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Sorption characteristics of rosehip, apple and tomato pulp formulations as determined by gravimetric and hygrometric methods

Silvana M. Demarchi^a, Natalia A. Quintero Ruiz^a, Antonio De Michelis^b, Sergio A. Giner^{a,c,d,*}

- a Centro de Investigación y Desarrollo en Criotecnología de Alimentos (CIDCA), 47 y 116, 1900 La Plata, Argentina
- ^b CONICET AER INTA, El Bolsón, Río Negro, Argentina
- ^c Comisión de Investigaciones Científicas (CIC), 526 y 10, 1900 La Plata, Argentina
- ^d Facultad de Ingeniería, Universidad Nacional de La Plata, 1 y 47, 1900 La Plata, Argentina

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ABSTRACT

The sorption characteristics of three formulations based on rosehip, apple and tomato pulp, added with saccharides, and aimed at preparing fruit leathers, were studied. Desorption isotherms were determined at 20 and 40 °C, both by the static gravimetric and the hygrometric method. Experimental isotherms were all J-shaped, as expected for rich-sugar matrices. A model previously developed for apple leather isotherms, the GAB equation and the Halsey model were fitted to the gravimetric data. Hygrometric isotherms exhibited a typical behavior at high moisture contents although, at lower values, the water activity readings were consistently higher than in the gravimetric method, and were never below 0.363. Apparently, the low-moisture, high-sugar samples behave as non-hygroscopic materials in the short times allowed by the hygrometric measurement, possibly due to the presence of crystallized sugars. On these grounds, the fast hygrometric method appears to be unsuitable to measure the water activity of such samples.

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1. Introduction

The general stability, and thus shelf-life of a foodstuff, is regulated by the interaction of water with the rest of food components. For this reason, knowledge on the sorption characteristics is essential for various branches of food technology. The water sorption isotherm for a product, i.e, the graph where the moisture content is plotted as a function of water activity (a_w) at constant temperature, describes the thermodynamic state of water. Knowledge of such relationship is useful to predict microbiological, enzymatic, physical and chemical stability, all parameters required to estimate the shelf life of dried foods (Al-Muhtaseb, Mcminn, & Magee, 2002). Sorption data is also utilized in formulation of new products, selection of packaging materials and storage conditions, as well as in the design of drying processes (Arévalo-Pinedo, Dos Santos, Salles Arévalo, Zuniga, & Arévalo-Pinedo, 2006; Rahman & Labuza, 1999; Sablani, Kasapis, Rahman, Al-Jabri, & Al-Habsi, 2004). Numerous investigations on sorption isotherms can be

E-mail address: saginer@ing.unlp.edu.ar (S.A. Giner).

found in the literature, especially for fruits and vegetables, in the context of dehydration/rehydration processes. For instance, the sorption-related properties of tomato have been measured in halved, sliced and pureed fruit by the gravimetric method (Akanbi, Adeyemi, & Ojo, 2006; Goula, Karapantsios, Achilias, & Adamopoulos, 2008; Viswanathan, Jayas, & Hulasare, 2003). Giovanelli, Zanoni, Lavelli, and Nani (2002) presented adsorption isotherms for dried tomato products obtained gravimetrically but applied the hygrometric method for desorption data. Gravimetric isotherms of apple cut to pieces of various shapes have been described by Kaymak-Ertekin and Gedik (2004) and Sá, Figueiredo, and Sereno (1999). In turn, Vullioud, Márquez, and De Michelis (2006) presented gravimetric adsorption and desorption isotherms for whole rosehip fruits. In the named and other investigations, isotherms were determined by hygrometric and gravimetric methods, being the latter much more frequently utilized. However, any studies were devoted to the comparison of both methods. This comparison is relevant, especially in sugar-rich matrices, because gravimetric and hygrometric techniques differ mostly in the time scale over which they are carried out: gravimetric equilibrium demands some weeks (Chirife & Buera, 1995), while fast hygrometric measurements, only minutes (Fontana, 2007). High-sugar foods can present crystallized, amorphous or soluble sugars, in a complex system which appears to be never in

