



DETERMINATION OF TOTAL AND INCIPIENT SOLUBILIZATION POINT OF FRUCTANS EXTRACTED OF *A. tequilana* Weber var. azul

DETERMINACIÓN DEL PUNTO DE SOLUBILIZACIÓN TOTAL E INCIPIENTE DE FRUCTANOS EXTRAÍDOS DE *A. tequilana* Weber var. azul

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Abstract

The aim of this work was to explain the effect of water in powder of fructans extracted of *A. tequilana* Weber var. azul, through the thermodynamics functions to know physical stability and suggest storage conditions. The results of thermodynamics analysis were confirmed with Environmental Scanning Electron Microscopy (ESEM). Adsorption isotherms of agave fructans were realized at 15, 25 and 45°C. ESEM was performed at the samples equilibrated in a range of water activitie (a_w) from 0.1 to 0.9. The results showed that the beginning of an incipient solubilization takes place at $a_w = 0.54$ and the total solubilization takes place at $a_w = 0.77$.

Keywords: fructans, thermodynamic analysis, water adsorption, solubilization, enthalpy.

Resumen

El objetivo de este trabajo fue explicar el efecto del agua en los polvos de fructanos extraídos de *A. tequilana* Weber var. azul mediante análisis termodinámico para conocer su estabilidad física y sugerir condiciones de almacenamiento. Los resultados del análisis termodinámico fueron confirmados con Microscopía Electrónica de Barrido Ambiental (MEBA). Las isotermas de adsorción de los fructanos de agave fueron realizadas a 15, 25 y 45°C. La MEBA fue realizada a las muestras equilibradas en un rango de actividad de agua (a_w) desde 0.1 a 0.9. Los resultados mostraron que el comienzo de la solubilización incipiente sucedió a $a_w = 0.54$ y la solubilización total sucedió a $a_w = 0.77$.

Palabras clave: fructanos, análisis termodinámico, adsorción de agua, solubilización, entalpía.

1 Introduction

Fructans are a heterogeneous mixture of fructose polymers linked by fructose-fructose glycosidic bonds (Wack and Blaschek, 2006). Due the structure and type of link (β 2-1 and β 2-6) of these molecules, fructans are considered as prebiotics (Roberfroid, 2005) which are being added to foodstuffs in order to improve texture or provide functional properties (Kip *et al.*, 2006; Moreno-Vilet *et al.*, 2014). In Mexico, fructans are isolated from the agaves, as the main photosynthetic product stored in the stem of these plants. Waleckx *et al.* (2008) reported that

fructans of *A. tequilana* Weber var. azul consist of a complex mixture, principally containing highly branched fructans and neo-fructans mixed fructans group. Traditionally, the stems of *A. tequilana* are applied in the tequila production; recently, they have been used in the production of highly pure fructans and fructo-oligosaccharides (FOSs) for novel healthy food products (Santos- Zea *et al.*, 2012).

Therefore, it is important to know the interaction between water molecules and the fructans in order to prevent spoilage reactions, collapse and cake of sugars in the fructans-products, and thus determine the better storage conditions improving stability. The role of water in food depends on the chemical composition, physical structure, and form in which it is present in a

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food matrix. Knowledge of the vapor pressure of foods as a function of moisture content and temperature is essential for the control of water content during processing, handling, packaging and storage (Azua and Beristain, 2006).

Stability of foods is mainly consequence of the relationship between equilibrium moisture content of the food material, and its corresponding water activity, at a given temperature (Myhara *et al.*, 1998). So it is greatly influenced by the moisture sorption characteristics of the product. Sorption characteristics of food are important in design of drying processes and equipment, shelf-life studies, and packaging requirements (Johnson and Brennan, 2000).

The thermodynamic of dried-foodstuffs water sorption has drawn interest, because it provides additional detailed interpretation of the sorption isotherm phenomenon, and helps to understand the sorption mechanism (Beristain, 2002). The thermodynamic functions are readily calculated from sorption isotherms, which enable interpretation of experimental results in accordance with the statement of the classic theory (Iglesias, Chirife, and Viollaz, 1976; Arslan and Togrul, 2006).

