

Effect of a Formulation with Agave Fructans as a Carrier Polymer on the Quality Properties of Chayote (*Sechium edule*) Powder

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Efecto de una formulación con fructanos de Agave como polímero acarreador sobre las propiedades de calidad de Chayote (Sechium edule) en polvo

Efecte d'una formulació amb fructans d'Agave com a polímer portador sobre les propietats de qualitat de Chayote (Sechium edule) en pols

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ABSTRACT:

The native agave fructans as carrier agent can be an alternative to improve quality properties of chayote powder, since in this form have some technological applications such as sweeteners and stabilizers. The objective of this study was to diminish the added maltodextrin and evaluate the effect of fructans concentration as carrier agent on the quality properties of chayote powder. The effect of the two stabilizing additives for chayote juice was studied through calorimetry, FT-IR spectroscopy and some physicochemical properties. The samples were prepared with 5, 7 and 10% maltodextrin concentration, added with 0, 2 and 4% fructans. The spray drying process was carried out with inlet temperature of 110, 115 and 120 °C. The treatments with added fructans showed lower moisture content than treatments with maltodextrin only. The powder stability was observed in the sorption isotherms, which presented a higher degree of hysteresis during storage where fructans were added. The addition of fructans significantly increased the glass transition temperature (T_g) of the powder. The FT-IR showed the ability of agave fructans to water retention. The micrographs revealed spherical particles in a range from 16 to 105 μm. The evaluation of physicochemical properties confirmed the addition of fructans confers stability to chayote powder.

Keywords: Chayote juice, Agave Fructans, Calorimetry, Spray drying, Sorption Isotherms.

RESUMEN:

Los fructanos de agave nativos como agente transportador pueden ser una alternativa para mejorar las propiedades de calidad del polvo de chayote, ya que en esta forma tienen algunas aplicaciones tecnológicas como edulcorantes y estabilizadores. El objetivo de este estudio fue disminuir la cantidad de maltodextrina añadida y evaluar el efecto de la concentración de fructanos como agente transportador en las propiedades de calidad del polvo de chayote. El efecto de los dos aditivos estabilizadores para el jugo de chayote se estudió mediante calorimetría, espectroscopía FT-IR y algunas propiedades fisicoquímicas. Las muestras se prepararon con 5, 7 y 10% de maltodextrina, añadidas con 0, 2 y 4% de fructanos. El proceso de secado por aspersión se realizó con una temperatura de entrada de 110, 115 y 120 °C. Los tratamientos con fructanos agregados mostraron menor contenido de humedad que los tratamientos con sólo maltodextrina. La adición de fructanos aumentó significativamente la temperatura de transición vítrea (T_g) del polvo. El FT-IR mostró la capacidad de los fructanos de agave para la retención de agua. Las micrografías revelaron partículas esféricas con diámetro de 16 a 105 μm. La evaluación de las propiedades fisicoquímicas confirmó que la adición de fructanos confiere estabilidad al polvo de chayote.

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Palabras clave: Jugo de chayote, Fructanos de Agave, Calorimetría, Secado por aspersión, Isotermas de sorción.

RESUM:

Els fructans d'atzavara nadius com a agent transportador poden ser una alternativa per millorar les propietats de qualitat de la pols de chayote, ja que en aquesta forma tenen algunes aplicacions tecnològiques com edulcorants i estabilitzadors. L'objectiu d'aquest estudi va ser disminuir la quantitat de maltodextrina afegida i avaluar l'efecte de la concentració de fructans com a agent transportador a les propietats de qualitat de la pols de Chayote. L'efecte dels dos additius estabilitzadors per al suc de Chayote es va estudiar mitjançant calorimetria, espectroscòpia FT-IR i algunes propietats fisicoquímiques. Les mostres es van preparar amb 5, 7 i 10% de maltodextrina, afegides amb 0, 2 i 4% de fructans. El procés d'assecatge per aspersió es va realitzar amb una temperatura d'entrada de 110, 115 i 120 ° C. Els tractaments amb fructans agregats van mostrar menor contingut d'humitat que els tractaments amb només maltodextrina. L'addició de fructans va augmentar significativament la temperatura de transició vítria (Tg) de la pols. El FT-IR va mostrar la capacitat dels fructans d'atzavara per a la retenció d'aigua. Les micrografies van revelar partícules esfèriques amb diàmetre de 16 a 105 µm. L'avaluació de les propietats fisicoquímiques va confirmar que l'addició de fructans confereix estabilitat a la pols de chayote.