^{*} Corresponding author. Centro de Investigación y Desarrollo en Criotecnología de Alimentos (CIDCA), 47 y 116, 1900 La Plata, Argentina. Tel./fax: +54 (221) 4254853.

true thermodynamic equilibrium, so there are time-dependent changes involved (Roos, 1995), unlike in other saccharide/polysaccharide matrices as cereals and oilseeds. Besides, isotherms of these products, owing to their specific sorption properties, are hardly affected by temperature (Tsami, Marinos-Kouris, & Maroulis, 1990). The samples chosen in this work to carry out the comparison of methods are high-sugar formulations, based on sucrose-added fruit pulps, which may lead to a family of intermediate moisture foods (IMFs) as jams, fillings or leathers. The rosehip, apple and tomato pulp formulations developed here, for the present and previous work in our laboratory (Fiorentini, Leiva Díaz, & Giner, 2008; Leiva Díaz, Giannuzzi, & Giner, 2009), are intended to produce leathers. These restructured, snack products can be consumed without further preparation or rehydration and are capable of being stored for several months at room temperature (Quintero Ruiz, Demarchi, Massolo, Rodoni, & Giner, 2012). A careful design is required for such IMF products, regulating parameters as composition, pH and $a_{\rm W}$ (Taoukis & Richardson, 2007). When dried, these fruit-based mixtures exhibit specific properties due to their high solids content, achieved by the addition of sucrose and/or polydextrose. Besides, in the presence of natural (or added) high methoxyl pectins (HMP) and, at pH below 3.5, the drying process allows the formation of a gel network by the "saccharide-acid-HMP" mechanism (Rinaudo, 1996). The aims of this work were to determine experimental isotherms of rosehip, apple and tomato formulations, at 20 and 40 °C, both by the fast hygrometric and the static gravimetric methods, to compare their results in the light of the characteristics of such food matrices and to draw conclusions that may be of interest to the instrumentation, academic and food processing sectors.

2. Materials and methods

2.1. Rosehip pulp formulation

Ripe rosehip fruits (*Rosa rubiginosa* L., syn. *Rosa eglanteria* L.) harvested in El Bolsón, Province of Río Negro, Argentina, were boiled during 30 min in an industrial cooker, adding 0.2 kg of drinking water/kg of whole fruit. The softened fruits were processed with a pulping machine and passed through a 0.5 mm sieve, to obtain a homogeneous pulp which was concentrated to reach 14 °Brix. Then the pulp (average moisture content 85.4 g water/ 100 g pulp or 5.85 dec., d.b.) was cooled, frozen and stored at $-20\,^{\circ}\text{C}$ until using. For preparing 100 g of formulation, 75 g of the thawed pulp were mixed with 20 g of sucrose and 5 g of a citric acid solution (5.8 g/100 g solution, concentration equivalent to that in lemon juice). The mixture was manually stirred for 3 min until obtaining a homogeneous fluid-like formulation.

2.2. Apple pulp formulation

Apples (*Malus domestica* Borkh. cv. Granny Smith), purchased on a local market in La Plata, Argentina, were stored at 4 °C for no longer than 10 days before using. Fruits were washed, peeled manually and cut into 10 mm dices, discarding the core. Dices were placed in a steamer at 99.1 °C (Moulinex, France) fed with distilled water, and processed for 10 min to avoid enzymatic browning and to soften the tissue. The inactivation of polyphenol oxidase enzyme in the whole volume of blanched apple cubes was confirmed by the Guaiacol test (Yemenicioğlu, Özkan, & Cemeroğlu, 1998). Then, apple dices were processed using an electric blender (Braun Multiquick Advantage, MR4050, 400 W, Spain) for two minutes until obtaining a homogeneous puree. As in Leiva Díaz et al. (2009), 79 g of apple puree were mixed with 18 g of sucrose and 3 g of the citric acid solution described in Section 2.1 to obtain 100 g of formulation.

