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# Spray drying of nopal mucilage (*Opuntia ficus-indica*): Effects on powder properties and characterization

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ABSTRACT

Aqueous solutions of mucilage were spray dried without the use of carrier agents and the influence of drying conditions on mucilage powder properties was evaluated. The drying parameters studied were inlet air temperature, feed flow rate, and atomization speed.

Statistical analysis carried out on the experimental data showed that the feed flow and atomization speed had significant effects on the dryer yield. Moisture content and yield had an inverse relationship to the inlet air temperature.

Furthermore, some physical properties of *Opuntia* mucilage powder were evaluated. The particle morphology observed was of spheres with collapsed fragments and agglomerate structure. The glass transition temperature and water adsorption behavior indicated that powder is stable for room temperature <45 °C and relative humidity <50%. The GAB model gave good fit ($R^2 > 0.99$) for the experimental data of adsorption isotherms. Net and total sorption heats were determined.



## 1. Introduction

Mucilage is a complex carbohydrate with a great capacity to absorb water. It is produced by the *Cactaceae* family and is considered a potential source of industrial hydrocolloids (Sáenz, Sepúlveda, & Matsuhiro, 2004). The main constituent of this substance is a hetero-polysaccharide with a molecular weight from $2.3 \times 10^4$ to $3 \times 10^6$ g mol$^{-1}$ (Cárdenas, Higuera-Ciapara, & Goycoolea, 1997; Medina-Torres, Brito-De La Fuente, Torrestiana-Sánchez, & Katthain, 2000). Multiples uses have been found for this component, for instance as a food thickener, food emulsifier, as a water purifier (polyelectrolyte molecule), as an adhesive for lime [$Ca(OH)_2$], as a natural super-plasticizer in mortars and as a food product (Cárdenas et al., 1997; Medina-Torres et al., 2000; Miller, Fugate, Craver, Smith, & Zimmerman, 2008; Sáenz et al., 2004; Torres-Acosta & Cano-Barrita, 2007). The *Opuntia ficus-indica* (*Ofi*) mucilage is a mixture of acidic and neutral polysaccharides consisting primarily of 24.6–42% of arabinose; 21–40.1% of galactose; 8–12.7% of galacturonic acid; 7–13.1% of rhamnose and 22–22.2% of xylose (Sáenz et al., 2004). Although mucilage gum is not yet commercially available, there is a marked interest among companies to begin producing it on large scale (Cárdenas et al., 1997).

Due to its high water activity (>0.8) and composition, fresh mucilage is susceptible to microbial attack, thus the need to extend its shelf life by multiple preservation process.

Spray drying can be used to convert liquid mucilage into powdered mucilage. Spray drying is the transformation of material from a fluid state into a dried particle form by spraying it through a hot drying medium. This method is widely used for both food and pharmaceutical manufacturing processes. This technique offers the advantages of relatively low temperatures and short particle residence times (5–100 s). In consequence, certain properties of food, such as flavor, color, and nutrients, are retained in high percentages (Masters, 1991; Rodríguez-Hérnandez, González-García, Grajales-Lagunes, & Ruiz-Cabrera, 2005).

Multiple organic materials have been dried using this technique, such as fruit juices, milk, tomato pulp, soybean extract, sweet potato puree, polysaccharides, food additives (i.e. flavor compounds), lipids and bioactive compounds (Chegini & Ghobadian, 2007; Goula, Konstantinos, & Adamopoulos, 2005; Grabowski, Truong, & Daubert, 2008; Narayan, Marchant, & Wheatley, 2001; Rodríguez-Hérnandez et al., 2005). In the majority of these studies, the materials were dried with drying aids, or carriers, to reduce the stickiness of sugar-rich foods and reduce wall deposition problems. Additionally, the use of these agents has had a positive effect on the protection of bioactive compounds (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007). Many of these drying aids, such as starches, maltodextrins, corn syrups and gum arabic, have high molecular weights and high glass transition temperatures. The dis-

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advantages of using drying agents include an additional cost and interference with the properties of the powder.

Non-sticky products can be dried using a simpler dryer design and the powder obtained is relatively less hygroscopic and more flowing (Goula et al., 2005). Materials with composition similar to *Ofi* mucilage have been dried by atomization without carrier agents, e.g. xanthan gum-gelatin dispersion (Salvador, Sanz, & Fiszman, 2001), and flaxseed gum (Oomah & Mazza, 2001; Wang, Wang, Li, Xue, & Mao, 2009). In all of these studies, only the rheological properties of reconstituted powders were evaluated.

Orozco, Díaz, and García (2007) dried *Ofi* mucilage in a laboratory spray drier, and then evaluated the rheological properties of rehydrated powders. They found a good rheological response and described intervals through which successful drying can be achieved in powders with a moisture content of 3–5%: 120–170 °C drying air temperature, 7 $m^3$/h drying air flow rate, 50 psi pressure atomizer, and a 0–0.1 L/h feed flow rate.

