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Critical water activity and critical water content of freeze-dried strawberry powder as affected by maltodextrin and arabic gum

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Nomenclature

w_e: equilibrium water content (g water/ g solids).

a_w: water activity.

w_o: GAB monolayer water content (g water/ g solids).

C: GAB constant related to monolayer sorption heat.

K: GAB constant related to multilayer sorption heat.

x_w: mass fraction of water (g water/ g product).

T_g: glass transition temperature (ºC).

$T_{g(w)}$: glass transition temperature for amorphous water: -135 ºC (Roos, 1995).

$T_{g(as)}$: glass transition temperature for anhydrous solids (ºC).

k: Gordon and Taylor constant model.

RSSg: residual square sum of the function fitted to a group of series.

RSSi : residual square sum of the function fitted to an individual series.

FDRg : freedom degrees of the residuals of the function fitted to a group of series.

FDRi : freedom degrees of the residuals of the function fitted to an individual series.

DFDR: difference between freedom degrees of the residuals of the function fitted to a group of series (FDRg) and the sum of freedom degrees of the residuals of the individual fittings of the series involved in the group (SFDRi).

Abstract

The adsorption isotherms (20ºC) and the relationship between water content and glass transition temperature were modeled in freeze-dried strawberry powder studying the effect of the addition of maltodextrin and arabic gum. Both compositional and physicochemical analyses of strawberry pulp were performed. If the midpoint of the glass transition is considered, the critical water activity that ensures the glassy state of the product during storage at 20ºC increased from 0.094 to 0.237 – 0.341 when maltodextrin and arabic gum were added, respectively. The increase in the critical water content was not so marked and it was noticeable only in arabic gum added sample (from 7.5 to 8.9 g water/100 g product), being this sample more stable.

Keywords: water sorption, glass transition, freeze-drying, fruit powders, maltodextrin, arabic gum.

Running title: Effect of solutes addition on the stability of freeze-dried strawberry powders.

1. Introduction

It is known that eating fruit is important to improve our health and well-being. The protection provided against degenerative diseases by fruits has been attributed to the fact that they provide an optimal mix of phytochemicals and other bioactive compounds such as vitamins and fiber (De Ancos, González, & Cano, 2000). Strawberries are of great interest among fruits because they have one of the highest antioxidant activities related to the high content in vitamin C and phenolic compounds, mainly anthocyanin (Odriozola-Serrano, Soliva-Fortuny, & Martín-Belloso, 2010). Various studies support the healing power of such compounds in strawberry (Pajk, Rezar, Levart, & Salobir, 2006; Tulipani et al., 2009; Mudni et al., 2009). From a therapeutical point of view, they have been related to a decreased risk of cardiovascular events, an improved endothelial function and a decreased risk of vascular thrombosis (Beattie, Crozier, & Duthie, 2005). Moreover, experiments have shown the anticancer activity of extracts of strawberries, as well as blocking the formation and spread of tumors. Roussos, Denaxa, & Damvakaris, (2009), in preliminary animal studies, also suggested that diets rich in strawberries may have the potential to provide benefits for the aging brain.

Fresh consumption of strawberry is the best way to get all its sensory, nutritional and functional properties. However, the seasonal production and/or the short shelf life, associated to its high water content, limit their availability. In this sense, strawberries can be processed by freezing and/or dehydration methods and consumed in many other forms such as juice, jam, jelly or dried fruit.

The preservation of biological products by reducing their water content can be achieved by several dehydration techniques. Among these, freeze-drying is considered as the reference process for manufacturing high-quality dehydrated products. Compared to classical dehydration techniques, the main advantages of the freeze-drying process are: (i) the preservation of most of the initial raw material properties such as shape, appearance, taste, colour, flavour, texture, biological activity, etc. and (ii) the high rehydration capacity of the freeze-dried product (Hammami & René, 1997).

The removal of water during freeze-drying often leads to the formation of an amorphous matrix, were soluble and insoluble biomaterials compatible with water appear molecularly disordered. This non-equilibrium thermodynamic state exhibits time-dependent changes approaching the equilibrium. Amorphous materials can be found in the glassy or rubbery state, being the glass transition temperature (T_g) which marks the corresponding change of state. At this transition temperature important changes in mechanical and diffusional properties occur. The main difference between the two states is related to molecular mobility, very low in the glassy state, due to the high viscosity of the matrix (about 10^{12} Pas), and higher in the rubbery. In fact, the glassy state can be considered stable in the characteristic shelf life of foods, while the changes are faster in the rubbery state whose are controlled, in many cases, by the difference between the storage temperature and the T_g of the amorphous matrix (Roos, 1995).