2.3. Tomato pulp formulation

Italian type, pear-shaped tomatoes (*Solanum lycopersicum* cv. Zorzal) selected for their high dry matter content, were washed, rinsed and cut along the longest axis to remove the seeds. Then, they were cut in small pieces and processed to puree consistency in the electric blender mentioned earlier. To obtain 100 g of formulation, 84.3 g of tomato puree, 12.95 g of an aqueous solution of polydextrose (44.4 g/100 g solution), 1.5 g of sucrose, 1 g of powder high methoxyl pectin (HMP) and 0.25 g of citric acid were mixed. A fluid homogeneous formulation was obtained, in which polydextrose contributes with soluble solids without sweetening the product (Fiorentini et al., 2008).

2.4. Characterization of products

The rosehip, apple and tomato formulations were stored in sealed jars at 4 °C for 24 h, allowing for total dissolution of added saccharides and uniform moisture distribution. Then, aliquots of the formulations were taken and allowed to equilibrate with room temperature, for determination of moisture content, water activity and pH. Moisture content was measured in 5 g samples introduced in a Mettler LP16 Moisture Analyser set at 105 °C, until reaching constant weight, according to the AOAC method 984.25 (AOAC, 1998). In turn, water activity ($a_{\rm w}$) was determined at 25 °C by the AOAC hygrometric method 978.18 (AOAC, 1998), using a temperature-controlled AquaLab 3TE water activity meter (Decagon Devices, Inc., USA). The pH was measured with an Alpha PW-40 electrode connected to an Altronix TPA-V pH-meter with digital display. All determinations were conducted in triplicate.

2.5. Determination of desorption isotherms by the static gravimetric method

The gravimetric method does not measure the water activity of the samples, but forces it to vary, in order to reach equilibrium with a constant relative humidity environment. Then, the sample moisture content at equilibration is determined. The methodology described by Rahman and Sablani (2008) was followed to determine desorption isotherms at 20 and 40 °C for the three formulations. Ten saturated salt solutions (slurries), generating constant $a_{\rm W}$ atmospheres, were prepared for sample equilibration following the AOAC method 978.18 (AOAC, 1998). Their water activities at the experimental temperatures are shown in Table 1. The solutions were placed in sealed flasks, with the precaution of covering the bottom with salt crystals, and a plastic structure was then put inside each flask to support the sample. Then, 2 g of formulation were placed in glass dishes, onto the plastic supports, and the flasks were sealed. For environments with $a_{\rm W} > 0.75$, a small container with

Table 1 Saturated salt solutions used for the static gravimetric method and their water activities at 20 and 40 $^{\circ}$ C (Adapted from TAPPI (2002)).

Saturated salt solutions utilized	Water activity		
	20 °C	40 °C	
LiCl	0.113	0.112	
KCH₃COO	0.234	0.208	
$MgCl_2$	0.330	0.320	
K ₂ CO ₃	0.432	0.433	
$Mg(NO_3)_2$	0.544	0.484	
NaNO ₂	0.654	0.614	
NaCl	0.754	0.747	
KCl	0.867	0.843	
BaCl ₂	0.904	0.884	
K ₂ SO ₄	0.976	0.964	

toluene was included in the flask to prevent microbial spoilage of the sample (Rahman & Sablani, 2008). Each point of the isotherms was determined in triplicate, i.e. three flasks were prepared for each salt solution. To maintain the set of thirty flasks at constant temperature, a thermostatic room was used at 20 °C, whereas an automatically-controlled convection culture oven with digital display was employed at 40 °C. The flasks were periodically open to weigh the samples. Equilibrium was assumed after the variation in the moisture content of samples (calculated by weight variations considering constant dry matter), was less than 0.003 kg water/kg dry matter, which is the ordinary error accepted for oven-moisture determinations (Lomauro, Bakshi, & Labuza, 1985).