In spray drying, the operating conditions and the dryer design used depend on the characteristics of the material to be dried and the desired powder specifications. Product characteristics are important as they have bearing on transport costs, packaging considerations, and achieving precise quality standards.

The physical properties of the material related to ease the reconstitution include particle porosity, particle size and its distribution, apparent and true density, friability, dispersibility, and moisture content (Souza et al., 2009; Walton & Mumford, 1999). An in-depth and complete understanding of the powder properties is essential in order to optimize the process in terms of functionality and cost (Kurozawa, Morassi, Park, & Hubinger, 2009). Furthermore, a pilot plant test is indispensable for designing new industrial spray dryers which meet performance specifications (Chegini & Ghobadian, 2007). Studying the effect of operating parameters on the physical properties of powder helps to identify the optimum operating conditions of spray dryers and their effect on powder characteristics (Chegini & Ghobadian, 2007).

However, the powder properties of *Ofi* mucilage have not been evaluated in previous research. Hence, the objectives of this study were to evaluate the effect of parameters processing on powder properties (bulk density, moisture content) and yield. Additionally, the powder obtained from the best drying conditions was used for the determination of particle morphology, glass transition temperature, as well as water adsorption behavior. The results of this study may be helpful in developing cost-effective commercial processes for *Ofi* mucilage as powdered food additive (e.g. thickener, emulsifier).

## 2. Materials and methods

### *2.1. Plant material*

Cladodes with a mean age of 13 months were harvested from a local farm ("Tlapanochestli Ranch", Santa María Coyotepec, Oaxaca, México). Cladodes with a moisture content of 91% dry basis (db) were washed with water to eliminate spines from the skin.

### *2.2. Mucilage extraction*

An aqueous extraction was used. Cladodes were cut into small slices with a contact area and thickness of 0.00375 $m^2$ and 2 ± 0.2 mm, respectively, using a steel knife and a vegetable grater. The slices were weighed and put into a stainless steel container, and distilled water was added in the ratio of 1:3, this was kept at 86 °C during 3.6 h under agitation (Cai, Lu, & Tang, 2008). Finally, the extracted mucilage with a concentration of 3° Brix was separated from solid mass by decantation, then was filtered using a metallic sieve (No. 100) and stored at 4 °C in refrigeration.

**Table 1**
Factorial design with three central points.

| Treatment | Inlet air temperature (°C) | Atomizer speed (rpm) | Feed flow rate (L/h) |
|---|---|---|---|
| 1 | 130 | 21,000 | 2.30 |
| 2 | 170 | 21,000 | 2.30 |
| 3 | 130 | 24,000 | 2.30 |
| 4 | 170 | 24,000 | 2.30 |
| 5 | 130 | 21,000 | 3.30 |
| 6 | 170 | 21,000 | 3.30 |
| 7 | 130 | 24,000 | 3.30 |
| 8 | 170 | 24,000 | 3.30 |
| 9 | 150 | 22,500 | 2.80 |
| 10 | 150 | 22,500 | 2.80 |
| 11 | 150 | 22,500 | 2.80 |

### *2.3. Spray drying*

A Mobile Minor concurrent flow spray dryer (Niro, Copenhagen, Denmark), equipped with a rotating atomizer nozzle (TS-Minor, M02/A) was used to dry the mucilage. The diameter of the drying chamber was 800 mm × 620 mm/cone 60°. The mucilage was fed into the drying chamber using a peristaltic pump (Watson-Marlow 505S/RL). A $2^3$ factorial design with three central points was used to evaluate the effect of the independent variables: inlet air temperature (130–170 °C), feed flow rate (2.3–3.3 L/h) and atomizer speed (21,000–24,000 rpm) on the powder properties. Drying air flow rate was fixed at 84 ± 2 kg/h. Distilled water at room temperature was used for start-up and shut down operations. The experimental design matrix is shown in Table 1.

### *2.4. Powder analysis*

The powder samples produced during the experiments were kept in polyurethane bags in vacuum conditions until the analysis stage. The powder properties measured include powder yield, moisture content, and bulk density. The experiment was conducted three times and the averages of the three measurements were recorded (Table 2).

#### *2.4.1. Yield*

Spray drying yield was evaluated through determination of recovered product, given by the ratio between the total recovered product mass and the mass of extract initially fed into the system, and is expressed by the following equation:

$$y = \frac{(W_2 - W_1) - X_{wb}(W_2 - W_1)}{M_V T_s} 100 \quad (1)$$

where $y$ is powder yield (%), $X_{wb}$ is the moisture content in wet basis (wb), $M_V$ is the volume of mucilage feed (L), $T_s$ is the content of total solids (g dry matter/L), and $W_1$ and $W_2$ are the weight (g) of the powder receptacle before and after spray drying, respectively.

#### *2.4.2. Moisture content*

The moisture content of the mucilage powders was determined gravimetrically (A.O.A.C., 1984) and moisture loss was expressed in terms of percent dry basis (db).