Paraules clau: Suc de chayote, fructans d'agave, calorimetria, assecat per aspersió, isoterms de sorció.

1. INTRODUCTION

Chayote (*Sechium edule* (Jacq.), Swartz) fruit is very popular in the diet of people living in Latin America countries with a low rate of exportation to other areas of the world. This fruit has a low lipid, protein and calorie content, but it is an important source of minerals, aminoacids, and vitamins. Chayote has a very high moisture content (> 90% w.b.), resulting in extensive postharvest losses due to enzymatic and microbial deterioration¹. The chayote fruits germination during horticultural maturation limits its shelf life in a fresh form which represents serious disadvantages during transportation. A feasible way to extend the shelf life of chayote is through the development of reduced moisture products².

Spray drying is a preservation alternative to process fruit and vegetable juices into stable powders with new possibilities for industrial applications. This technique has been widely used in the food and pharmaceutical industries. Processing of biological materials by spray drying presents some difficulties because they are generally thermolabile and great care needs to be taken to prevent denaturation, including proper dryer design, drying operating conditions and types of stabilizers³.

Fruit juice powders have many benefits and economic potential over liquids such as reduced volume or weight, reduced size packaging, easier handling and transportation, and much longer shelf life. Powders have the advantage of being easily measurable ingredients, and then could be used in many foods and pharmaceutical products such as flavoring and coloring natural agents⁴.

The carrier agents normally used in the spray drying of fruit juices are maltodextrins and arabic gum because of their high solubility and low viscosity, important conditions for the spray drying process. Arabic gum is effective as carrier agent due to its emulsifying properties, since it has low protein content. Moreover, it exhibits high solubility and low viscosity in aqueous solution, which facilitates the spray drying process⁵. Arabic gum and maltodextrin mixtures have proved to be more efficient than separately used in spray drying⁶. Recent studies have attributed agave fructans with techno-functional applications such as thermal protectives, encapsulants, and stabilizers⁷.

The fruit juices viscosity can difficult the spray drying process in terms of low yield, equipment wear and powders adherence to the dryer walls. The sugar content in the fruit juice, such as sucrose, fructose and glucose contribute to increased viscosity values of the juice and powders⁸. The presence of these sugars causes a higher glass transition temperature (Tg). The stickiness of the powders is therefore related to the Tg⁹. Glass transition is dependent (without phase change) of time, temperature and moisture content, that is, cooling or heating rates as well as temperature affect the transition phase, which is generally characterized by a discontinuity in physical, mechanical, electrical, thermal, and other properties on a material¹⁰. In cases of complex multicomponent mixtures, such as foods, the discontinuity occurs within a range of temperatures (ΔC_p). The most common method to determine glass transition is the differential scanning calorimetry (DSC), which detects the change in heat capacity occurring over the transition temperature range. In order to reduce stickiness, the glass transition temperature of the juice powder can be increased with the addition of maltodextrin¹¹. The Tg of an amorphous or semi-crystalline polymer is inferior to its melting point, it is important to differentiate between these two temperatures that describe a phase change. The Tg is observed as a change in heat flow or heat capacity. The advantage of DSC is the ease and speed to analyze transitions in materials.

This technique is based in the fact that when samples experiment a physical transformation such as a phase transition, more heat should flow than in the reference in order to maintain both samples at the same temperature. This is due to heat absorption on behalf of the sample as it experiences an endothermic phase transition. Based on the difference in heat flow between the sample and the reference, differential scanning calorimeters are able to measure the Tg during such transitions and are reflected in the curves as deviations from the base line¹⁰.

The composition studies of the fruit powders can be performed by Fourier transformer IR-spectroscopy

(FTIR). FTIR is generally employed as a rapid and non-destructive method to identify specific functional groups and molecules which can establish fingerprint a food ¹⁴.

The applications of agave fructans in the food industry have a promising future. However, to this day, no studies that consider the relation between thermal and water sorption properties as stabilizers in spray drying have been developed. These enable evaluation of stable storage conditions for fruit juice powders as a function of water activity. Thus, in this research the main objective was to evaluate the effects of agave fructans concentration as carrier agent to decrease the added maltodextrin and to improve the physicochemical properties of chayote powder.