2.6. Determination of desorption isotherms by a fast hygrometric method

In contrast with the gravimetric method described above, the construction of a sorption isotherm by the fast hygrometric method requires the measurement of the water activity of samples in a wide range of moisture contents. This method allows a rapid determination of isotherms at various temperatures from a common set of samples (Fontana, 2007) and is acceptable as long as the $a_{\rm w}$ measuring device is properly calibrated in the entire $a_{\rm w}$ range (Bell & Labuza, 2000). In this work, before and after carrying out all hygrometric determinations, aw readings of saturated salt solutions of low and high water activity were taken, ensuring almost exact coincidence ($a_{\rm W}$ ± 0.003) with the values of Table 1. To construct desorption isotherms for the rosehip, apple and tomato pulp formulations, samples of various moisture contents were produced in triplicate by partial hot-air drying at 60 °C of each formulation in an automatic-controlled mechanical convection oven with digital display of measured and target temperatures. The corresponding moisture contents were calculated with a mass balance, considering constant dry matter, knowing the samples initial moisture contents by the AOAC method 984.25 (AOAC, 1998) described in Section 2.4. The samples so prepared were kept in individual sealed containers for 48 h to allow moisture distribution. Finally, $a_{\rm w}$ of all samples were measured, firstly at 20 °C and then at 40 °C, with an Aqualab 3TE water activity meter (Decagon Devices, Inc., USA). Data of room temperature and ambient relative humidity was recorded during $a_{\rm w}$ measurement, using a Testo 608-H2 thermohygrometer.

2.7. Mathematical description of desorption isotherms

The Halsey model (Eq. (1)), which is usually adequate for fruit and oilseed-based products (Giner & Gely, 2005) was proposed to represent the sorptional behavior of rosehip, apple and tomato pulp formulations. For isothermal conditions, the expression can be written as:

$$W = \left(\frac{-A}{\ln(a_{\rm w})}\right)^{\frac{1}{r}} \tag{1}$$

where W, the moisture content in kg water/kg dry matter, is related to $a_{\rm w}$, the water activity, being A and r the fitting parameters of the model. The Guggenheim, Anderson, De Boer (GAB) model (Eq. (2)), based on the theory of multi-layer adsorption, was also fitted to the experimental data:

$$W = \frac{W_{\rm m}Cka_{\rm w}}{(1 - ka_{\rm w})(1 - ka_{\rm w} + Cka_{\rm w})} \tag{2}$$

being $W_{\rm m}$ the monolayer moisture content in dec., d.b., and C and k parameters of sorption dynamic equilibrium. Another equation (Eq.

(3)), purpose-developed for apple leathers by Leiva Díaz et al. (2009), was also tested:

$$W = C_1 \exp\left(C_2 a_{\rm w}^{C_3}\right) \tag{3}$$

This phenomenological model presents three fitting parameters C_1 , C_2 and C_3 .

3. Results and discussion

3.1. Physicochemical parameters of the formulations

The rosehip, apple and tomato pulp formulations are described in Table 2, which includes three parameters that are critical in the design of intermediate moisture foods (IMFs), as moisture content, water activity and pH. The composition of the formulations was not determined experimentally, but calculated by mass balances based on the composition of ingredients, taken from USDA (2010). As the tomato formulation is not intended to be sweet, it contains more fiber (provided by polydextrose) and less sugar than the apple and rosehip formulations. It also presents higher water activity and moisture content, because the water content of fresh tomato is higher than that of the other fruits.

3.2. Isotherms determined by the static gravimetric method

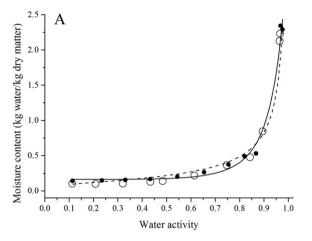
The time elapsed until reaching apparent equilibrium varied between 21 days for isotherms at 20 °C, and 18 days at 40 °C. Experimental data for rosehip, apple and tomato pulp formulations at both temperatures is shown in Fig. 1A-C, respectively, which also illustrates predictions by Eqs. (1) and (3), which provided more accurate fitting. As expected for high-soluble solids matrices, curves are "J" shaped, corresponding to type III in the BET classification (Brunauer, Deming, Deming, & Troller, 1940; Rao & Rizvi, 1995). Several researchers have found similar sorption curves for fruits and high-sugar matrices (Arévalo-Pinedo et al., 2006; Chinachoti & Steinberg, 1984; Djendoubi Mrad, Bonazzi, Boudhrioua, Kechaou, & Courtois, 2012; Sablani et al., 2004; Tsami et al., 1990). If experimental data is carefully observed, a weak but conventional effect of temperature (lower moisture contents at the same water activity for higher temperature) is detected at low to moderate water activities. However, this effect disappears or even reverses at higher water activities, due to the increased solubility of sugar at higher temperature, which results in a reduction of the mobility of water. This behavior was described by Bhandari and Adhikari (2008) and is in agreement with the isotherms showed by Tsami et al. (1990) for various fruits. Model-