#### *2.4.3. Bulk density*

The bulk density of the powder was measured by weighing out 1 g of the sample and placing it into a 10 mL graduated cylinder (Cai & Corke, 2000; Chegini & Ghobadian, 2007). This was tapped 10 times onto a rubber mat from a height of 10 cm. The volume was then recorded and used to calculate bulk density as g/mL.

**Table 2**
Properties of mucilage powders.

| Treatment | Yield (%) | Moisture content (db) | Bulk density (g/mL) | $T_{oulet}$ (°C) |
|---|---|---|---|---|
| 1 | 70.74 ± 1.68 | 9.32 ± 1.19 | 0.769 ± 0.014 | 74.33 ± 2.08 |
| 2 | 42.44 ± 4.36 | 4.42 ± 0.77 | 0.660 ± 0.024 | 92.67 ± 0.58 |
| 3 | 68.88 ± 1.52 | 8.41 ± 0.64 | 0.641 ± 0.022 | 70.00 ± 1.00 |
| 4 | 49.36 ± 6.51 | 4.50 ± 0.43 | 0.719 ± 0.012 | 91.00 ± 1.00 |
| 5 | 0.77 ± 0.12 | 24.81 ± 1.20 | N/A[a] | 53.00 ± 1.73 |
| 6 | 29.60 ± 5.75 | 8.01 ± 0.26 | 0.679 ± 0.010 | 74.33 ± 1.15 |
| 7 | 67.47 ± 2.74 | 10.79 ± 0.68 | 0.623 ± 0.006 | 63.33 ± 0.58 |
| 8 | 42.72 ± 1.73 | 8.78 ± 0.52 | 0.593 ± 0.033 | 67.67 ± 0.58 |
| 9 | 54.88 ± 3.14 | 8.67 ± 0.13 | 0.570 ± 0.003 | 72.00 ± 1.00 |
| 10 | 51.10 ± 6.81 | 9.24 ± 0.55 | 0.624 ± 0.008 | 72.33 ± 0.58 |
| 11 | 49.22 ± 1.25 | 8.08 ± 0.79 | 0.614 ± 0.015 | 71.67 ± 0.58 |

[a] Not available.

#### 2.4.4. Statistical analysis

The statistical analysis of yield, bulk density and moisture content were done with NCSS© software (Hintze, J., Kaysville, Utah, USA). The data were subjected to analysis of variance (ANOVA) using general linear models (GLM) approach.

### 2.5. Characterization of mucilage powder

The sample used for the determination of glass transition temperature ($T_g$), water adsorption behavior, and particle morphology was obtained by the following drying conditions: inlet air temperature, 150 °C; feed flow rate, 2.8 L/h and atomizer speed, 22,500 rpm. The resulting powder had a humidity of 7.2 ± 0.1% db.

#### 2.5.1. Glass transition temperature

Mucilage $T_g$ was determined by differential scanning calorimetry (DSC). The analysis was performed with a DSC-7 calorimeter (PerkinElmer, Norwalk, CT, USA). A sample of 8 ± 0.2 mg was inserted into an aluminum pan cell (PerkinElmer, 0219-0071). The temperature range was from 25 to 70 °C with a heating rate of 10 °C/min. An empty pan was used as a control.

#### 2.5.2. Adsorption equilibrium experiments

The static gravimetric method was used to evaluate the equilibrium moisture content (EMC) of *Ofi* mucilage powder (Lahsasni, Kouhila, Mahrouz, & Fliyou, 2003). Adsorption isotherms were determined at working temperatures of 20, 30, 40 and 50 °C. Nine salts were chosen (LiCl, $CH_3COOK$, $MgCl_2{\cdot}6H_2O$, $K_2HPO_4$, $Na_2Cr_2O_7{\cdot}2H_2O$, KI, NaCl, $(NH_4)_2SO_4$ and $BaCl_2{\cdot}2H_2O$) to provide a range of $a_w$ of 0.11–0.89. The EMC was calculated after three consecutive weight measurements showed a difference less than 0.001 g.

The experimental data were fitted to the GAB (Guggenheim-Anderson-DeBoer) equation using non-linear regression. The GAB equation is normally written as follows:

$$X_{eq} = \frac{X_m C k a_w}{(1 - k a_w)(1 - k a_w + C k a_w)} \quad (2)$$

where $X_{eq}$ is the equilibrium moisture content (g $H_2O$/g dry matter), $a_w$ is water activity, $X_m$ is the monolayer moisture content (g $H_2O$/g dry matter), and $C$ and $k$ are constants related to temperature effects.

The solid surface area ($S_A$) of the mucilage sample was determined using the Cadden equation (3) (Cadden, 1988):

$$S_A = X_m \left(\frac{1}{MW_{H_2O}}\right)(N)(A_{H_2O}) \quad (3)$$

where $S_A$ is the solid surface area ($m^2$/kg solid), $X_m$ is the monolayer moisture content (kg $H_2O$/kg dry matter), $MW_{H_2O}$ is the molecular weight of water (18 kg/kmole), $N$ is Avogadro's number ($6 \times 10^{26}$ molecules/kmole), and $A_{H_2O}$ is the area of a water molecule ($10.6 \times 10^{-20}$ $m^2$).