2. MATERIALS AND METHODS

2.1. Raw materials

Chayote in consumption degree of ripeness was obtained in Tepic, Nayarit and subsequently washed and disinfected in a sodium hypochlorite solution (NaClO) at 200 ppm (0.02 %). Then, they were peeled and the juice of these fruits was obtained using a conventional extractor. The juice contained 13% total soluble solids. Maltodextrin D10 or native agave fructans, which are a mixture of different polymerization degree fructans, including high, medium and low molecular weight, from 500 to 3500 Da. These polymers were added to the juice for homogenization using an electric laboratory homogenizer (PRO Scientific. Inc. 300 PC, USA) at 350 rpm during 15 min and filtered in a No. 50 filter sieve with a particle size of 300 μ m. One liter of homogenized juice was used for each test.

2.2. Spray drying

A pilot spray dryer (SINOTEK S.A de C.V., model LPG5, Mexico) equipped with a rotatory nozzle was used with a feed rate of 15 mL \cdot min⁻¹, 2.5 bar and juice feed temperature at 25 °C, air inlet 9.4 m/s and three temperatures; 110, 115 and 120 °C, binaries mixes with three maltodextrin concentration (5, 7 and 10 %, m/v) and three fructans concentrations (0, 2 and 4 %, m/v). The outlet temperature range was from 74 to 85 °C.

2.3. Physicochemical properties

2.3.1. Moisture content

The content of moisture was determined using the thermobalance method ¹⁵. A thermobalance (Sartorius, MA 35, Germany) was used and operated at 75 °C at constant weight.

2.3.2. Water activity

Water activity was determined by an Aqualab 4TEV (Decagon devices). 5 g of sample were placed inside the chamber where the water activity was determined by the dew point principle at temperature of 35° C.

2.3.3. Hygroscopicity

This analysis was carried out according to the methodology described by Al-Kahtani and Hassan ¹⁶. Five grams of the sample were placed in a desiccator at 21 °C and at 76% relative humidity (solution of 36 g of NaCl in 100g of water) in a Petri dish of 9 cm in diameter. The weight was registered every 15 min in balance (Precisa BJ 4100D, Switzerland). The results were reported in grams of water absorbed by 100 g of dried solids (g/100 g). The absorbed water percentage (%HG) was calculated using (eq. 1):

$$HG(\%) = \frac{\Delta m / (M + M_i)}{(1 + \Delta m) / M} \quad (1)$$

Where Δm (g) is the increase in weight of powder after equilibrium, M is the initial mass of powder and M_i (% wb) is the free water content of the powder before exposure to the humid air environment.

2.3.4. Solubility

One gram of powder was diluted in 100 mL of distilled water, manually stirred until the entire sample was solubilized and centrifuged at 2598 g during 5 min (Hermle Z326K, Germany). A 2 mL aliquot was taken from the supernatant and was placed in Petri dish. Finally, it was dried in a stove (Novatech BTC-9100) at 105 °C for 5 hours. The solubility (%) was calculated by the weight difference.

2.3.5. Bulk density

Twenty grams of sample were placed in a test tube of 100 mL, then stirred in a vortex for 5 min. Bulk density was calculated by the powder mass flow rate and the final volume, the result was reported in g/cm³ ¹⁷. The Hausner index (HI) was calculated according to (eq. 2).

$$HI = \frac{V_t}{V_b} \quad (2)$$

Where V_t is the tapped density and V_b is the bulk density.

2.3.6. Wettability

The static method described by Freudig et al ¹⁸. One gram of powder was sprinkled over the surface of 100 mL of distilled water at 20 °C without agitation. The wettability was expressed as the necessary time for 1 g of powder to disappear from the water surface.

2.3.7. Color

The color analysis was performed using a colorimeter (Konica Minolta, CR-400, Japan). This colorimeter meets CIE-specifications (Commission Internationale de L'Eclairage), the color space uses three values (L^* , a^* y b^*) to describe the location of a color inside a three-dimensional space. The tone angle (h°) was determined with (eq. 3):

$$h^\circ = \tan^{-1}(b^*/a^*) \quad (3)$$

2.4. Scanning Electron Microscopy

A SEM (SEC, 3200 M, Korea) was used with an acceleration voltage of 20 kV. Two to five mg of powder sample

were fixed on double sided carbon adhesive tape and placed in a metal microscope slide, the samples were then coated with a very thin layer of gold, during 2 min under high vacuum conditions. The samples were systematically observed with 500 and 1500x magnification.