In powder products, the change in mechanical properties have been related to the development, in the rubbery state, of phenomena such as stickiness, caking and structural collapse, as well as solute crystallization (Telis & Martínez-Navarrete, 2009). On the other hand, the rate of diffusion controlled reactions, such as non-enzymatic or enzymatic browning, oxidation of compounds, etc., is highly increased at temperatures higher than T_g (Roos, 1995). In this sense, the T_g can be used, together with a_w, as a reference parameter to characterize properties, quality, stability and safety of food systems (Ahmed & Ramaswamy, 2006; Roos, 1995). Depending on storage temperature, the glass transition will occur at a critical value of water content (CWC) and water activity (CWA) of the sample, which can be considered determinant for the stability of the powder product.

Powdered foods rich in low molecular weight sugars, such as fruits, are very sensitive to environmental conditions having a hard handling and being susceptible to quality damages due to its characteristic higroscopicity. To prevent this undesirable situation the use of different high molecular weight substances, such as maltodextrin with different dextrose equivalent (DE), arabic gum, etc., has been described, in order to increase glass transition temperature and reduce the stickiness, producing free flowing powders with improved handling and quality properties (Barbosa-Canovas, Ortega-Rivas, Juliano, & Yan, 2005; Gabas, Telis, Sobral & Telis-Romero, 2007; Sablani, Shrestha, & Bhandari, 2008; Silva, Sobral, & Kieckbusch, 2006; Telis & Martínez-Navarrete, 2009). Nevertheless, different results are obtained depending on the fruit composition.

The aim of this work was to evaluate the effect of adding maltodextrin or arabic gum to strawberry pulp previously to the freeze-drying process. In order to contribute to optimize the technological process of powdering, guaranteeing the quality and longer shelf-life of the obtained product the influence of solutes addition on the water sorption isotherm and glass transition temperature was studied.

2. Materials and methods

2.1. Sample preparation and analyses

Strawberries (*Fragaria ananassa* var. Camarosa) were purchased in a local market in Valencia (Spain). In order to obtain the strawberry pulp, these were cut and the hull was removed. A part of the pulp was used to analyze the composition of the fruit, as described below, and the rest was freeze-dried both with and without the addition of maltodextrin DE 16.5–19.5 and arabic gum, which were obtained from Sigma–Aldrich (USA).

The two different additives were added in proportion of 1kg per kg of soluble solids of the strawberry pulp and homogenized by using a high-shear prober mixer (Ultra-Turrax T25, IKA). Both mixtures and natural pulp were immediately frozen at -40ºC in thin layers for 24 h before the freeze-drying in a Telstar Lioalfa-6 Lyophyliser at 10^{-2} Pa and -40ºC for 48 h. The freeze-dried products were ground in a crushing machine producing powders whose water content and water activity were measured (three replicates) with a Vaciotem, J.P. Selecta vacuum oven (60 ± 1ºC and pressure <100 mm Hg) and an Aqualab CX-2 Decagon Devices, respectively.

Three replicates of each of the following analyses were carried out on strawberry pulp. The water content and water activity was obtained by using the same previously described methodology. ºBrix were measured in an Atabbé Atago 3-T refractometer. Sugars (glucose, fructose and sucrose) were analyzed by high performance liquid chromatography (HPLC) using a Waters 600E system controller (Waters Corporation, Milford, MA) along with the Waters 464 Pulsed Detecting Electrochemical Detector (Waters Corporation, Milford, MA). A23–24C thermostated Hamilton RCX-10 column (Hamilton Company, Reno, NV) was used with a NaOH 0.15-M flow solution at a 1.0-ml/min flow rate. Sugar extraction was carried out in HPLC water in an ultrasounds bath (Ultrasons-H; J.P. Selecta, Barcelona, Spain) for 30 min at T < 50ºC. Lactose, used as internal standard, was added to the extract, which was clarified with the reagents

Carrez I and II. After centrifugation, the extract was purified through filtration (Millex filter 0.45 mm and cartridge Sep-Pack 0.2 mm). The *p*H was measured by a *p*H meter (Crison micro *p*H 2001, Spain) (AOAC 981.12) and titrable acidity was calculated as a percentage of citric acid by titrating 10 g of the sample with a solution of NaOH (0.1 N) until pH 8.1 (Crison micro *p*H 2001, Spain), according to AOAC 942.15. Total Dietary Fibre was analyzed as described by AOAC 985.29. The method of Yu et al. (1996) was used to determine pectin content. Total polyphenols were analyzed according to Igual, García-Martínez, Camacho, & Martínez-Navarrete, (2010).