The net isosteric heat of sorption was determined by applying Eq. (4) to adsorption isotherms at different temperatures (Soysal & Öztekin, 2001). The total heat required for this process was obtained by adding the latent heat of vaporization for pure water (Eq. (5)):

$$q_{sn} = -R\frac{\partial \ln(a_w)}{\partial(1/T)} \quad (4)$$

$$Q_{st} = q_{sn} + L_v \quad (5)$$

where $T$ is the absolute temperature (K), $R$ is the gas constant (0.46188 kJ/kg $H_2O$ °K), $q_{sn}$ is the net isosteric heat of sorption (kJ/kg $H_2O$), $Q_{st}$ is the total isosteric heat of sorption (kJ/kg $H_2O$), and $L_v$ is latent heat of vaporization (2413.57 kJ/kg $H_2O$).

The experimental data of the relation between the $Q_{st}$ and the EMC was fitted to Eq. (6), $C$ (kJ/kg $H_2O$) and $b$ (dimensionless) are model constants and were determined by applying non-linear regression method (Soysal & Öztekin, 2001).

$$Q_{st} = C\ \exp(-bX_{eq}) + L_v \quad (6)$$

#### 2.5.3. Particle morphology

Examination of the surface morphology of mucilage powder was carried out with images taken using a JEOL JSM-6360ZX scanning electron microscope.

## 3. Results and discussion

### 3.1. Mucilage powder properties

The values of the variable operating conditions and a compilation of the resulting data for moisture content, yield, bulk density and outlet air temperature are listed in Table 2.

#### 3.1.1. Effects of the process parameters on yield

Analyzing Fig. 1, it can be observed that that increases in feed flow rate or in inlet air temperature reduce the yield. These results are in agreement with spray dried orange juice (Chegini & Ghobadian, 2007). The effect of the inlet air temperature on yield can be due to powder to melt and cohere to the chamber wall, thus reducing the amount of powder production and, consequently, reducing yield.

However, others studies reported an opposite effect of inlet air temperature on yield. Higher drying temperature usually resulted in faster drying (higher drying rate) and higher powder productivity (Cai & Corke, 2000; Goula et al., 2005; Tonon, Barbet, & Hubinger, 2008). The behavior observed by these studies could be attributed to the addition of carrier agents and higher drying air temperatures, resulting in faster drying times and higher powder productivity.

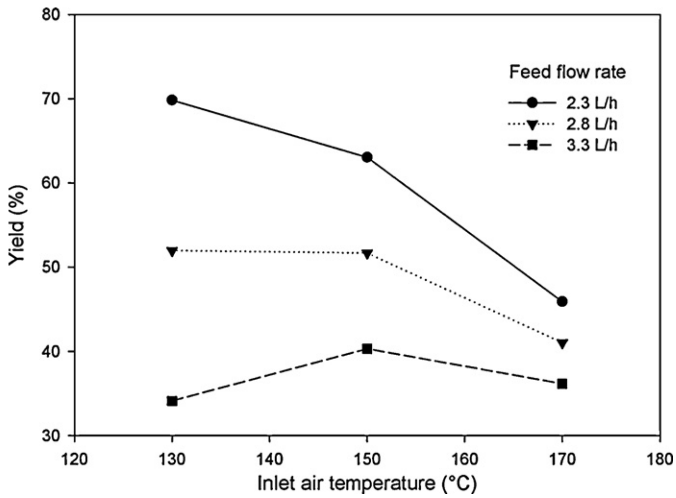


**Fig. 1.** Effects of inlet air temperature and feed flow rate on yield.

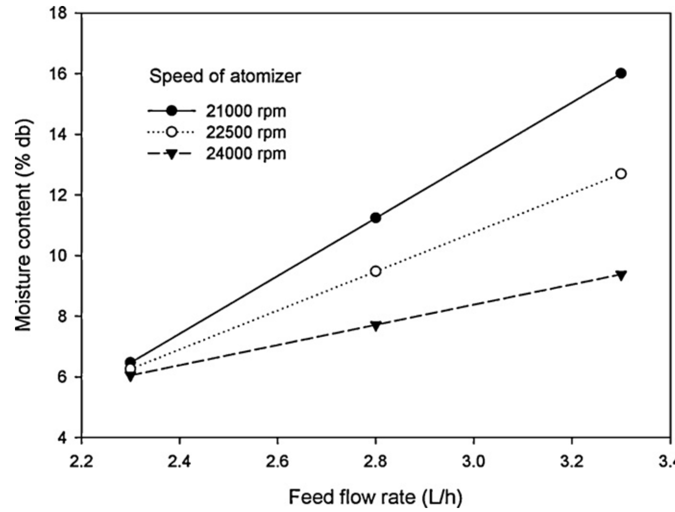


**Fig. 3.** Effects of atomizer speed and feed flow rate on moisture content.

In addition, high inlet air temperature can affect the state of the mucilage polymer (glassy or rubbery state), producing the problem of powder stickiness. The effect of the feed flow rate on yield can be attributed to the slower heat and mass transfer which occurs when the process is carried out with higher feed flow rates (Tonon et al., 2008).

