role. As water is driven from the particles in the form of water vapor, it must be carried away, or the moisture will create a saturated atmosphere at the particle surface. This will slow down the rate of subsequent water removal. The hotter the air, the more moisture it will hold before becoming saturated.

Moisture content shows an increase with an increase in maltodextrin concentration. This can be attributed to the fact that it is difficult for water molecules to diffuse past the larger maltodextrin molecules (Adhikari et al., 2004). In addition, higher maltodextrin dextrose equivalent causes an increase in powder moisture content. This may be due to the fact that high-DE maltodextrins develop stickiness slower and reach a state of non-adhesion slower than low-DE maltodextrins (Goula & Adamopoulos, 2008a). The stickier a material is, the lower its drying rate.

Fig. 7 shows powder bulk density in relation to inlet air temperature, (concentrated orange juice solids)/(maltodextrin solids) ratio (o:m), and dextrose equivalent values (DE).

As it can be drawn from Fig. 7, increased inlet air temperature causes a reduction in bulk density, as evaporation rates are faster and products dry to a more porous or fragmented structure. Walton (2000) reported that increasing the drying air temperature generally produces a decrease in bulk and particle density, and there is a greater tendency for the particles to be hollow. The former can be caused by particle inflation-ballooning or puffing and is particularly common in skin-forming materials. According to Chegini and Ghobadian (2005), the

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Table 1
Moisture content and $T_g$ of the powder at the exit of the dryer chamber ($X_p$, $T_g$) and outlet air temperature ($T_o$) for each experiment.

<table>
<thead>
<tr>
<th>$\alpha$ (°C)</th>
<th>DE = 6</th>
<th>DE = 12</th>
<th>DE = 21</th>
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<tbody>
<tr>
<td>$T_i$ = 110 °C</td>
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<td></td>
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<td>4.1 28 58</td>
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<td>3.5 51 61</td>
<td>4.4 45 60</td>
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<tr>
<td>1.0</td>
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<td>5.0 67 63</td>
</tr>
<tr>
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<td>5.1 117 66</td>
<td>6.0 105 66</td>
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<td>3.7 29 61</td>
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<tr>
<td>2.0</td>
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<td>3.1 52 64</td>
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</tr>
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<tr>
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<td>3.9 126 70</td>
<td>4.7 118 69</td>
<td>5.6 106 69</td>
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</table>

$^a T_g$ is $\geq T_d + 10°$. 

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Fig. 5. Relationship between residue yield ($n_r$) and $(T_o - T_g + 10)$, for experiments where the deposits are due to the sticky nature of the product.

Fig. 6. Powder moisture content ($X$) at various inlet air temperatures ($T_i$), (concentrated orange juice solids)/(maltodextrin solids) ratios (o:m), and dextrose equivalent values (DE).

Fig. 7. Powder bulk density ($d$) at various inlet air temperatures ($T_i$), (concentrated orange juice solids)/(maltodextrin solids) ratios (o:m), and dextrose equivalent values (DE).
effect of temperature on bulk density depends on its effect on powder moisture content, as a product of higher moisture would tend to have a higher bulking weight caused by the presence of water, which is considerably denser than the dry solid.

Bulk density shows a decrease with an increase in maltodextrin concentration. This effect may be attributed to the fact that maltodextrin addition minimizes thermoplastic particles from sticking. In addition, an increase in maltodextrin concentration may cause an increase in the volume of air trapped in the particles, as maltodextrin is a skin-forming material. According to Kwapinska and Zbicinski (2005), particles of skin-forming spray dried materials often contain air bubbles, which can occur as a result of desorption of air that was initially present in the liquid feed or was absorbed during atomization. Generally, an increase in the volume of trapped air causes a decrease in the apparent density of the particles and this apparent density primarily determines the powder bulk density.

As shown in Fig. 7, an increase in maltodextrin dextrose equivalent leads to an increase in bulk density. This can be attributed to the fact that the higher the maltodextrin DE, the lower its glass transition temperature and, as a consequence, the lower the elevation of the T_g of the orange juice concentrate–maltodextrin mixture is and the more stickier the mixture is (Adhikari et al., 2004; Goula & Adamopoulos, 2008a).

Rehydration results, ranging from 77 to 200 s, are given in Fig. 8. Rehydration ability showed an increase with an increase in inlet air temperature. Increasing the drying air temperature generally produces an increase in particle size (Walton, 2000), and so a decrease in time required for the powder to be rehydrated. Large particles may sink, whereas small ones are dustier and generally float on water, making for uneven wetting and reconstitution.

The effect of maltodextrin dextrose equivalent on powder rehydration depends on its effect on powder moisture content. This can be attributed to the fact that a low-moisture content seems to be associated with fast rehydration (Goula & Adamopoulos, 2008b), since the lower the moisture content the less sticky the powder is and, thus, the higher will be the surface area in contact with the rehydration water. Maltodextrin DE increases lead to an increase in powder moisture content and a decrease in powder rehydration ability. However, increased maltodextrin concentration does not cause a reduction in powder rehydration ability, although it increases its moisture content. This variation may be attributed to the fact that maltodextrin has superior water solubility and is mainly used in process of spray drying due to its physical properties, such as high solubility in water (Cano-Chauca, Stringheta, Ramos, & Cal-Vidal, 2005; Grabowsk, Truong, & Daubert, 2006).