In thermodynamic studies, essential functions are enthalpy and entropy to determine food stability. The isosteric heat or differential enthalpy of sorption is a differential molar quantity derived from the temperature dependence of the isotherm, and it represents the energy for water molecules binding at a particular hydration level, in contrast to the integral heat, which is the average energy of all molecules already bound at that level (Schneider, 1981; Beristain *et al.*, 2002).

The differential entropy of a material is proportional to the number of available sorption sites at a specific energy level (Madamba, Driscoll and Buckle, 1996) and integral entropy describes the degree of disorder and randomness of motion of water molecules (Mazza and LeMaguer, 1978; Rizvi and Benado 1984; Beristain *et al.*, 2002).

The aim of this work was to explain the effect of water in powder of agave fructans, through the thermodynamics functions and confirm them with ESEM results.

2 Material and methods

2.1 Raw material

Fructans of agave were provided by “Inulina y Miel de Agave, S.A. de C.V.” (Jalisco, Mexico).

2.2 Adsorption isotherms

Sorption isotherms were performed by using the gravimetric method proposed by Lang *et al.* (1981). To achieve almost zero moisture, the fructans powder was placed in vacuum desiccators containing P₂O₅ until constant weight was achieved. After that, 1-2 g samples were placed in triplicate in desiccators with salt solutions in a range from 0.103 to 0.892 water activity, salt solutions were prepared according to Labuza *et al.* (1985) The samples were stored at 15, 25 and 45 °C to achieve equilibrium. Every three days the samples were weighed on an analytical balance (OHAUS Analytical Plus, AP210S, USA) until constant weight was achieved. The equilibrium moisture content on dry-basis was determined for each water activity. Isotherms were determined by triplicate, and fitted to the model of Guggenheim-Anderson-De Boer (GAB) using KaleidaGraph v. 4.1 software (Synergy software, Reading PA, USA) (Weisser, 1985):

$$M = \frac{M_0 C K a_w}{(1 - K a_w)(1 - K a_w + C K a_w)} \quad (1)$$

Where M is the equilibrium moisture content (g of water/100 g of dry solid), a_w is the water activity, M_0 is the monolayer moisture content, C and K are constants related to temperature as follows:

$$C = c \exp[(hm - hn)RT_a] \quad (2)$$

$$K = k \exp[(h_i - h_n)/RT_a] \quad (3)$$

Where c and k are entropic accommodation factors; hm , hn and h_i are molar sorption enthalpies of the monolayer, the multilayers on top of the monolayer, and the bulk liquid, respectively (J/mol); while R (J/mol K) is the ideal gas constant and T_a (K) is the absolute temperature. C , K and M_0 , were obtained from the fitted GAB model data by using KaleidaGraph software (Synergy software, Reading PA, USA).

Goodness of fit was evaluated using the average of the relative percentage difference between the experimental and predicted values of the moisture

content or mean relative deviation modulus (P), defined by the following equation (Lomauro *et al.* 1985):

$$P(\%) = \frac{100}{N} \sum_{i=1}^N \frac{|Me_i - Mc_i|}{Me_i} \quad (4)$$

Where Me_i is the experimental moisture content; Mc_i is the calculated moisture content; and N is the number of experimental data.

2.3 Thermodynamic properties

2.3.1 Differential properties

Thermodynamic properties were determined with fitted data to the GAB equation. Changes in differential enthalpy at the water-solid interface at different stages of the adsorption process were determined using Othmer equation (1940):

$$\ln P_v = \left[\frac{H_v(T)}{H_v^0(T)} \right]_M \ln P_v^0 + C \quad (5)$$

where P_v is vapor pressure of water in the food; P_v^0 is vapor pressure of pure water at the same temperature; $H_v(T)$ is isosteric heat for water adsorption; $H_v^0(T)$ is the heat of condensation of pure water; M is moisture; and C is adsorption constant.

A plot of $\ln P_v$ against $\ln P_v^0$ gives a straight line if the ratio $H_v(T)/H_v^0(T)$ is maintained constant in the range of temperatures studied.

The net isosteric heat of adsorption or differential enthalpy is defined by Eq. 6.

$$(\Delta H_{dif})_T = \left[\frac{H_v(T)}{H_v^0(T)} - 1 \right]_M H_v^0(T) \quad (6)$$

Calculating $H_v(T)/H_v^0(T)$ with Eq. 5 and substituting into Eq. 6, it is possible to estimate the net isosteric heat of adsorption at different temperatures using steam tables.