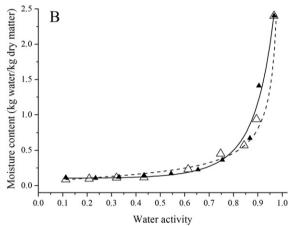
Table 2 Characterization of the fruit formulations prepared.

	•	*	
Formulation	Rosehip	Apple	Tomato
Moisture content (kg water/kg dry matter) ^a	$2,\!205 \pm 0.155$	2.337 ± 0.094	7.697 ± 0.180
Water activity ^a	0.967 ± 0.001	0.965 ± 0.001	0.989 ± 0.002
pH ^a	3.42 ± 0.03	3.34 ± 0.04	3.61 ± 0.04
Sugars (g/100 g) ^b	21.44	24.39	3.69
Dietary fiber (g/100 g) ^b	1.01	0.82	7.51
Total carbohydrates (g/100 g) ^b	29.12	26.27	12.46
Protein (g/100 g) ^b	0.88	0.19	0.93
Fat (g/100 g) ^b	0.11	0.06	0.17
Ashes (g/100 g) ^b	0.67	0.06	0.17

 $^{^{\}rm a}\,$ Expressed as average \pm standard deviation of three determinations.

^b Calculated by mass balance.





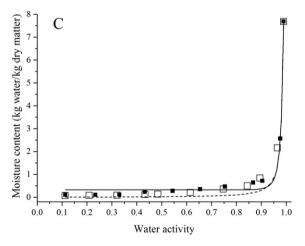


Fig. 1. Gravimetric isotherms for rosehip (A), apple (B) and tomato (C) formulations. Experimental data are represented in solid symbols for 20 °C and in empty symbols for 40 °C. The respective model predictions are presented in lines: Leiva Díaz model at 20 °C (——) and Halsey model at 40 °C (——).

predicted values also suggest the crossover of isotherms at high water activities in the three formulations, despite the weak effect of temperature. A one-way analysis of variance (ANOVA) with randomized block design was carried out to analyze the effect of temperature on the curves, taking the water activity as the block condition. The effect of temperature was not significant for the rosehip formulation at 95% confidence level, whereas isotherms for apple and tomato products were significantly affected by temperature, though not to a large extent (p=0.049). Other authors have described fruit isotherms not

depending on temperature (Ratti, Crapiste, & Rotstein, 1989). Table 3 lists the fitting parameters of the GAB, Halsey and Leiva Díaz et al. (2009) models and their corresponding coefficients of determination (R^2), calculated by nonlinear least squares, using a statistical software (Origin, 2009). For the three formulations studied, the Halsey model gave the best representation of experimental data at 40 °C, whereas the Leiva Díaz model resulted more accurate at 20 °C. For a specific study of isotherm modeling, a two-zone fitting may be more exact e.g. for water activities varying from 0.1 to 0.85 and from 0.85 to almost unity, as shown by Giner, Torrez Irigoyen, Cicuttín, and Fiorentini (2010).