2.5. Analysis of moisture adsorption

The isotherms were determined using a vapour sorption analyzer (VSA) (AquaLab, Decagon Devices, Inc. Pullman, USA) employing the software VSA Dowlander. The isotherms were obtained at 45 °C using approximately 300 mg of the sample. The mathematical model was adjusted with GAB model (eq. 4).

$$M = \frac{m_0 \cdot C \cdot k \cdot a_w}{(1 - k \cdot a_w) \cdot (1 + (C - 1) \cdot k \cdot a_w)} \quad (4)$$

Where:

M: moisture content, kg water/kg dry mass.

m_0 : monolayer moisture content, kg water/kg dry mass

C: Guggenheim constant, product characteristics and related to monolayer heat sorption.

k: Correction factor related to multi-layer heat sorption.

a_w : water activity

2.6. Differential Scanning Calorimetry.

The glass transition temperatures (T_g) of the treatments at different concentrations of native agave fructans were estimated using a calorimeter (DSC, Q2000 TA-Instruments, New Castle, DE, USA). In general, 4 milligrams of sample equilibrated at 25 °C were heated in hermetic aluminum crucibles. A heating ramp of 20 to 200 °C at 10 °C/min was used. An empty hermetically sealed aluminum crucible was used as reference. The DSC was calibrated with metallic Indian standard for temperature. The calorimeter was purged with nitrogen at a flow rate of 50 mL/min. All measurements were made in triplicate. The data were analyzed using the Universal Analysis 2000 software, version 4.7a (TA Instruments, New Castle, USA). The T_g was calculated by evaluating the midpoint of the inflection region in the heat flow signal.

2.7. Fourier-Transform infrared spectroscopy (FTIR)

The transmittance spectroscopic analysis by FTIR was carried out to detect characteristic chemical functional groups in the powders. A small mass of each of powder and compressed into small discs. A spectrophotometer (Perkin Elmer, Spectrum 1000, USA) was used. The spectra were obtained using 32 scans per sample, with a 2 cm^{-1} resolution in the 400–4000 cm^{-1} range.

3. RESULTS AND DISCUSSION

3.1. Effect of fructans on physicochemical properties

The results showed that the physicochemical properties depend on the structure and composition of the native agave fructans that were used as stabilizers in the drying process. The formulation with better physicochemical characteristics such as, moisture, solubility wettability, bulk density, a_w , Hausner Index, hygroscopicity, was obtained at 120 °C with 10% of maltodextrin. The values obtained for the physicochemical properties with the temperatures of 110 and 115 °C, as well as with concentrations of 5 and 7% of maltodextrin were higher than those obtained with this formulation (data not shown). The addition of 4% (w/v) of native agave fructans shown lower values in the physicochemical properties and increase in the solubility (Table 1). The chayote powders, added with mixtures of maltodextrins and fructans presented a low hygroscopicity. The spray dried powders presented very thin particle in comparison with the other types of drying that result in porous and brittle particles (10-200 μm)¹⁹.

3.2. Moisture and water activity

Moisture content is one of the factors related to drying efficiency²⁰. Moisture content of chayote powders varied from 1.71 to 2.57% wb (Table 1), which were very close to values reported by Fazaeli et al²¹ for spray dried mulberry and bayberry. For moisture content, it was observed that the inlet temperature and agave fructans concentration had an important effect on moisture content. However, the moisture content of chayote powder decreased when the carrier agent concentration increased. According to Fazaeli et al²¹ these findings could be explained by the fact that additional concentrations of the carrier agent resulted in an increase in feed solids and a reduction in total moisture for evaporation. The effects of independent variables on water activity are in agreement with the effects of them on moisture content. The results showed the moisture content of the spray dried powders decreased with the increase in inlet air temperature and the increase in the concentration of fructans. These values are very close to the values reported by Fang and Bhandari²². The values fall within those commonly observed in industrial spray drying²³. All powders with the addition of fructans showed water activity below 0.20, which confirmed all of the powders were chemically stable. Moreover, powders produced with different agave fructans concentration had significant differences in a_w and moisture content values, comparing with treatments containing maltodextrin only. Similar results were reported for persimmon pulp²⁰.

The highest temperature in this study (120 °C) shown the lowest values of moisture and a_w . Using higher inlet temperature offers higher energy to drying medium and increase the heat transfer. However, using very high temperature may cause instant crust formation

on the surface of the particle which led to difficulties in water diffusion. Therefore, care must be taken when choosing the drying temperature. As a general rule, increasing inlet temperature results in lower moisture content of the product ^{24,25}.