With values obtained for enthalpy changes, the variation in the molar differential entropy $(\Delta S_{dif})_T$ may be estimated using Eq. 7:

$$(\Delta S_{dif})_T = S_1 - S_L = -\frac{(\Delta H_{dif})_T}{T} - R \ln a_w \quad (7)$$

where $S_1 = (-\partial S / -\partial N_1)_{T,P}$ is molar differential entropy of water adsorbed in the food. S_L is molar entropy of pure water in equilibrium with the vapor. S is total entropy of water adsorbed in the food. N_1 is number of moles of water adsorbed in the food. R is universal gas constant. a_w is water activity, and T is absolute temperature (K).

2.3.2 Integral properties

Molar integral enthalpy is calculated using an expression similar to that for differential enthalpy, maintaining diffusion pressure constant:

$$\ln P_v = \left[\frac{H_v(T)}{H_v^0(T)} \right]_\varphi \ln P_v^0 + A \quad (8)$$

Where A is the adsorption constant and φ (J/mol) is the pressure of diffusion or surface potential. A plot of $\ln P_v$ versus $\ln P_v^0$ gives a straight line if the ratio $H_v(T)/H_v^0(T)$ is constant within the range of temperatures used.

The molar integral enthalpy $(\Delta H_{int})_T$ can be calculated using Eq. (9), at constant pressure of diffusion (Beristain *et al.*, 1994).

$$(\Delta H_{int})_T = \left[\frac{H_v(T)}{H_v^0(T)} - 1 \right]_\varphi H_v^0(T) \quad (9)$$

$$\varphi = \mu_{ap} - \mu_a = RT \frac{W_{ap}}{W_v} \int_0^{a_w} M d \ln a_w \quad (10)$$

Where μ_a (J/mol) is the chemical potential of the adsorbent participating in the condensed phase. μ_{ap} (J/mol) is the chemical potential of the pure adsorbent. W_{ap} (g/mol) is the molecular weight of the adsorbent. W_v (g/mol) is the molecular weight of water.

By calculating $H_v(T)/H_v^0(T)$ from Eq. (8) and substituting it into Eq. (9) it is possible to calculate the integral enthalpy at different temperatures, provided that a good means of $H_v^0(T)$ estimating is available, such as that proposed by Wexler (1976):

$$H_v^0(T) \text{ J/molK} = 6.15 \times 10^4 - 94.14T + 17.74 \times 10^{-2} T^2 - 2.03 \times 10^{-4} T^3 \quad (11)$$

By using the obtained values for $(\Delta H_{int})_T$, it was possible to estimate the changes in the integral molar entropy $(\Delta S_{int})_T$ by means of the following equation:

$$(\Delta S_{int})_T = S_S - S_L = -\frac{(\Delta H_{int})_T}{T} - R \ln a_w \quad (12)$$

Where $S_S = S/N_1$ (J/mol K) is the integral entropy of water adsorbed in the material. S (J/mol K) is the total entropy of water adsorbed. N_1 is the moles of water adsorbed in the sample and S_L (J/mol K) is the molar entropy of pure liquid water in equilibrium with vapour.

2.4 Microstructure by Environmental Scanning Electron Microscopy (ESEM)

An environmental scanning electron microscope (ESEM, XL 30, Philips, USA) was used for the observation of the microstructure of fructans powder without the dehydration effects and gold coating. This operation mode allows the examination of wet biological specimens without sample preparation (Barrera *et al.*, 2013). Eight fructans samples equilibrated at different water activities (0.12, 0.23, 0.32, 0.44, 0.54, 0.65, 0.77, 0.85) were sprinkled onto double-sided tape attached to the specimen stubs. The samples were observed at 5 kV using the gaseous secondary electron detector. The micrographs were captured in 32 bits (RGB) and crops of 712×484 pixels were stored in TIFF format. Observations of the samples at magnifications of 350x were acquired for image analysis.

3 Results

3.1 Adsorption isotherms

Figure 1 shows the experimental data (markers) and the fitted data (lines) of water vapor adsorption isotherms at 15, 25 y 45°C. The experimental data were fitted with GAB equation. The equilibrium moisture content increased with increasing water activity following the Type II sigmoid-shaped isotherm, according to Brunauer's classification (Viveros-Contreras *et al.*, 2013).