For sorption experiments, freeze-dried powder samples obtained with and without maltodextrin and arabic gum addition (FS-M, FS-A and FS, respectively), were placed at 20 ºC in hermetic chambers containing saturated salt solutions (LiCl, CH_3COOK, $MgCl_2$, K_2CO_3, $Mg(NO_3)_2$, $NaNO_2$, and $NaCl_2$). Three replicates of about 2 g were placed in each chamber with different relative humidities (RH) ranging between 11% and 75% (Greenspan, 1977). The sample weights were controlled till a constant value ($\Delta m < \pm 0.0005$ g) was reached, where the equilibrium was assumed (Spiess & Wolf, 1983). In this moment, the a_w of each sample was assumed to be equal to the corresponding RH/100. In each equilibrated sample, the final water content was obtained from the initial water content data and the change in the registered weight till equilibrium. These values were used in order to construct the sorption isotherms.

Calorimetric analyses were carried out in each equilibrated sample in order to analyze the glass transition temperature by differential scanning calorimetry. About 10 mg of each sample were placed into DSC pans (P/N SSC000C008, Seiko Instruments) sealed and analyzed using a DSC 220CU-SSC5200 (Seiko instruments Inc.). The heating rate was 5 ºC/min and the temperature range varied between -100 and 200 ºC, depending on the sample water content and the kind of sample. The onset, midpoint and endpoint of the glass transition were obtained from each thermogram.

2.2. Fitted models and statistical comparison among the different experimental series

In order to predict the water sorption behaviour of samples, the GAB (Guggenheim, Anderson and de Boer) (Van den Berg and Bruin, 1981) model (Eq. 1) was used. A polynomic fitting of data was used to obtain the GAB parameters.

$$w_e = \frac{w_o \cdot C \cdot K \cdot a_w}{(1 - K \cdot a_w) \cdot (1 + (C - 1) \cdot K \cdot a_w)} \qquad (1)$$

Experimental T_g–water content (g water/g product) data were fitted to the Gordon & Taylor model (1952) (Eq. 2) considering the onset, midpoint and end of the transition.

$$T_g = \frac{(1 - x_w) \cdot T_{g(as)} + k \cdot x_w \cdot T_{g(w)}}{(1 - x_w) + k \cdot x_w} \qquad (2)$$

A non-linear fitting of data was carried out in both cases, applying the CHI^2 procedure using ORIGIN Pro 6.1 software.

To evaluate the differences in sample behaviour (a_w or T_g changes with x_w) as a function of solutes addition, both equations fitted to each individual series and those fitted to different groups of the series, depending on the obtained results, were statistically compared through the values of statistics E (Eq. 3) which was compared with tabulated F-Snedecor as a function of the values of DFDR and SFDRi (Eq. 3), at a 95 % significance level (Moraga, Martínez-Navarrete, & Chiralt, 2004).

$$E = \frac{(RSSg - \sum_{i=1}^{n} RSSi)/DFDR}{\sum_{i=1}^{n} RSSi / \sum_{i=1}^{n} FDRi} \qquad (3)$$

3. Results and discussion

3.1. Compositional and physicochemical analyses of the strawberry pulp

Because of components such as proteins, fat and ash are fewer representatives in the composition of this kind of fruits, for the characterization of the strawberry pulp, only essential components involved in the sorption phenomena and some bioactive compounds have been considered. Table 1 shows the results of compositional and physicochemical analyses performed in the strawberry batch used in this study. The high water activity of the fruit (0.982 ± 0.01) is related to its high water content (90.7 ± 0.1 g water / 100 g fresh product) which was similar to that found by other authors (Hammami & René, 1997; Moraga, Martínez-Navarrete, & Chiralt, 2006) and higher than that present in other berries like gooseberry (84.00 ± 0.01 g of water / 100 g fresh product) (Wang, Zhang, & Chen, 2008). The low pH found in the strawberry batch (3.71 ± 0.01) was close to that reported by other authors for the same strawberry variety and similar to the pH of typical acid fruits such as orange or grapefruit. Similar values were also obtained for the acidity of the samples, expressed as the citric acid content, the major organic acid in those fruits (Ayala-Zavala, Wang, Wang, & González-