3.3. Hygroscopicity determination

Hygroscopicity is an index of water absorption relating to the storage stability of products. The hygroscopicity of powders obtained with agave fructans as a carrier agent ranged from 10 to 13%, this results indicating that the chayote powders had low hygroscopic, similar results were observed in the case of blackberry powder ²⁶ and pitanga *Eugenia uniflora* L. powder ²⁷. Increases in inlet air temperature and maltodextrin concentration as well as fructans led to a lower hygroscopicity. This observation is in accordance with those of other researchers in mango and pineapple powder ²⁸ respectively. The moisture content behaviour keeps a direct relation with the hygroscopicity profile, as shown in Table 1. Tonon et al ²⁹ mention that spray dried açai (*Euterpe oleraceae* Mart.) with low moisture content has a greater capacity to absorb water in the surrounding air and therefore is more hygroscopic. Ahmed et al ³⁰ reported that the hygroscopicity of spray dried sweet potato was affected by the stabilizers, without having a direct relation to moisture content. According to Nurhadi et al ³¹ powder having less than 20% are classed as not very hygroscopic. The powder hygroscopicity could be affected by its inherent compositions, carrier agent type and concentration, moisture content and particle size ³². Increasing the agave fructans concentration resulted in decreased hygroscopicity with the whole range of inlet air temperature tested during spray drying. Ferrari et al ³² showed a similar effect in the hygroscopicity of blackberry powder. On the other hand, by increasing the concentration of fructans, the chayote powder presented a higher hygroscopicity during the storage evaluated after 2 months. Powders containing lower moisture content had a greater capacity to absorb environment humidity, which is related to the higher water concentration gradient between the product and the surrounding air. Hygroscopicity of the powder is generally related to its composition, type and carrier agents concentration and powder size ^{20,33}.

3.4. Bulk density and solubility

Bulk density of the chayote powders varied from 0.55 to 0.62 g/mL. Increasing inlet air temperature caused a reduction in bulk density and an increase in the solubility of chayote powders. There is an inverse relation between the bulk density and solubility of powders. This is consistent with the findings of a number of studies ^{34,35}. At high temperatures, evaporation rates are faster, products dry to a more porous or fragmented structure, implying a lower shrinkage of the droplets, and so a lower density of the powder. The increase in the concentration of fructans had a significant effect causing a decrease in the moisture content, due to an increase in the feed solids. This increment allowed

a higher degree of dehydration of the product and a decrease in the bulk density due to the lesser moisture content of the products. The sticky nature of a powder is associated with a high bulk density ³⁴ because the particles that tend to stick together leave less space between them and consequently result in a smaller bulk volume. The bulk density decreases as the fructans and maltodextrin concentration increases because adding stabilizers minimizes the number of thermoplastic particles that stick (Table 1). In addition, an increase in additive concentration may cause an increase in the volume of air trapped in the particles formed and spray dried particles often contain air bubbles, which can occur as a result of the deposition 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.

The Increase of carrier agent concentration caused an increase in powder solubility. This may be attributed to the fact that maltodextrin and fructans had superior high water solubility and is mainly used in process of spray drying due to its physical properties, such as high solubility in water ³⁶. Solubility is an inverse function of the inlet air temperature and the fructans concentration. The solubility percentage of chayote powders varied from 94 to 99%. An increase in the agave fructans concentration was only significant when used the inlet air temperature of 120 °C which showed lower moisture. This can be attributed to the fact that low-moisture content seems to be associated with fast rehydration. Also the solubility of a powder is influenced by properties of raw materials, carrier agents materials, and properties of the powders such as moisture content, particle size and physical state of the particle (amorphous state is more soluble) ²⁰.

Hausner ratio (HR) values of the produced chayote powders ranged from 1.03 to 1.11 (Table 1). According to Geldart et al ³⁷ the classification, powders with HR values below 1.25 are referred to as free flowing with low cohesiveness, while powders with HR values within the range of 1.25–1.4 are of average cohesiveness, and powders with HR higher than 1.4 are highly cohesive. Cohesiveness of powders determines their consistency and flow properties, the lower the cohesiveness the better the flowability of powders, so that with the HI obtained the chayote powder, is considered with excellent flow properties.

3.5. Wettability analysis

The results showed that at lower air inlet temperature during the course of drying, less time is required for the powder to become fully wet. Wettability is correlated with the moisture content of the powder. Reconstitution of the powder increases with increased moisture content. At higher temperatures a hard surface might form, thus preventing the easy diffusion of water molecules through the particles ³⁵. The combination maltodextrin and fructans influenced the hygroscopicity of the spray dried powders, it reduced the powder's water adsorption

capacity, such as water activity and wettability due to its less hygroscopic nature²⁸.