A clear-cut inversion was, however, noticed in $a_w = 0.6$ approximately, where starts an endothermic process, implying a higher equilibrium moisture content at higher temperatures of sorption. This behaviour is typical of sugar-rich food systems and it is probably caused from the gradual dissolution of sugar, which results in complete leaching of sugar in solution. Fructans are a heterogeneous mixture of fructose polymers, that can become very hygroscopic due to the presence of short-chain fructans. Iglesias and Chirife (1982) indicated that some food became less hygroscopic at higher temperatures. Conversely, the equilibrium moisture content of the fructans samples increased with increasing temperature at water activity values greater than 0.6. It has been suggested that low molecular weight food constituents, such as salts and sugars, become more hygroscopic at increased temperatures thereby

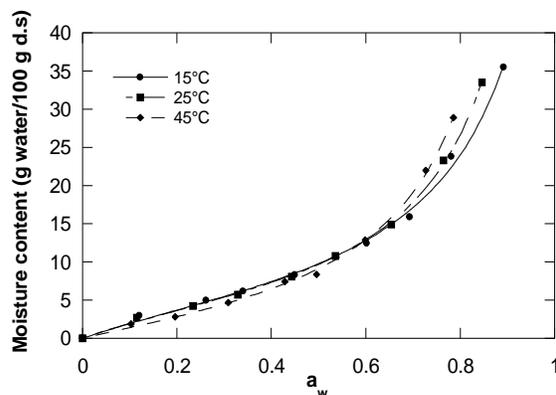


Fig. 1 Adsorption isotherms of Agave Fructans at 15, 25 and 45°C.

Table 1. Estimated parameters of the GAB model fitted to the adsorption data for Agave Fructans

Parameters	Temperature (°C)		
	15	25	45
M_0 (% d.b.)	7.46	6.74	6.63
C	3.08	3.34	1.98
K	0.9	0.96	1.01
P (%)	1.05	1.83	0.93

offsetting the latter temperature effects (Kapsalis, 1987).

This behavior has been reported in the literature for many high-sugar content foods, such as apricots, grapes, apples, pestil (Kaymak-Ertekin and Gedik, 2004; Kaya and Kahyaoglu, 2005) and orange peels (Kammoun *et al.*, 2012).

Table 1 shows the estimated parameters of the GAB model fitted to the sorption data for fructans (M_0 , C, K). M_0 is the moisture content at the monolayer, which was 6.74g water/100g dry solids at 25°C ($a_w = 0.36$). It has been assumed that the monolayer value is the saturation of polar groups corresponding to water adsorbed at the most active sites (Beristain *et al.*, 2002; Pérez-Alonso *et al.*, 2006). Espinosa-Andrews and Urias-Silvas (2012) stored agave fructans in different water activities and, they showed a value of $M_0 = 7.3$ g water/100g dry solids at 20°C, according to the GAB model. C is a constant related to the adsorption energy of the water by the food, in this case C was 3.34 at 25°C, means that attraction forces between the adsorbate and the adsorbent are larger than the attraction forces between adsorbate molecules in the liquid state and K is a constant that relates the

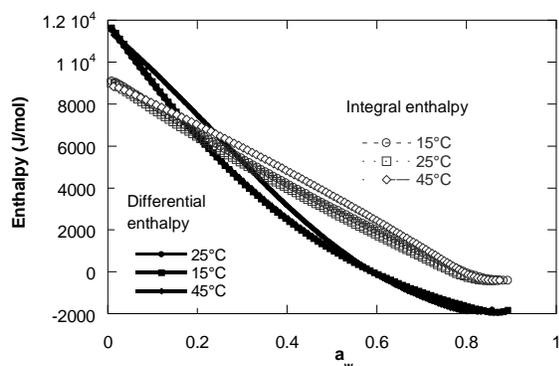


Fig. 2 Integral and differential enthalpy of Agave Fructans.

adsorption energy of the multilayer with the surface of the food (Velazquez-Gutierrez *et al.*, 2015). P (%) indicated a good fit of adsorption data because the values were minor than 3% (Lomauro *et al.*, 1985). Pascual *et al.* (2017) used Gab equation to model the water adsorption process in different dehydrated foods: sucrose-calcium powder, pineapple powder and capsules of paprika oleoresin, in which they reported a well fit with $P < 10\%$.