Moisture adsorption of the spray dried powders at 23 °C and 76% relative humidity after 90 min is shown in Fig. 9. Orange juice powder is evidently hygroscopic. Spray dried particles can easily absorb moisture from the surrounding air and, unless necessary precautions are taken, the surface of the powder becomes sticky and powder caking occurs. The high hygroscopicity of orange juice powder points up need for further investigation on its water content and corresponding relationship to stability and shelf life.

According to Roos (1992), physical changes in low-moisture, high-sugar dehydrated powdered foods, including hygroscopicity, are attributable to the glass transition temperature and the higher the powder T_g, the lower its hygroscopicity. Thus, the effect of the process variables on powder hygroscopicity depends on their effect on T_g. Increases in inlet air temperature and maltodextrin concentration and decreases in maltodextrin dextrose equivalent lead to higher powder T_g (Table 1) and, as a result, to lower hygroscopicity. This observation is similar to that reported by other researchers (Jaya & Das, 2004; Phanindrakumar, Radhakrishna, Mahesh, Jagannath, & Bawa, 2005).

Caking degree results, ranging from 5.9 to 24.8%, are given in Fig. 10. The CD values are within the reported desired values for foodstuffs powders, which vary between 5 and 34% (Jaya & Das, 2004). The effect of the process variables on powder caking depends
on their effect on powder hygroscopicity, since baking of high-sugar dehydrated powdered foods can be attributed to moisture absorption. According to Down at al. (1982), water absorbs on particle surfaces forming a saturated solution and thereby making the particles sticky and capable of forming liquid bridges. Thus, increases in inlet air temperature and maltodextrin concentration and decreases in maltodextrin dextrose equivalent lead to lower hygroscopicity and, as a result, to lower baking degree. A similar trend was reported during spray drying of tomato pulp–maltodextrin mixture (Goula & Adamopoulos, 2008b).

Table 2 presents the best model predictions for the orange powder properties in relation to the process variables, inlet air temperature (Ti), (concentrated orange juice solids)/(maltodextrin solids) ratio (o:m), and dextrose equivalent of maltodextrin (DE).

<table>
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<tr>
<th>Property</th>
<th>Model</th>
<th>R²</th>
<th>Cp</th>
<th>S'</th>
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<td>28.6</td>
<td>0.237 + 6.41</td>
<td>0.289</td>
</tr>
</tbody>
</table>

References


Cano-Chauca, M., Stringheta, P. C., Ramos, A. M., & Cal-Vidal, J. (2005). Effect of the temperature and maltodextrin concentration and decreases in maltodextrin dextrose equivalent on their effect on powder hygroscopicity, since caking of high-sugar dehydrated powdered foods can be attributed to moisture absorption. According to Downton et al. (1982), water absorbs on particle surfaces forming a saturated solution and thereby making the particles sticky and capable of forming liquid bridges. Thus, increases in inlet air temperature and maltodextrin concentration and decreases in maltodextrin dextrose equivalent lead to lower hygroscopicity and, as a result, to lower baking degree. A similar trend was reported during spray drying of tomato pulp–maltodextrin mixture (Goula & Adamopoulos, 2008b).

Table 2 presents the best model predictions for the orange powder properties in relation to the process variables, inlet air temperature, (concentrated orange juice solids)/(maltodextrin solids) ratio, and dextrose equivalent of maltodextrin.

4. Conclusions

A new technique for spray drying orange juice concentrate using dehumidified air as drying medium and maltodextrin as drying agent was developed and the effect of maltodextrin type and concentration on the residue formation and the main powder properties was studied. It was found that:

— Residue formation decreases with an increase in maltodextrin concentration and a decrease in inlet air temperature and dextrose equivalent.

— Moisture content decreases with an increase in inlet air temperature and a decrease in maltodextrin concentration and dextrose equivalent.

— Bulk density increases with an increase in dextrose equivalent and a decrease in inlet air temperature and maltodextrin concentration.

— Hygroscopicity increases with an increase in inlet air temperature and maltodextrin concentration and a decrease in dextrose equivalent.

— Hygroscopicity and degree of caking decrease with an increase in inlet air temperature and maltodextrin concentration and a decrease in maltodextrin dextrose equivalent.

The achieved values of residue yield, ranging from 5 to 14%, were much lower than those reported by other researchers, who added maltodextrins to sugar-rich foods to reduce wall depositions problems, although they used similar operating conditions and maltodextrin contents. This difference is due to the lower air temperatures and higher drying rates when dehumidified air is used as the drying medium instead of dehumidified air. In addition, the lower the moisture content, the hygroscopicity, and the baking degree and the higher the density and the rehydration ability, the better the powdered product will be considered. Thus, maltodextrins improved powder hygroscopicity, baking, and rehydration, whereas deteriorated slightly its moisture content and density. However, in experiments conducted using dehumidified air, powder moisture content was much higher and bulk density was much lower. Thus, the combination of maltodextrin addition and use of dehumidified air as drying medium seems to be an effective way of producing a free-flowing orange powder.