The effects on yield of uncontrolled factors such as relative humidity of drying gas, the air temperature used for atomization, fluctuations in the compressed air flow rate and the drying gas flow rate, and feed parameters (temperature and the degree of feed aeration) could explain the deviation of values generated in some of the data obtained for yield (see Table 2).

The drying conditions in experiment 5 (see Table 2) gave the lowest yield. These results are due to the interaction between the high feed flow rate and low inlet temperature, thus producing the lowest outlet air temperature with the highest moisture content, preventing appropriate drying. Under these drying conditions, mucilage adhered to the wall of the drying chamber was observed.

Increasing atomization speed increases yield. At a constant feed flow rate of 2.3 L/h, atomizer speed behavior is more homogenous than at a feed flow rate of 3.3 L/h (Fig. 2). This suggests that feed flow rate has a greater effect on yield than atomizer speed.

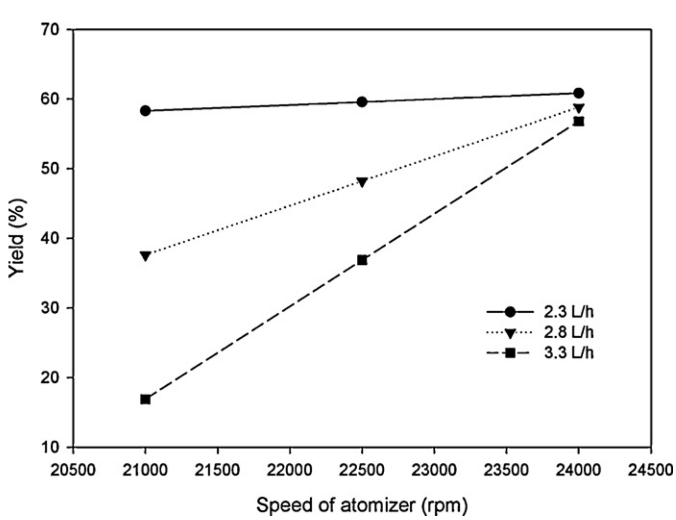


**Fig. 2.** Effects of atomizer speed and feed flow rate on yield.

*3.1.2. Effects of the process parameters on moisture content*

The moisture content of mucilage powders varied from 4.41 to 10.79% db (see Table 2). Powder moisture content was observed to increase as the feed flow rate increases, and decrease as inlet air temperature and/or atomizer speed increases (Fig. 3). At higher inlet air temperatures, there is a greater temperature gradient between the atomized feed and the drying air, resulting in a greater driving force for water evaporation, thus producing powders with lower moisture content (Tonon et al., 2008).

Higher feed flow rates reduce the contact time between droplets and drying air, producing a less efficient heat transfer. This results in less water evaporation, and therefore higher moisture content (Kurozawa et al., 2009).

Higher speeds of atomization produce smaller droplets, and high feed flow rates produce a larger droplet size, therefore affecting the transfer of mass and energy.

These results are consistent with those obtained by Tonon et al. (2008) with açai powder, by Goula et al. (2005) with tomato powder, and by Cai and Corke (2000) with betacyanin extracted from *Amaranthus*.

Generally, in a spray drying system, the temperature of air leaving the drying chamber (exhaust) controls residual moisture content. This behavior can be seen in Table 2; with an increase in the outlet air temperature, the moisture content in the powders decreased. The range of outlet air temperatures seen in this study was from 53 to 92 °C. Outlet air temperature is principally controlled by the inlet air temperature and the feed flow rate. Both higher inlet temperatures and lower feed flow rates produce higher outlet temperatures. A lower drying air flow rate causes an increase in product residence time in the drying chamber, leading to a greater degree of moisture removal and thus a reduction in outlet temperature (Tewa-Tagne et al., 2007).

*3.1.3. Effects of the process parameters on bulk density*

The bulk density of mucilage powders varied from 0.570 to 0.769 g/mL and decreased when spray drying air temperature increased (see Table 2). Cai and Corke (2000) observed similar trends for the bulk density of pigment powders. This behavior is most likely due to the higher drying rate obtained at higher drying temperatures that produce a higher ratio of surface to volume for the spray dried capsules, thus causing lower bulk density of the powders. This leads to the formation of vapor impermeable films on the droplet surface, followed by the formation of vapor bubbles and, consequently, droplet expansion.

**Table 3**
Analysis of variance for the overall effect of process variables on powder properties.

| Process variables | Sum of squares | | |
|---|---|---|---|
| | Yield | Moisture content | Outlet air temperature |
| Inlet air temperature | 819.48 | 296.14* | 1595.29* |
| Feed flow rate | 2882.04* | 234.38* | 1820.04* |
| Atomizer speed | 2882.04* | 84.38* | 2.04 |
| Inlet air temperature × atomizer speed | 805.04 | 100.04* | 77.04 |

* Significant at 0.01 level.