3.2 Differential and integral enthalpy

Figure 2 shows differential and integral enthalpy at different water activities. At low water activities, the differential enthalpy values of agave fructans was high, and then the values decreased until zero. The physical explanation for the rapid increase in heat isosteric at low moisture contents is that, in the initial stages of sorption (low moisture content), there were highly active polar sites on the surface of the food material, which were covered with water molecules to form a monomolecular layer (Al-Muhtaseb *et al.*, 2004). The decrease in the differential enthalpy was probably because the newly arriving water vapor molecules interacted with other alternative lower energy active sites, leading eventually to the formation of multilayers (Bonilla *et al.*, 2010).

A positive value for ΔH was expected as a result of the exothermic interaction of the water molecules with the sorption sites on the surfaces of the powders at low water activities. However, negative values was expected when is present an endothermic process. A negative value is usually expected for ΔH due to the weaker bonding of the multilayer molecules (Gabas, Menegalli, and Telis-Romero, 2000; Tsami *et al.*, 1990). Iglesias and Chirife (1976) explain that moisture contents with negative isosteric heat values

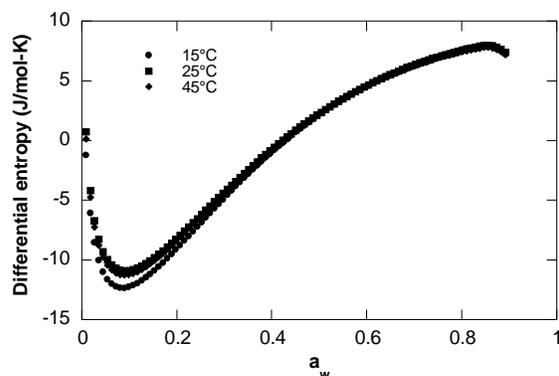


Fig. 3 Differential entropy of Agave Fructans.

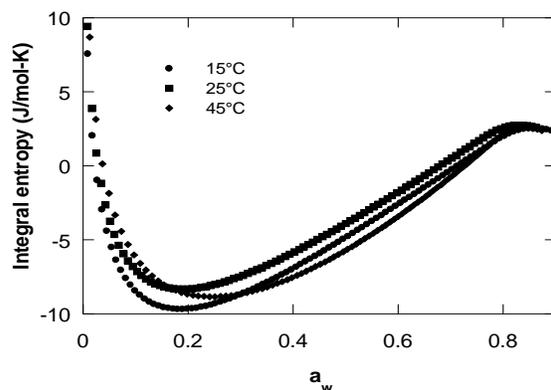


Fig. 4 Integral entropy of Agave Fructans

indicate the presence of free water. The decreased of interaction energy between the water and food surface has been related to the endothermic dissolution of sugars in the absorbed water (Maroulis *et al.*, 1988; Quirijns *et al.*, 2005b).

ΔH has been calculated by differents authors in certain fruits with high sugar contents such as orange juice powder (Sormoli and Langrish, 2015) and dried fruits (Kaymak-Ertekin and Gedik, 2004; Tsami *et al.*, 1990) obtaining very low isosteric heat values at water activities higher than 0.6.

In conclusion, the high value of isosteric heat in the initial stages of adsorption of the agave fructans, suggests a monomolecular physical sorption on highly active polar sites. Its diminution in higher moisture content indicates that the sorption occurs in multilayers on less active sites. When isosteric heat was close to zero, the beginning of an incipient solubilization takes place ($a_w=0.54$) and the negative values has been expected for heat isosteric due to the weaker bonding of the multilayer molecules.

The behavior of integral and differential enthalpy

was similar, since the equilibrium heat increased sharply to a maximum in the lowest moisture content (9 kJ/mol and 11 kJ/mol for integral enthalpy and differential enthalpy, respectively); and then gradually decreased in magnitude with further increase in moisture content. However, integral enthalpy decreased to values close zero in $a_w = 0.77$, where occurred the total solubilization of fructans, which is possible to confirm by ESEM (Figure 5).