3.3. Isotherms measured by the fast hygrometric method

Once the set of partially dried samples was prepared according to the method described in Section 2.6, and their moisture content was known, the measurement of $a_{\rm w}$ by the fast hygrometric technique took no longer than 5 min per sample. Experimental isotherms obtained for rosehip, apple and tomato pulp formulations at 20 and 40 °C are shown in Fig. 2A–C. Although the "J" shape is also evident in hygrometric isotherms, the GAB, Halsey and Leiva Díaz et al. (2009) models were not fitted to these data because unexpected results were obtained for the three formulations, both at 20 and at 40 °C: surprisingly, measured values of $a_{\rm w}$ were never below 0.363 in any fruit formulation, in spite of the fact that the driest samples were dehydrated at 60 °C for 53 h, at an average relative

Table 3Fitting parameters of the models which describe the desorption isotherms for the three formulations at 20 and 40 °C.

Formulation	Model	Parameters	Values at 20 °Ca	Values at 40 °Cª
Rosehip	Leiva Díaz	C ₁	0.164 ± 0.044	0.130 ± 0.019
		C_2	3.082 ± 0.240	3.400 ± 0.133
		C_3	5.487 ± 1.137	5.262 ± 0.491
		R^2	0.986	0.997
	Halsey	Α	0.082 ± 0.008	0.087 ± 0.002
		r	1.327 ± 0.112	1.140 ± 0.027
		R^2	0.972	0.998
	GAB	$W_{\rm m}$	0.147 ± 0.027	0.125 ± 0.011
		С	0.707 ± 0.000	0.700 ± 0.000
		k	0.968 ± 0.010	0.981 ± 0.004
		R^2	0.974	0.994
Apple	Leiva Díaz	C_1	0.106 ± 0.045	0.130 ± 0.032
		C_2	3.560 ± 0.401	3.425 ± 0.230
		$\frac{C_3}{R^2}$	3.712 ± 0.780	4.713 ± 0.676
		R^2	0.982	0.991
	Halsey	Α	0.107 ± 0.010	0.098 ± 0.002
		r	1.243 ± 0.113	1.182 ± 0.031
		R^2	0.960	0.997
	GAB	$W_{\rm m}$	0.184 ± 0.026	0.147 ± 0.013
		С	0.700 ± 0.000	0.700 ± 0.000
		k	0.964 ± 0.010	0.977 ± 0.005
		R^2	0.974	0.992
Tomato	Leiva Díaz	C_1	0.319 ± 0.073	0.224 ^b
		C_2	4.503 ± 0.240	4.242 ^b
		$\frac{C_3}{R^2}$	31.380 ± 4.233	16.472 ^b
		R^2	0.991	0.994
	Halsey	Α	0.059 ± 0.007	0.083 ± 0.003
		r	0.827 ± 0.064	0.990 ± 0.018
		R^2	0.988	0.999
	GAB	$W_{\rm m}$	0.056 ± 0.005	0.086 ± 0.004
		C	0.700 ± 0.000	0.700 ± 0.000
		k_	1.004 ± 0.000	1.000 ± 0.000
		R^2	0.991	0.998

^a Expressed as the confidence interval for the fitting parameter (P < 0.05).

b Due to lack of convergence of the Leiva Díaz et al. (2009) model implemented in Origin software when fitting to the isotherm of tomato pulp formulation measured at 40 °C, these parameters were optimized with the nlinfit function in the Matlab programming environment, which does not report the confidence interval for the parameters.

