

Water Desorption Isotherms of Raw and Osmotically Dehydrated Garlic

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ABSTRACT

Water desorption isotherms of raw and osmotically dehydrated (at 25°C, for 2 hours, with 10 and 26% NaCl solutions) garlic slices at different temperatures (25, 40, 55, and 70°C) were experimentally determined over a range of water activity of 0.05–0.90 using a static gravimetric method. Desorption isotherms can be classified as Type III. The water desorption isotherms of raw garlic clearly showed the influence of temperature i.e. increasing water activity at a fixed moisture content with increasing temperature; whereas for the osmotically dehydrated garlic the effect of the temperature was less important. The increase of NaCl content in osmotically dehydrated garlic led to a decrease in water activity at the same moisture content, and this effect was more accentuated at high temperatures. Equilibrium moisture content data at each temperature were satisfactorily fitted with Henderson model and a six-parameters equation based on Henderson model was obtained in order to correlate simultaneously moisture content with water activity, temperature, and osmotic solution concentration. A prediction model based on concentration and physical state of garlic compounds was successfully used to reproduce the experimental data.

Keywords: Drying, Equilibrium moisture content, Modelling, Water activity.

INTRODUCTION

Garlic (*Allium Sativum* L.) is an herbaceous perennial plant cultivated in mild and dry regions. It has a bulbous root made up of several fleshy sections called cloves. These cloves are a traditional dietary ingredient of several cultures from Asia, Africa, and Europe and are currently around the world. The main world producer in 2009 was China (18.0×10^6 t), followed by India (1.1×10^6 t) and Republic of Korea (0.4×10^6 t) [8].

Garlic is used as a food ingredient because of its very characteristic flavor. Furthermore, in recent years, many research works have associated garlic intake to significant health benefits. These benefits are mainly due to

some sulfur compounds, which also contribute to the smell and taste of the bulb cloves. In this sense, it has been found that garlic has antiseptic, antifungal, antibacterial and depurative properties and its regular intake may prevent cardiovascular diseases and development of tumors [6].

Garlic is available in different forms, including fresh, frozen, fermented, canned, and dehydrated products, among others [18]. Dried or semidried garlic is one of the most common ways of presentation. The removal of water from the food material leads to a decrease of its water activity and the shelf life of the product is prolonged. The transport properties and quality changes during dehydration of garlic by different methods, such as convective drying [30],

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[21] freeze drying [23], microwave-convective drying [27] and vacuum-microwave drying [7] have been studied.

Osmotic dehydration consists of the immersion of a material into a hypertonic solution; the difference of chemical potential between the components in the solution and the material leads mainly to a water transfer from the material to the solution and solutes uptake into the material from the solution [26]. Sugars and salts are often used as solutes in the soaking solution [25]. The low water activity of salt solutions leads to a strong driving force for the process. Osmotic dehydration can be used as a pretreatment of other drying method, reducing process time and energy consumption; it also may preserve or even improve some sensory characteristics of the raw material [3].

Water sorption equilibrium data are important for the prediction of microbiological, enzymatic, and chemical activity, selection of packaging materials, design of drying and concentration processes, as well as selection of adequate storage conditions [12]. In all of these cases, the water sorption isotherms are required for design purposes. A large number of publications have reported water sorption (adsorption and desorption) isotherm data for different products, with some important compilations [11]. Although the literature of water desorption equilibrium data for raw garlic is available [2],[14]and[31], no data are found for osmotically dehydrated garlic.

When osmotic dehydration is employed, while water removal takes place, the acquisition of osmotic solute occurs in such a way that the composition of the final product is changed and the hygroscopic characteristics may also be modified. One of the aims of this work is to determine the effect of NaCl impregnation of garlic on its water desorption isotherm. Another objective is to establish the effect of temperature on water desorption isotherms of raw and osmosed garlic by considering the conditions used during storage and processing. Experimental data are modeled by using selected correlations from the

literature as well as a prediction model based on the composition and physical state of garlic components.

MATERIALS AND METHODS

Raw Material and Pretreatments

Garlic (*Allium Sativum L.*) bulbs were bought in a local supermarket from the same batch and selected with homogenous size, moisture content, color, and ripeness. Cloves were extracted from the bulbs by hand and their skin was removed carefully with a knife. Then, slabs (thickness 2 mm) were cut and directly used in the osmotic dehydration experiments.