Increasing of integral enthalpy at low moisture content can be explained by the location of the bound water in the most active available sites, where occurs the greater water-solid interaction compared to the interaction of the water-solid at higher moisture content, the enthalpy values at lowest water activities indicate the occupation of highly active sites (Wang and Brennan, 1991).

3.3 Differential and integral entropy

Differential and integral entropies changed with respect to moisture content at 15, 25 and 45 °C for agave fructans (Figure 3 and 4), but moisture content values corresponding to minimum integral entropy and minimum differential entropy are different. Figure 3 shows that differential entropy had a minimum at $a_w = 0.09$ (2.01 g water/100 g dry solids) at 25°C, and Figure 4 shows that integral entropy had a minimum at $a_w = 0.19$ (3.46 g water/100g dry solids) at 25°C. The differential entropy represents the algebraic sum of the integral entropy at a particular hydration level, plus the change of order or disorder after new water molecules were adsorbed by the system at the same hydration level. Nevertheless, this parameter does not mean order or disorder of the total system. If the moisture content values corresponding to minimum integral entropy and minimum differential entropy are different, this particular hydration level at the minimum differential entropy cannot be considered as the maximum stability point. This is because not all available active sites have been occupied at that particular water content (Beristain *et al.*, 2002). Therefore, it is possible to say that agave fructans going to have a better stability at $a_w = 0.19$ at 25°C.

The minimum integral entropy can be considered as the moisture content of the monolayer, where exist strong bonds between adsorbent and adsorbate and therefore the minimum integral entropy is the maximum stability point of foods (Azua and Beristain, 2007; Nunes and Rotstein, 1991). It is the zone where the water molecules are best organized and less available for taking part in spoilage reactions

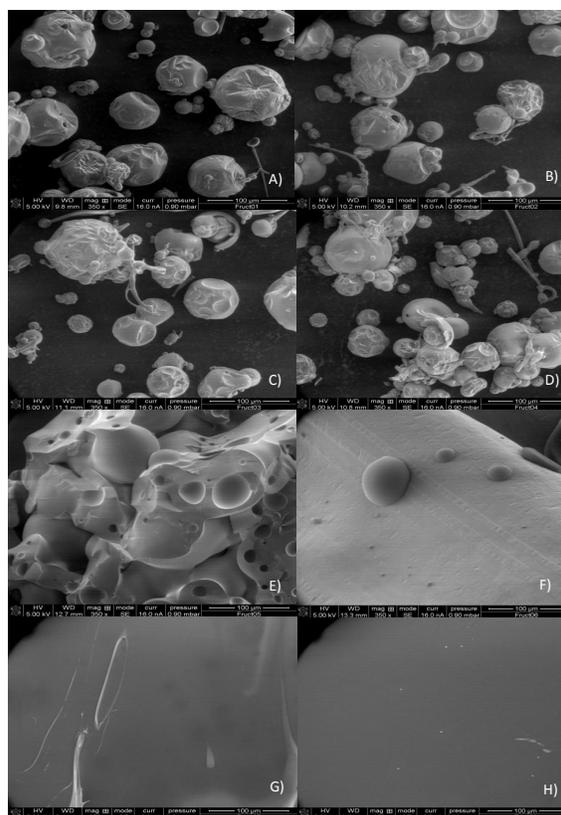


Fig. 5 ESEM Images of Agave Fructans powders, at different water activities. A) $a_w = 0.11$, B) $a_w = 0.22$, C) $a_w = 0.33$, D) $a_w = 0.44$, E) $a_w = 0.54$, F) $a_w = 0.65$, G) $a_w = 0.77$, H) $a_w = 0.85$.

(Beristain *et al.* 2002).

The point of minimum integral entropy for fructans and theoretically of maximum stability (3.46 g water/100g dry solids) was different from the GAB monolayer value (6.74 g water/100 g dry solids) (Table 1). The criteria of minimum integral entropy has been experimental confirmed in different foods such as: orange oil, macadamia nuts and ascorbic acid microcapsules (Beristain *et al.*, 2002; Domínguez *et al.*, 2007; Viveros-Contreras *et al.*, 2013).

As mentioned earlier, Figure 4 shows the variation of integral entropy with the water activity at different temperatures. The minimum integral entropy occurs in a water activity of 0.19 at 25°C, so the agave fructans should be stored in this water activity to avoid spoilage reactions, in this water activity occurs the major order of water molecules, the water molecules are more bound in the surface of foods and have less mobility and interaction between them.