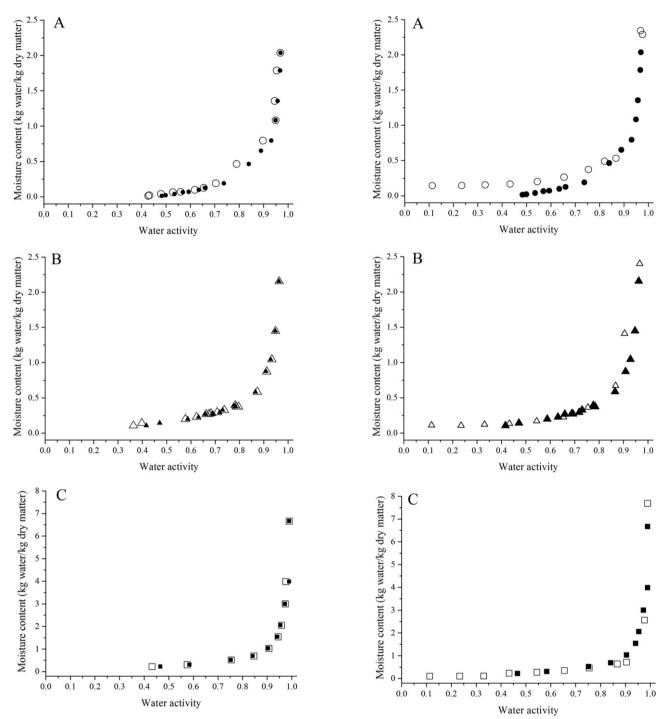


Fig. 2. Hygrometric isotherms for rosehip (A), apple (B) and tomato (C) formulations at 20 $^{\circ}C$ (solid symbols) and 40 $^{\circ}C$ (empty symbols).

humidity of 7.5% and, therefore, the water activity of such samples should have reached an equilibrium value close to 0.075. Besides, in the low-moisture range, $a_{\rm w}$ values were lower at 40 °C than at 20 °C, in contrast with the expected behavior.

3.4. Comparison of methods and discussion

When comparing isotherms obtained at 20 °C by hygrometric and gravimetric methods in Fig. 3A–C, a consistent behavior was observed for all the formulations, and the same occurred when

Fig. 3. Comparison of isotherms obtained by the gravimetric (empty symbols) and the hygrometric (solid symbols) methods for rosehip (A), apple (B) and tomato (C) formulations at $20~^{\circ}$ C.

comparing methods at 40 °C: for $a_{\rm w}$ above 0.7–0.85, depending on the formulation, the two methods led to similar results. However, in the low-moisture range, isotherms differed substantially because the hygrometric curves did never reach $a_{\rm w}$ values lower than 0.36. This phenomenon is attributed here to the non-hygroscopic behavior of the low-moisture, sugar-rich samples during the short times involved in hygrometric determinations. Therefore, the relative humidity sensing system in the headspace of the measuring chamber would be reading a value of $a_{\rm w}$ not affected by the sample, but by the relative humidity and temperature of the room

where the instrument is placed. This would also explain the inverse effect of temperature observed in Fig. 2: as the temperature of the measuring chamber rises from 20 to 40 °C, the instrument reading tends to decrease, because the relative humidity of the air contained in the measuring chamber is lower for higher temperature.

Apparently, the non-hygroscopic character of the low-moisture, high-sugar samples must obey to the presence of a sugar crust. through which the water diffusion coefficient is extremely low and avoids water to be absorbed or desorbed in the short time period allowed by the hygrometric method. In fact, Fontana and Campbell (2004) said that glassy food matrices, such as hard candy, can absorb only small amounts of water from the air due to their physical state, leading sometimes to mistaken $a_{\rm w}$ readings by hygrometric methods. Our hypothesis was also supported by experimental data: a_w values found for the driest samples by the fast hygrometric method were very close to that measured, under the same room conditions, for the empty sample-holder (0.404 for the headspace air of the chamber, when the room conditions were 20 °C and 42% relative humidity, as determined with a Testo 608-H2 thermohygrometer). For a further confirmation of this behavior, pure crystals of sodium chloride and sucrose (both anhydrous, nonhygroscopic materials), were placed in the measuring chamber to find water activity readings of 0.399 and 0.395, respectively, which are considerably more affected by the relative humidity of the room than by the characteristics of the sample.

4. Conclusions

The desorption isotherms of three formulations of rosehip, apple and tomato pulp added with saccharides, measured by the static gravimetric and the fast hygrometric methods, were comparable at high water activities, both at 20 and 40 $^{\circ}$ C. However, at low moistures, hygrometric data was unexpected and very different from gravimetric curves. The low-moisture, sugar-rich samples behave as a non-hygroscopic material during the short times allowed in the hygrometric determination, being its readings higher than those of the gravimetric method, and more related to the relative humidity of the room than to the characteristics of the samples. This behavior was not observed during the gravimetric determinations, in which the samples are allowed to equilibrate its water activity with the environment during 18–21 days.

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