Aguilar, 2004; Moraga et al., 2006; Moraga, Moraga & Martínez-Navarrete, 2010; Topuz, Topakci, Canakci, Akinci, & Ozdemir, 2005). The obtained soluble solids content (9.3 ± 1.0) is associated with a higher proportion of sugars (5.9 ± 0.2) (glucose, fructose and sucrose) than other carbohydrates with more complex structures. Other components whose contribute to the soluble solid content of the fruit, related to the ºBrix value, are the components present in the soluble fraction of the total dietary fibre (TDF), mostly composed by pectin (Table 1). The consumption of foods rich in fibre such as fruits may improve health, being the recommended intake 30–45 g/day (Colin-Henrion, Mehinagic, Renard, Richomme, & Jourjon, 2009).

In this study, the bioactive compounds with antioxidant activity were in the range of that reported in other studies. Klopotek, Otto, & Böhn, (2005) reported vitamin C contents between 37 and 69 mg/100 g in different strawberry juices, being the ascorbic acid concentration of the strawberry batch analyzed in this study 38.5 mg/100 g. The amount of phenolic compounds (0.215 g/100 g) was within the range of those observed in different strawberry cultivars, varying from 0.173 to 0.318 mg/100 g (Da Silva-Pinto, Lajola, & Genovese, (2008). It was higher than those found for orange (0.154 ± 0.10 g/100 g) (Gorinstein et al., 2001).

3.2. Sorption isotherms

The water sorption isotherms (20 ºC) of the strawberry powders obtained with and without the respective high molecular weight solute addition are plotted in Fig. 1. They show the g of water adsorbed by g of dry solids (w_e) as a function of water activity in each studied sample. Data showed the typical behavior of rich sugars foods: a slow increase in the equilibrium moisture content in the low a_w range, and a sharp increase at intermediate a_w values (0.600) due to the prevailing effect of solute–solvent interactions associated to sugar dissolution (Alhamdam & Hassan, 1999; Hubinger, Menegalli, Aguerre, & Suarez, 1992; Saravacos, Tsiourvas, & Tsami, 1986; Tsami, Krokida, & Drouzas, 1990). The effect of solute addition was noticeable in the whole range of evaluated relative humidities. The equilibrium water content at a given water activity of strawberry samples containing solutes were lower than those corresponding to the solute-free fruit powder, suggesting a decrease of water uptake capability of freeze-dried powdered strawberry, in agreement to the results obtained by other authors (Gabas et al., 2007; Gabas, Telis-Romero, & Menegalli, 2009; Kurozawa, Park, & Hubinger, 2009; Silva et al., 2006; Telis & Martínez-Navarrete, 2009).

To predict the sorption properties of foods, several empiric and theoretical equations have been described (Chirife & Iglesias, 1978; Van den Berg & Bruin, 1981), but the GAB model is the one most extensively used in foodstuffs, such as fruits. In our study, GAB model was well fitted to sorption data and the obtained parameters showed the solute addition effect. The value of the monolayer moisture content is of particular interest, since it indicates the amount of water that is strongly adsorbed to specific sites on the food surface and may be related to food stability. According to the fitted GAB model, the monolayer moisture content was 0.102 g water/g solids in FS, being lower in the samples with maltodextrin or arabic gum (0.075 and 0.065 g water/g solids, respectively). The K values of the samples presented small variations, all near to 1 (FS: 1.01; FS-M: 1.00 ; FS-A: 1.03), demonstrating multilayer properties which are similar to liquid water (Pérez-Alonso, Beristain, Lobato-Calleros, Rodríguez-Huezo, & Vernon-Carter, 2006). In all cases were obtained values of C parameter higher than 2, (FS: 18.81 ; FS-M: 8.48 ; FS-A: 13.94), related to the presence of an inflection point and an evolution of type II according to Brunauer´s classification. Despite the observed differences in the GAB parameters obtained, FS-M and FS-A samples showed a similar trend with respect to the adsorption when they are incorporated to the strawberry pulp. The GAB model predictions suggest that both samples with solute addition behave in a similar way in all the range of water activity.