3.4 Microstructure of fructans powders

Figure 5 shows the ESEM images of the particles of the fructans powders. The particles were highly heterogeneous in size and morphology. Some particles with smooth surface were found, but the most of the particles in the fructans powders exhibited indented and wrinkled surface morphology. This phenomenon can be attributed to uneven shrinkage during drying and/or cooling process. According to Ré (1998), the surface depressions are related to the collapse suffered by the droplets during the initial stages of drying.

Figure 5 shows that at low water activity values (< 0.54), agave fructans remained in a powdered amorphous state; however, at $a_w = 0.54$, there was an agglomeration of particles due to incipient dissolution of fructans and a weaker sorption on active sites; and at intermediate a_w (0.65) occurred the compaction. Guadarrama-Lezama *et al.* 2014 observed similar results by SEM when they analyzed the effect of storage temperature and water activity in carotenoid microcapsules; at high water activities (> 0.6), the microcapsules structure changed, and stickiness was observed with the formation of agglomerates due to incipient dissolution of biopolymers.

Finally, in the same figure is observed that at $a_w = 0.77$, fructans changed in a highly viscous liquid-like solution due to total solubilization of fructans, so at moisture contents above 30%, complete dissolution of biopolymers takes place.

It is possible to see the process of caking of fructans in the Figure 5. According to Porras-Saveedra *et al.* 2015, water vapor acts to increase union between individual microparticles of powders forming stable agglomeration. This process involves binding, agglomeration and compaction. These stages are identified as bridging, contact points between particles; agglomeration, an irreversible union of bridges, resulting in a particles clumps with structural integrity; and compaction, loss of system integrity and reduction of interparticle spaces and deformation. In the final stage of caking, bridges disappear as a result of liquefaction.

Conclusions

The relation between water activity and microscopy analysis improve the knowledge about structural changes developed during the storage of hygroscopic powders, identifying conditions where incipient and

total solubilization take place. This information is important when highly hygroscopic food-powders have to be used as raw material. This also allows improving the knowledge of biotechnological applications that can be given to this product, when a caked food is not desired. In this case is necessary that the agave fructans have a water activity minor than 0.54 to avoid the solubilization during its use as powder in new products processing.

The construction of adsorption isotherms is important to calculate the thermodynamics parameters to give information about physical stability of Agave fructans powder. In this context, the evidence reported by thermodynamic analysis, can be supported, applying ESEM as a useful tool to confirm any structural change associated

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Nomenclature

M	equilibrium moisture content, g water/100 g dry solids
a_w	water activity
M_0	monolayer moisture content, g water/100 g dry solids
C, K	constants related to temperature
T	absolute temperature (K)
c, k	factors of entropic accommodation
h_m	molar sorption enthalpy of the monolayer, J/mol
h_n	molar sorption enthalpy of the multilayers on top of the monolayer, J/mol
h_i	molar sorption enthalpy of bulk liquid, J/mol
Me_i	experimental moisture content, g water/100 g dried solids
Mc_i	calculated moisture content, g water/100 g dried solids
N	number of experimental data
P	relative deviation module, %
P_v	vapor pressure of water in the food, atm
P_v^0	vapor pressure of pure water, atm
$H_v(T)$	isosteric heat for water adsorption, J/mol

$H_v^0(T)$	heat of condensation of pure water, J/mol
(ΔG)	change in Gibbs free energy, J/mol
A	adsorption constant
φ	surface potential, J/m ²
$(\Delta H_{int})_T$	molar integral enthalpy, J/mol
μ_{ap}	chemical potential of the pure adsorbent, J/mol
μ_a	chemical potential of the adsorbent participating in the condensed phase, J/mol
W_{ap}	molecular weight of the adsorbent, g/mol
W_v	molecular weight of the water, g/mol
$(\Delta S_{int})_T$	change in entropy integral, J/mol-K
S_s	integral entropy of water adsorbed in the adsorbent, J/mol-K
S	total entropy of adsorbed water molecules, J/mol-K
N_1	moles of adsorbed water
S_L	molar entropy of pure liquid water in equilibrium with vapour, J/mol-K
B	constant related to the microporous structure of the adsorbent

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