3.6. Color determination

There is a direct relation between the inlet temperature and the hue angle (h°). This indicates that there was a decrease in green color when the inlet temperature was increased. In other words, there is a substantial degradation of pigments at higher temperatures. The results found in Table 2 can be used to determine the color loss in the obtained powders with respect to the feed juice entered in the spray dryer. Paleness can be observed in the final product which can be attributed to the loss of the juice pigments when reaching temperatures higher than 60 °C. The resultant chayote powders show light green color, which is demonstrated by the luminosity values (L^*) closer to 100 and lower levels in a^* , which represent the parameter green to red. The tone hue angle of the powder had low values, which also indicates a trend for white. This is due to the fact that outlet temperatures of the drying process were found in a range between 70 and 80 °C, the own fruit sugars did not suffer high oxidation because the process time was not very long. This effect could be attributed to the carrier agents added, because during the powder rehydration the color is intensified because the stabilizer is solubilized allowing the release of the pigments³⁸.

3.7. Differential Scanning Calorimetry

The glass transition temperature (T_g) is the main characteristic in the transformation of the amorphous phase. Figure 1 shows thermograms of encapsulated chayote powders. The highest glass transition temperature showed in the treatment of chayote powder dried at 120 °C, 10% of maltodextrin and 4% of agave fructans concentration (w/v). An increase of T_g was observed when the fructans concentration was increased, 48.04, 49.68 and 52.95 °C for 0, 2 and 4% fructans concentration, respectively. This behaviour conferred techno-functional properties as carrier agent to fructans used in this study. An increase in inlet air temperature and agave fructans concentration represented an increase in powder T_g . According to Goswani et al¹⁰ T_g decreased by increasing moisture content due to the plasticizing effect of water. The thermograms obtained through DSC analysis of chayote powder presented curves with the similar profile for all treatments with different concentrations of fructans (Figure 1). In all the obtained curves, a typical second-order transition was observed; identified by the fall in the curve that demonstrates that a variation in specific heat (ΔC_p) exists; this change in heat capacity causes a difference in the heat flow of the sample which is characteristic for glass transitions. The thermal footprint of the samples was different in each case where a slide of the inflection area as well as the area of relaxation enthalpy with respect to the addition of fructans was observed. The values denoted the increment of the T_g when water activity and moisture content decreased, according to the plasticizing effect of water. A similar profile was observed

by various authors that evaluated other fruits such as carrot-celery and tomato³⁹. The values of T_g obtained in this study indicated a positive effect of the addition of fructans since these had a higher molar mass than maltodextrin and the T_g was positively related to the molar mass. These results match with those reported by De Barros Fernandes et al⁴⁰ where addition of different encapsulating materials, among maltodextrin (DE 10) gave the lowest values for T_g , because it was the coating agent with the lowest molar mass (1800 g mol⁻¹). Also the T_g depends on the degree of ramifications of the molecules. Meanwhile, a combination with inulin that belongs to the fructans group increased the T_g .

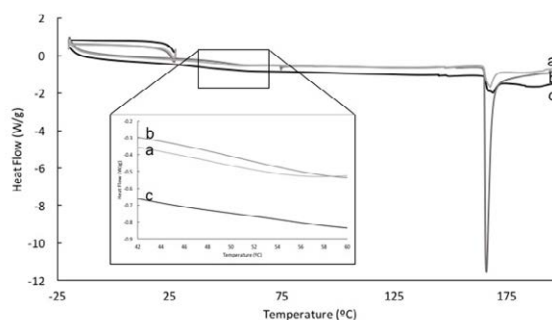


Figure 1. Thermograms (T_g) for treatments at 0% (a), 2% (b) and 4% (c) of agave fructans.

3.8. Moisture sorption properties of chayote powder

The estimated values of the coefficients and the statistical parameters used to evaluate the adequacy of the models for the moisture adsorption behaviour are shown in Table 3. The equilibrium moisture content in the treatments formulated with fructans was greatly increased when exposed to relative humidity higher than 0.60. On the other hand, the equilibrium moisture content of the treatments containing maltodextrin remained relatively constant, with the relative humidity variation, and not for the treatments applying fructans and maltodextrin. At low water activities values, water can be adsorbed on the surface of carbohydrates at the polar sites, where as at high water activity values, the dissolution of carbohydrates occurs¹².