In order to evaluate the statistical differences in the water sorption behaviour of strawberry samples with and without solutes addition, the GAB model fitted to each individual series and the model fitted to different groups of data series was compared. The value of statistic E and the tabulated F-Snedecor as a function of the values of DFDR and SFDRi (Eq. 3), at 95% significance level, was used to this end. Table 2 shows the groups of experimental series in which the statistical differences among the fitted GAB functions were analysed. As significant differences were obtained in the comparison of the three series, the value of statistic E being higher than the tabulated F Snedecor ($\alpha < 0.05$), these were compared in pairs: FS and FS-M; FS and FS-A; FS-M and FS-A. The addition of both high molecular weight solutes implied significant differences ($\alpha < 0.05$) in the water sorption behaviour of samples, confirming the less hygroscopic nature of FS-M and FS-A than the FS powder. No significant differences between the strawberry powders containing the two different added solutes were obtained from the statistical analysis. In this sense, any of them can be used to reduce the adsorption capability of freeze dried strawberry powder. This results are in agreement with that obtained for Jaya & Das, 2004. In the same way, Tonon et al., (2009) and Pérez-Alonso et al., (2006) observed a similar trend to asses sorption isotherms in powdered products from açaí with and without solutes addition and maltodextrin 10 DE and arabic gum, respectively.

3.3. Glass transition temperatures

The relationship between the T_g and x_w of the strawberry powders obtained with and without both solutes addition are plotted in Fig. 2, considering the onset, midpoint and endpoint of the transition. As the x_w of every sample increased, there was an observed decrease in their T_g value. Table 3 shows the obtained Gordon and Taylor parameters: the empirical constant k and the T_g of the anhydrous solids (T_{gs}) for each fitting. The k value was similar in all samples, but, as expected, the glass transition temperature of the anhydrous solids was higher in FS-M and FS-A samples, especially when the arabic gum was added. This is related to the increase of the average molecular weight due to the solids addition. The same trend was observed by several authors working with other freeze-dried fruits (Sablani et al., 2008; Silva et al., 2006; Telis & Martínez-Navarrete, 2009; Telis & Sobral, 2001, Takeiti, Kieckbusch, & Collares-Queiroz, 2008). The close fit of this model to experimental data series can be observed in Fig. 2.

To evaluate the different behaviour of the strawberry samples as a function of the solutes addition, the statistical significance of the differences observed, considering the midpoint of the transition, among the T_g–x_w modeled curves was analyzed, in the same way as for sorption isotherms. The results appear in Table 2, at 95% significance level. The addition of both solutes to strawberry fruit pulp implied statistical differences ($\alpha < 0.05$) between samples, increasing the T_g value throughout the whole range of x_w studied. There also were significant differences between FS-M and FS-A samples, being the last ones more stables at room temperature.

In order to obtain the critical water content and water activity values related to glass transition, the combined T_g–x_w–a_w data and the corresponding GAB and Gordon and Taylor fitted models for the samples FS, FS-M and FS-A were used (Fig. 3). This figure can be considered as a state diagram, showing the relationship between the water interaction in the product and its physical state as a function of the temperature. This tool allows us to predict the critical variables at which the glass transition occurs, at a determined storage temperature of the product (Roos, 1993). At room temperature (20 ºC), the CWA for the glass transition of the freeze-dried strawberry considering the midpoint of the glass transition, was 0.094 and, therefore, the maximum relative humidity of the atmosphere that would ensure the glassy state of the product during the whole storage period is 9.4 %. The corresponding CWC was 7.5 g water/100 g product. In the case of freeze-dried products, stickiness development could occur around and above those critical values. The low critical values obtained for strawberry justify the use of high molecular weight solutes in order to increase the T_g of the product and so the stability during handling and storage. The CWA increased till 0.237 or 0.341 when maltodextrin or arabic gum were used, respectively. The increase in CWC was not so marked and only was noticeable in FS-A samples (from 7.5 to 8.9 g water/100 g product). Thus, the arabic gum seems to be more accurate to improve the stability on the powdered strawberry.

4. Conclusions

Adding maltodextrin and arabic gum to the strawberry pulp before freeze-drying was of use in the improvement of the stability of strawberry powders, decreasing the hygroscopicity of the powder and increasing the T_g. No significant differences were observed in the sorption isotherms (20ºC) of the strawberry with solutes added, although samples with arabic gum had higher values of the glass transition temperature. The critical water activity was increased from 0.094 to 0.237 – 0.341 when maltodextrin and arabic gum were added at the concentration considered in the study, respectively. Nevertheless, the increase in the critical water content was not so marked and it was noticeable only in arabic gum added sample (from 7.5 to 8.9 g water/100 g product). Arabic gum was shown more effective than maltodextrin to improve the handling of the powder.

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FIGURE CAPTIONS

Figure 1. Water sorption isotherms (20ºC) of strawberry powders obtained without (FS:○) and with maltodextrin or arabic gum addition (FS-M:☐ and FS-A:◇). Experimental data (points) and fitted model (lines).