Therefore, there is a greater tendency for the particles to be hollow.

The effect could also be attributed to that a product with higher moisture content (most often dried at low temperatures and high feed flow rates) would tend to have a higher bulking weight caused by the presence of water, which is considerably denser than the dry solids (Chegini & Ghobadian, 2007).

Decrease in the bulk density with the increase of the atomization speed was a result of the particle size and the moisture content of the samples. Rotary atomization generally produces a larger particle size in comparison to nozzle atomization. Two-fluid nozzle atomizers obtain the smallest particles sizes. The atomizer is the most important feature of a spray dryer. Its selection and operation are of great importance in achieving cost-efficient production while maintaining product quality (Masters, 1991).

Other factors such as feed temperature, feed rate, feed concentration, residual particle size and distribution, as well as powder temperature, also influence bulk density (Walton & Mumford, 1999).

Statistical analysis revealed the significant linear effects of atomizer speed and feed flow rate ($P < 0.01$) on yield (Table 3). Each drying variable had significant effect on moisture content, as well as that of the interaction between inlet temperature and atomizer speed. Outlet temperature had a significant effect on feed flow rate, and on inlet air temperature.

### 3.2. Characterization of mucilage powder

The mucilage powder had a glass transition temperature ($T_g$) of 45 °C with a moisture content of 7.2% db (Fig. 4). This value of $T_g$ is consistent with those found for others polymers such as mal-

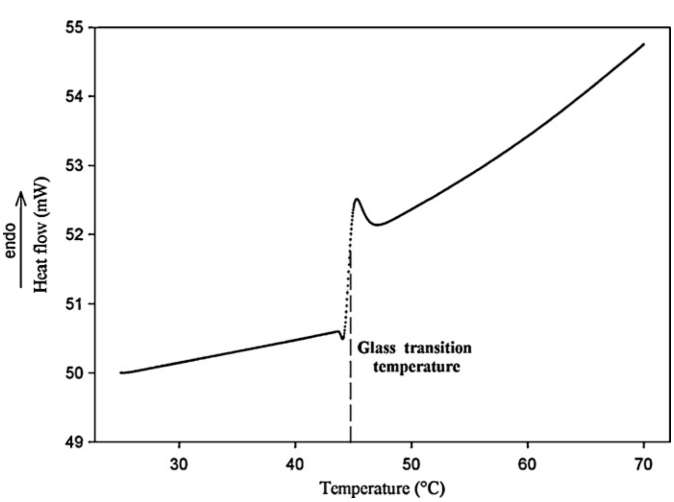


**Fig. 4.** Thermogram of spray dried mucilage powder.

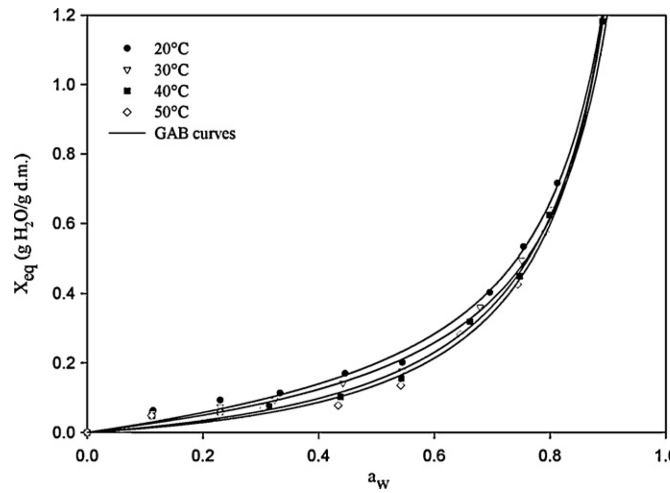


**Fig. 5.** Adsorption isotherms of mucilage powder at different temperatures.

todextrin solutions (10–25 DE), where the $T_g$ ranged from 45.4 to 54.7 °C at 32% RH (Cai & Corke, 2000), amylose with a $T_g$ of 43.8 °C, and synthetic polymers, e.g. poly(hexamethylene adipamide) with a $T_g$ ranged from 46.8 to 56.8 °C and poly(butylene terephthalate) of $T_g$ ranged from 29.8 to 59.8 °C. The molecular weight of *Ofi* mucilage reported by other researchers ranged from $2.3 \times 10^4$ to $3 \times 10^6$ g mol$^{-1}$ (Cárdenas et al., 1997; Medina-Torres et al., 2000) and this could explain the value of $T_g$ obtained. This suggests a glassy state of the amorphous polymer at the room temperature (22–28 °C). $T_g$ is related to chain stiffness and polymer chain structure; it increases as cross-link density increases. A lower $T_g$ causes higher hygroscopicity in the spray dried powder, as lower molecular weight implies shorter chains and more hydrophilic groups (Cai & Corke, 2000).