The model that best fit to the variations in isotherm data for all the treatments was the GAB model, when considering both the lowest value of the average relative deviation and the highest coefficient of determination (R^2). Regarding the GAB parameters, the X_m or moisture content of the monolayer of fructans and fructans plus maltodextrin were higher than treatments with only maltodextrin, indicating the increased availability of active sites bonds with water¹². For powders elaborated with 4% fructans, lower hygroscopicity values were observed than powders with only maltodextrin. The GAB model provides important information about food products because the moisture content of the mono-layer (X_m) estimation is important to establish appropriate storage conditions for dehydrated foods. The physical characteristics of the powders, subjected to different humidity levels, demonstrated that there

was no apparent phase change in the particles produced because of fructans addition. The powders with fructans and maltodextrin plus fructans remained in the glassy state during analysis and storage. These results suggest that the fructans are an interesting carrier material of spray drying processes. For better particle conservation and stability, lower water absorption values were desirable; these conditions improved also the reconstitution properties, while reducing the caking occurrence.

The study of water sorption covers two phenomena: adsorption and desorption. The desorption isotherm is always larger than the adsorption isotherm, forming a loop called hysteresis that generally decreases when the temperature of the isotherm increases⁴¹. The hysteresis loop is observed beginning at the monolayer in the a_w range from 0.18 to 0.90 (Figure 2). The hysteresis curves can be variable for different foods⁴²; even identical foods can present different isotherm patterns, when a variable in the process is altered. This diversity can be interpreted as a consequence of the variability that comes with different concentrations of chemical components, as well as some changes produced in the factors influencing capillary porosity characteristic for foods. This hysteresis loop presented in foods' sorption isotherms as in chayote powders is generally influenced by factors; such as isotherm generation temperature. Considering that for two sorption isotherms obtained at different temperatures, different hysteresis loops have been observed for the same product, the existence of an exothermic phenomenon in moisture adsorption provides an explanation. Increasing the isotherm's temperature translates to a loss of moisture at equilibrium with a given relative humidity, with an approximation of the isotherm towards the axis of water activity with the same moisture content⁴³.

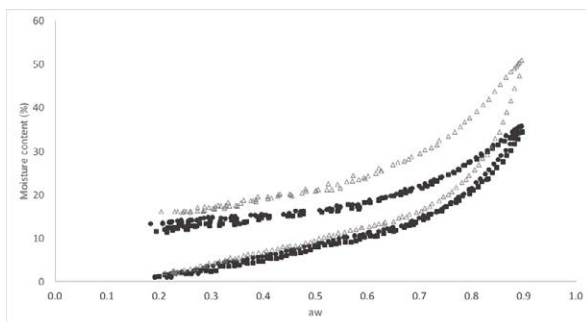


Figure 2. Chayote isotherms at different agave fructans concentration (■) 0%, (●) 2%, (△) 4%.

3.9. Microstructure and glass transition temperature

The microstructure of the powders produced with the addition of fructans showed amorphous spherical particles with contractions (Fig. 3). The micrographs of the microcapsules obtained with maltodextrin presented rounded external surfaces with cavities and dents, probably due to the mechanical atomization, air-drop interaction and capsule cooling, occurring during spray drying process⁴⁴. Additionally, particles

agglomeration with a strong attraction among the microcapsules was observed. This can be explained by the fructans ramified molecular structure, which decreases the moisture content and incremented the T_g ; causing a possible interaction with maltodextrin whose structure is linear. Spray dried chayote powders produced with fructans has higher T_g point due to its larger molecule comparing with powders with only maltodextrin. The T_g is the characteristic transformation temperature of the amorphous phase. The event of T_g occurs when a sugar that is hard, solid, and amorphous suffers a transformation into a liquid, soft or rubbery phase. The glass transition temperature is observed as a gradual variation in heat flow (ΔC_p)⁹. According to the micrographs of this study, the particles with the least caking and highest T_g were found in the powders produced by a mixture of maltodextrin and fructans.