Figure 2. Glass transition temperature – water content relationship of strawberry powders obtained a) without (FS) and with b) maltodextrin (FS-M) or c) arabic gum addition (FS-A). Experimental data (points) and fitted model (lines), considering the onset, midpoint and endpoint of the transition.

Figure 3. Temperature - water activity (open symbols) and water activity - water content (solid symbols) relationship of strawberry powders obtained a) without (FS) and with b) maltodextrin (FS-M) or c) arabic gum addition (FS-A). Experimental points and GAB and Gordon & Taylor fitted models, considering the onset, midpoint and endpoint of the transition.

Figure 1

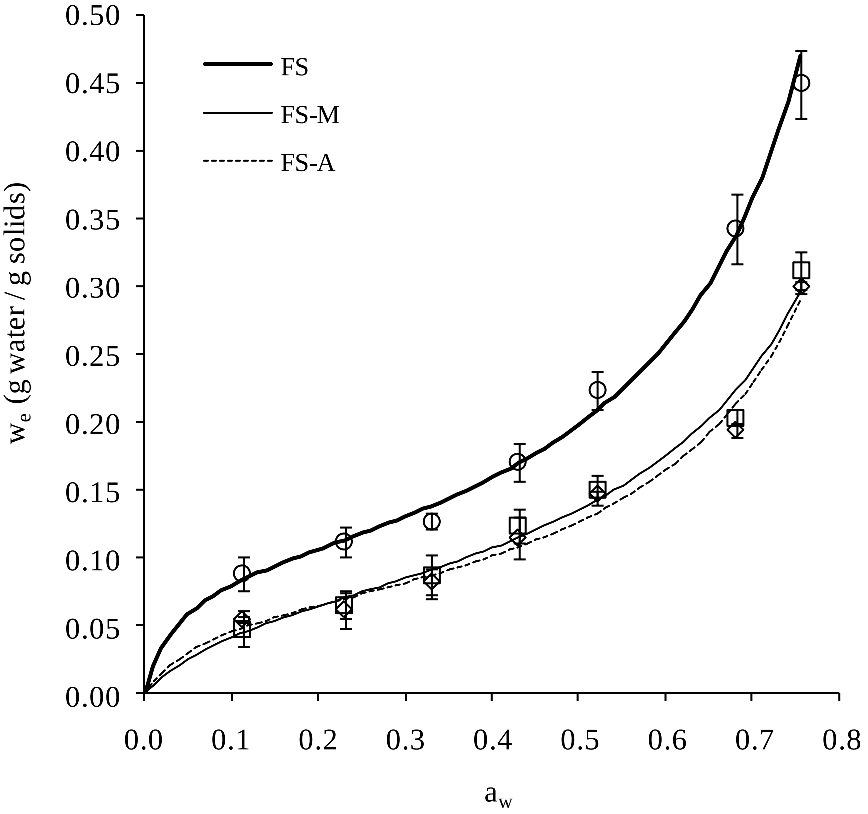
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
w_e (g water / g solids)
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
a_w
FS
FS-M
FS-A