The structure of mucilage generated by the spray drying process explains the behavior of water adsorption and is related to the $T_g$. The equilibrium moisture content (EMC) results obtained at each water activity level and at each working temperature are shown in Fig. 5. In general, all obtained isotherms fell into type III. This isotherm type is observed in high sugar foods, and is characteristic of weak adsorbate–adsorbent interactions (Rao & Rizvi, 1986; Yañez-Fernández, Orozco-Álvarez, & Velásquez-Sánchez, 2006). It is quite often associated with both non-porous and microporous adsorbents (Ayranci, Ayranci, & Dogantan, 1990; Rao & Rizvi, 1986). At low $a_w$, on the surface of the carbohydrate, water can be adsorbed only in polar sites; at high $a_w$ (>0.7), dissolution of carbohydrates occurs (Ayranci et al., 1990).

Temperature exerts a clear influence on adsorption isotherm. At a constant water activity level, the EMC increases with the decrease of work temperature. This implies an inverse relationship of the EMC with respect to temperature. Similar results for many plants and food materials have been reported (Lahsasni, Kouhila, & Mahrouz, 2004; Uribe, Miranda, Lemus, & Vega-Gálvez, 2008). This effect could be due to changes in the bonding of the water

**Table 4**
The adsorption parameters for the GAB model.

| GAB parameters | | | | |
|---|---|---|---|---|
| $T$ (°C) | $X_m$ (kg/kg dm) | $C$ | $K$ | $R^2$ |
| 20 | 0.4021 | 0.6271 | 0.9838 | 0.9980 |
| 30 | 0.2638 | 0.7842 | 0.9899 | 0.9981 |
| 40 | 0.0925 | 1.4208 | 0.9742 | 0.9979 |
| 50 | 0.0595 | 1.7533 | 0.9782 | 0.9960 |

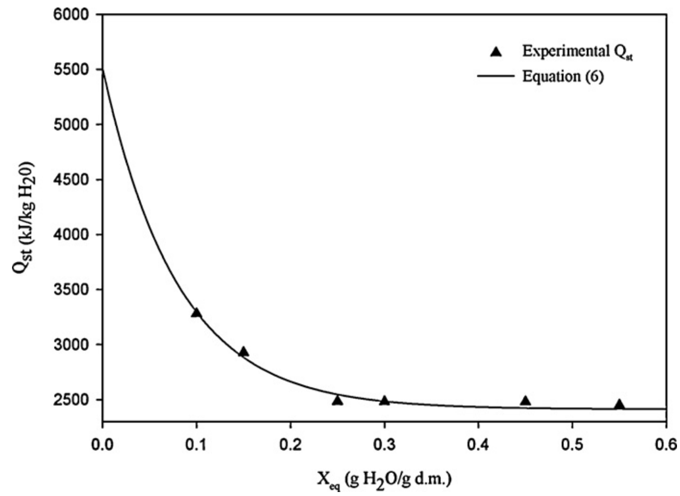


**Fig. 6.** Total isosteric heat of adsorption as a function of moisture content.

molecules, or to a decrease of the solubility of the solids in water (Uribe et al., 2008).

In regards to the GAB parameters and $S_A$ are presented in Table 4, it can be observed that $X_m$ decreases as temperature increases. This behavior is explained by the reduction in the total number of active sites for water binding as a result of physical or chemical changes induced by temperature (Quirijns, van Boxtel, van Loon, & van Straten, 2005). On the other hand, the Guggenheim constant $C$ increases proportionally to temperature, while $k$ decreases slightly. This is in agreement with the results of Cadden (1988) for microcrystalline cellulose and guar gum.

The low values of $C$ (<2) and high values of $k$ (>0.9) would mean that the monolayer and multilayer molecules do not greatly differ and that the multilayer molecules behave more like liquid molecules (Quirijns et al., 2005). A good fit ($R^2 > 0.95$) with the experimental of the data of isotherm adsorption of mucilage powder was obtained using the GAB model (Table 3).

The $S_A$ values were from 142.07, 93.21, 32.68, 21.00 m$^2$/kg solid for temperatures of 20, 30, 40, and 50 °C, respectively. This decreased with increased temperature, indicating that active sites in the monolayer are less prevalent at high temperatures.

The total isosteric heat of sorption ($Q_{st}$) for mucilage powder is presented in Fig. 6 as function of the moisture content of the material. $Q_{st}$ was fitted to an exponential function (Eq. (6)), where $C$ had a value of 3095.7 kJ/kg $H_2O$ and $b$ a value of 12.6 with $R^2 > 0.98$. At low moisture content (<0.2 g $H_2O$/g dry matter) the value for $Q_{st}$ was higher. This value dropped rapidly from 5500 to 2650 kJ/kg $H_2O$. This is indicative of intermolecular attraction forces between sorptive sites and water steam. In the monolayer region, water is tightly bound to material. This corresponds with high interaction energy. As moisture content increases, most active sites become occupied and sorption occurs on less active sites, thus giving lower heats of sorption (Quirijns et al., 2005). The EMC at which the heat of sorption approaches the heat of vaporization of water is 0.6 g $H_2O$/g dry matter.