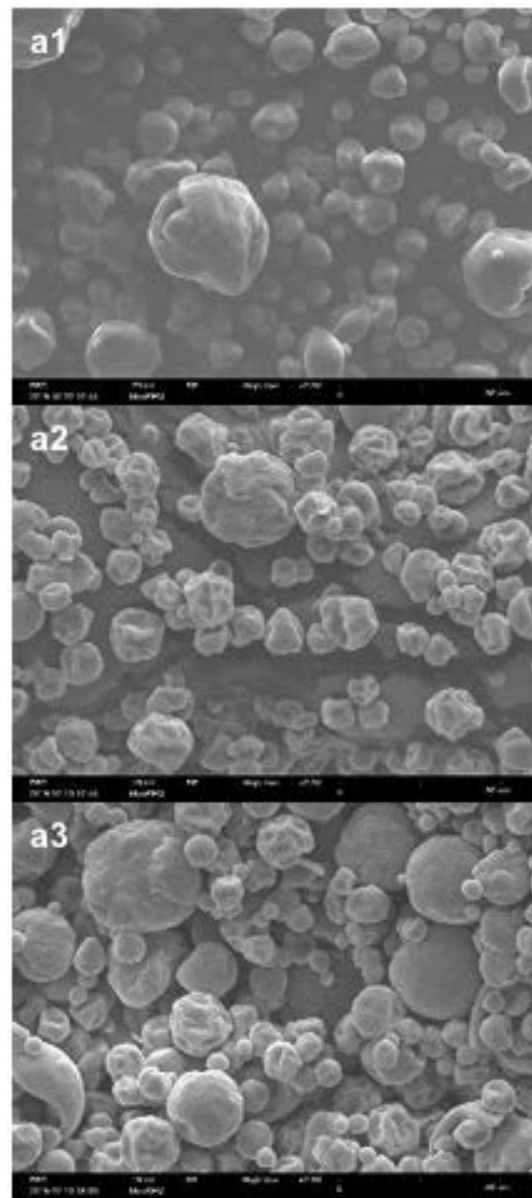


Figure 3. Photographs of scanning electron microscopy of chayote powder. a1) 10% MD y 0% FT, a2) 10% MD y 2% FT a3) 10% MD y 4% FT.

3.10. FT- IR spectroscopic studies

The FTIR analysis provided information about the chemical bonds and structure of the compounds ⁴⁵. FTIR spectra obtained for different chayote powders shown the same pattern (Fig. 4), then a compatibility can be presumed. In fresh juice chayote, a peak at 3339 cm^{-1} was observed which is also assigned to $-\text{OH}$ stretching. Peaks in the region from 3700 to 3000 cm^{-1} correspond to water and OH absorption frequencies ⁴⁶. The peak near 1630 cm^{-1} in fresh juice chayote represent $\text{C}=\text{O}$ stretch and its conjugation with $\text{C}=\text{C}$, bands that appear at 1743 and 1630 cm^{-1} can be assigned to $\text{C}=\text{O}$ stretching vibration of methyl esterified carboxylic group and vibrations of the carboxylate ($-\text{COO}-$) group, respectively. The broad absorption of $\text{C}=\text{O}$ at 1680–1630 cm^{-1} partially overlaps with $\text{N}-\text{H}$ absorption band at 1640–1620 cm^{-1} , reported for primary and secondary amides. FTIR spectrum of the powders shown two characteristic peaks, one in a range 3222 cm^{-1} and 3199 cm^{-1} for O-H group attached to the aromatic ring. The other peak at 988 and 990 cm^{-1} . The bands in the region 900–1153 cm^{-1} have been assigned to $\text{C}-\text{O}$ and $\text{C}-\text{C}$ stretching modes. This band is characteristic of carbohydrates, which showed high absorbance between 1200 and 950 cm^{-1} ⁴⁷. The peaks appearing in individual components spectra matched with picks obtained for microcapsule spectrum, indicated that no structural changes have occurred between components and microcapsules.

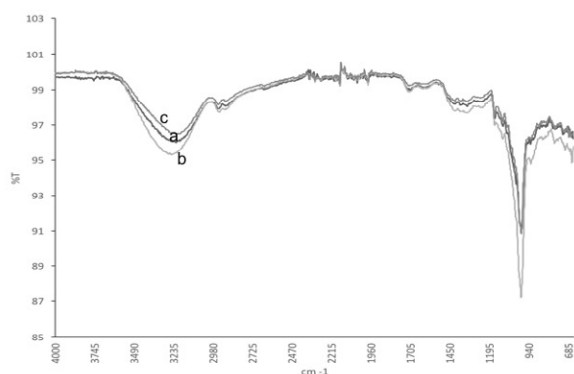


Figure 4. FTIR spectra of chayote at different agave fructans concentration (a) 0%, (b) 2% and (c) 4%.

4. CONCLUSIONS

The combination of concentration different of native agave fructans with maltodextrin as stabilizers improved the physicochemical properties in spray dried chayote powder. The addition of native agave fructans contributed to a significant decrease in the mass fraction of maltodextrin added in this type of industrial products. High concentrations of agave fructans decreased bulk density and increased solubility, the yield, hygroscopicity and glass transition temperature as positive effects. The obtained results indicate that the agave fructans concentration of 4% is an excellent choice as an encapsulating additive. The isotherms

showed evidence of a non-porous food or a food with micropores. The sorption properties were modified in the multi-layer region as a result of physical adsorption. The mixture of maltodextrin and fructans at 10 and 4%, respectively improved solubility, bulk density, as well as a high glass transition temperature.

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