Figure 2

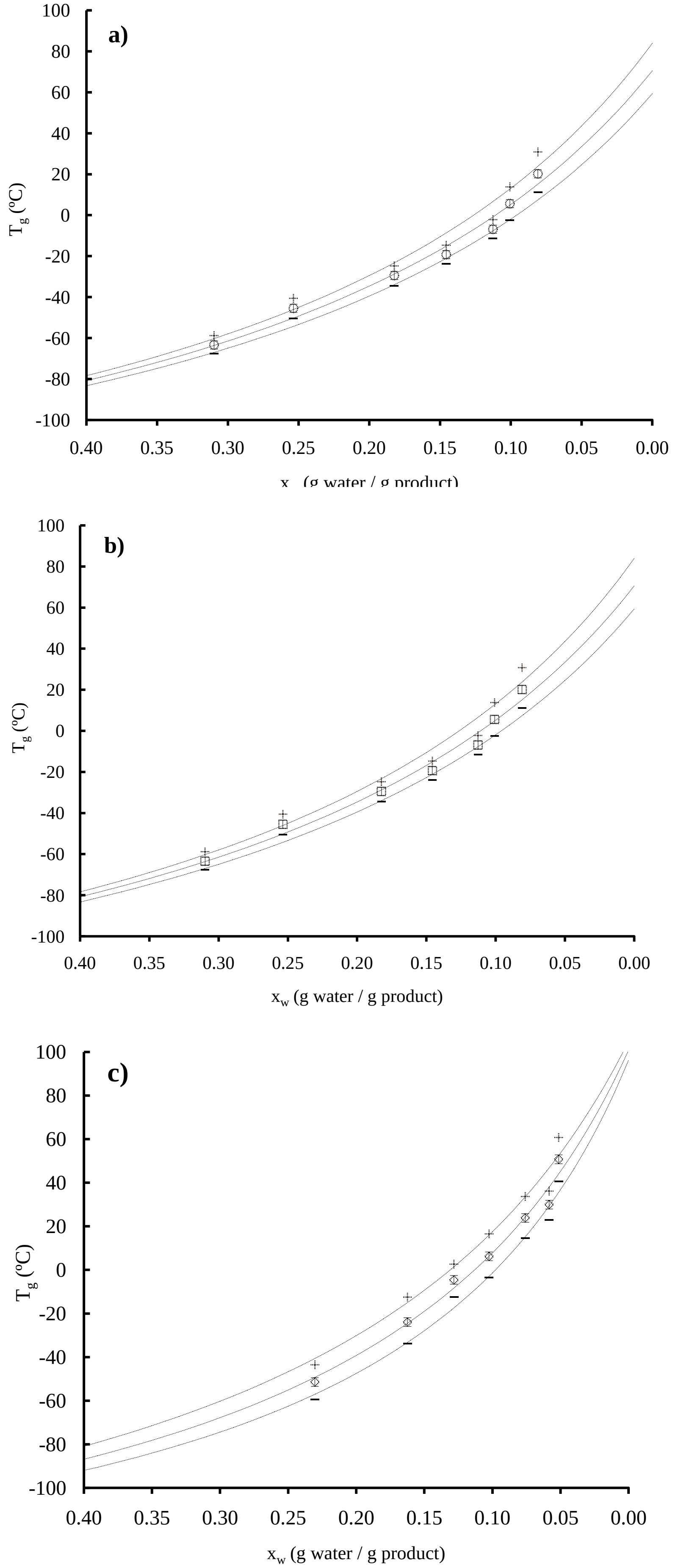
a)
b)
c)
100
80
60
40
20
0
-20
-40
-60
-80
-100
T_g (ºC)
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
x_w (g water / g product)

Figure 3

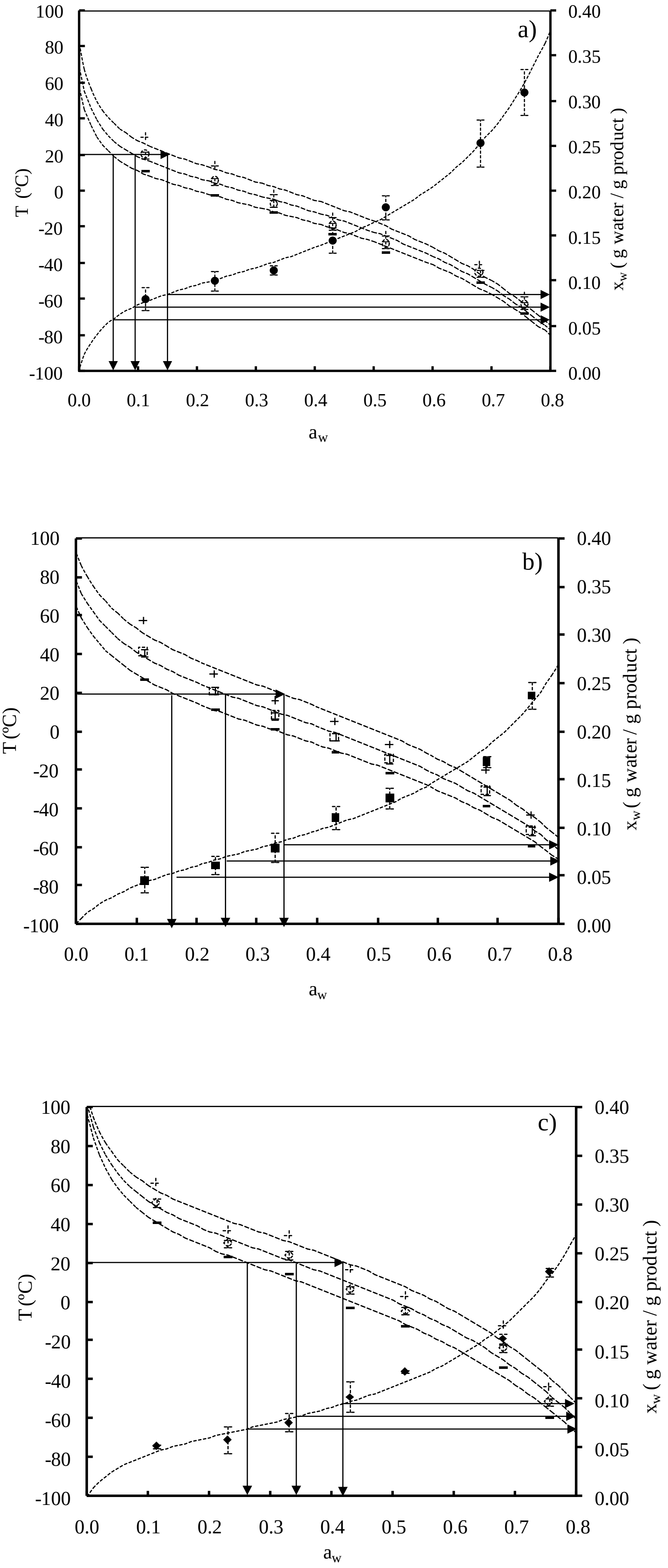
a)
b)
c)
T (ºC)
x_w (g water / g product)
a_w
100
80
60
40
20
0
-20
-40
-60
-80
-100
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8