Examination of SEM micrographs shows polydisperse powder particles, with a mucilage powder particle size ranging from approximately 100 to 2 μm (Fig. 7(A and B)). These particles were observed to be sphere-like with collapsed walls, and of agglomerate structure. Some authors have suggested that agglomeration occurs because of static electrical effects and van der Waals forces. The particles with an agglomerate structure are composed of individual grains of material bound together by sub-micron dust (of the same material) and the agglomerated structures may bind to each other as well (Walton & Mumford, 1999). In addition, the mucilage powder was humidified to a moisture content of 19.1% db and its micrographs are presented in Fig. 7(C and D). In this figure, the adsorption of water of the particles, as well as the permanence of the agglomerated structure, is observable.

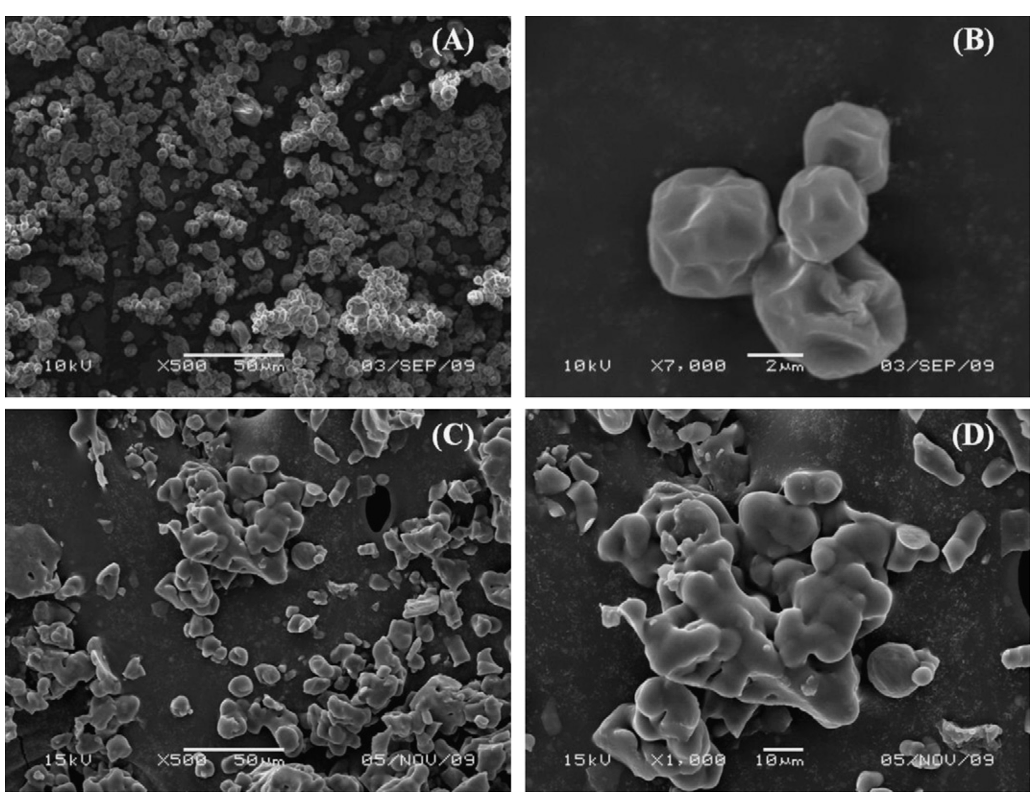


**Fig. 7.** Micrographs of spray dried mucilage in agglomerate form at $a_w$ of 0.2 (A and B), and of hydrated powder at $a_w$ of 0.4 (C and D).

Other studies indicate that spray dried materials may have less hydrogen bonding sites available for water molecules in sorption than freeze-dried materials (Haque & Roose, 2006). This is in agreement with the water adsorption behavior observed in mucilage powder (Fig. 5).

## 4. Conclusions

This study has shown that the use of spray drying to process *O. ficus-indica* mucilage produces a stable, powdered product of low higroscopicity (moisture content less than 0.2 g $H_2O$/g dry matter), without the need for carrier agents. The mucilage powder characterized in this work had a glass transition temperature of 45 °C and a polydisperse particle size distribution with agglomerate structure. The water adsorption behavior of the mucilage powder indicated a weak adsorbate–adsorbent interaction, without much difference between the mono and mutilayers, based on GAB parameters.

Furthermore, from the effects of spray drying conditions, it was observed that:

- Yield increases as inlet air temperature and feed flow rate decrease, and as atomization speed rises.
- Moisture content diminishes as inlet air temperature and atomizer speed increase, or with a decrease in the feed flow rate.
- Bulk density decreases with an increment in inlet air temperature, or a diminution in moisture content.

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