Table 1

Table1. Compositional and physicochemical properties of strawberry pulp.

Components	
Water content (g/100g)	90.7 ± 0.1
Carbohydrates (g/100g)	
Sugars	5.9 ± 0.2
Glucose	2.6 ± 0.3
Fructose	2.8 ± 0.2
Sucrose	0.5 ± 0.1
TDF	2.2 ± 0.5
Pectin	2.5 ± 0.3
Bioactive components (mg/100g)	
Ascorbic acid	38.5 ± 0.7
Total phenols	215.4 ± 0.6
Antioxidant activity (%DPPH)	82 ± 2
Physicochemical properties	
°Brix (g/100g)[b]	9.3 ± 1.0
Water activity	0.982 ± 0.001
Tritable acidity (g/100g)	1.1 ± 0.4
*p*H	3.71 ± 0.01

[a] Total Dietary Fibre.

[b] g soluble solids/100 g fruit liquid phase.

Table 2

Table 2. Statistical comparison among samples in terms of the fitted models.

GAB Compared samples					Gordon and Taylor Compared samples			
FS	x	x	x		x	x	x	
FS-M	x	x		x	x	x		x
FS-A	x		x	x	x		x	x
SCRG	7.70	4.94	6.07	1.09	1617.93	849.36	865.68	1059.33
SCRi	1.11	0.52	0.72	0.98	927.93	660.06	660.25	535.54
GLRG	18	11	11	11	40	26	26	26
DGLR	6	3	3	3	4	2	2	2
SGLR	12	8	8	8	36	24	24	24
E	11.8	22.8	19.7	0.3	6.7	3.4	3.7	11.7
F (95%)	4.8*	7.6*	7.6*	7.6$^{(ns)}$	2.7*	3.4*	3.4*	3.4*

* Significant differences ($\alpha < 0.05$).

$^{(n.s)}$ Non-significant differences ($\alpha > 0.05$).

Table 3

Table 3. Parameters of Gordon and Taylor models fitted to experimental data considering the onset midpoint and endpoint of the glass transition (R^2: determination coefficient). Critical water content (CWC) and water activity (CWA) values related to glass transition.

Samples	T_{gs}	k	R^2	CWA	CWC
FS					
onset	59 ± 8	4.1 ± 0.4	0.988	0.062	0.061
midpoint	68 ± 11	4.2 ± 0.6	0.979	0.094	0.075
endpoint	84 ± 15	4.3 ± 0.8	0.965	0.159	0.096
FS-M					
onset	64 ± 4	5.2 ± 0.3	0.995	0.157	0.055
midpoint	76 ± 6	5.1 ± 0.4	0.989	0.237	0.075
endpoint	92 ± 11	5.0 ± 0.7	9.690	0.336	0.092
FS-A					
onset	96 ± 6	6.6 ± 0.4	0.984	0.260	0.075
midpoint	101 ± 6	5.9 ± 0.4	0.981	0.341	0.089
endpoint	105 ± 7	5.2 ± 0.4	0.972	0.421	0.106

T_{gs}: glass transition of anhydrous solids (ºC).
k: Gordon and Taylor constant model.