Osmotic Dehydration

Osmotic solutions, 10 and 26% (saturated solution) (w/w), were prepared with commercial NaCl (99% purity) and distilled water. Nine to twelve garlic slabs were introduced in a plastic basket, which was fully immersed in a glass vessel containing the osmotic solution. Samples were weighed (Denver Instrument, SI234, USA, $\pm 10^{-4}$ g) before dehydration experiments. The ratio samples:osmotic solution (w/w) was 1:15 (150 g of osmotic solution), in order to keep constant the solution concentration along the dehydration experiment. The vessel was immersed in a thermostatic bath to carry out the dehydration treatment at 25°C. After 2 hours of treatment, samples were removed from the salt solution and blotted with paper tissue to remove adhered osmotic agent. This time was selected because previous experimental osmotic dehydration kinetics reported that water and salt transfers practically occur in the first hour of the process. As a consequence, it can be considered that the samples and solution achieved a pseudo-equilibrium state in 2 hours. Two slabs were used to determine water loss (WL), solids gain (SG), weight reduction (WR), normalized moisture

content (NMC) and normalized solids content (NSC), whereas the other samples were carefully cut in small pieces and put in vials (ca. 2 g vial⁻¹). These vials were used in the experimental determination of the water desorption isotherms. Samples used to determine the dehydration parameters were weighed and then fully dehydrated in a vacuum oven (Heraeus, Vacutherm VT 6025, Germany) at 70°C and less than 13 kPa till constant weight [1]. The aforementioned parameters were evaluated using the following equations:

$$WR = \frac{m_0 - m}{m_0} \quad (1)$$

$$SG = \frac{s - s_0}{m_0} \quad (2)$$

$$WL = SG + WR \quad (3)$$

$$NMC = \frac{X}{X_0} \quad (4)$$

$$NSC = \frac{s}{s_0} \quad (5)$$

Where, m and m_0 , s and s_0 and X and X_0 are sample mass, solids weight and moisture content before and after the osmotic treatment, respectively.

Experimental Determination of Sorption Isotherms

Equilibrium moisture content of raw and osmotically dehydrated garlic corresponding to water desorption process was determined by the static equilibrium method [32]. All measurements were done in triplicate using ca. 2 g of sample in each measurement. Different water activities were selected (from 0.05 to 0.90), using several saturated salt solutions at several temperatures (25, 40, 55 and 70°C) [4]. The selected salts were KOH, LiCl, MgCl₂, K₂CO₃, Mg(NO₃)₂, NH₄NO₃, NaCl, KCl and BaCl₂. Each saturated salt solution was introduced in a hermetically closed vessel, along with vials containing sample. Equilibrium between sample and environment given by each salt

solution was attained when sample weight variations were less than 0.1%. Finally, moisture content of equilibrated samples was determined as explained before.

Sorption Isotherms Modeling

Two approaches were used to modeling water desorption isotherms of garlic. In the first one, some mathematical models available from literature were fitted to experimental sorption data by using the nonlinear least square regression software package Tablecurve 2D (AISN Software). The goodness of non-linear fit was evaluated through the coefficient of determination (r^2), percent average relative deviation (P) and the mean squared error (MSE) given by:

$$P(\%) = \frac{100}{n} \sum_{i=1}^n \frac{|X_{i,cal} - X_{i,exp}|}{X_{i,exp}} \quad (6)$$

$$MSE = \frac{1}{(n-p)} \sum_{i=1}^n (X_{i,cal} - X_{i,exp})^2 \quad (7)$$

Where, $X_{i,cal}$ and $X_{i,exp}$ are the calculated values and experimental equilibrium moisture content data, respectively, n is the number of experimental data points, and p is the number of parameters of the model.

In the second approach, a simplified algorithm to predict water desorption isotherms from the chemical composition and physical state of the garlic compounds was used. This algorithm is explained in depth in a previous work and is briefly summarized here [15]. Since food materials are multicomponent and multiphasic systems, in order to simplify the calculations, only the main components (glucose, fructose, sucrose, starch, proteins, fiber and salt) were considered. In this model, food water activity is evaluated by means of the product of the water activity of each component assuming that each component has independent behavior (but considering the respective soluble and insoluble content). Particularly, water activity of soluble compounds was evaluated in the case of non-electrolytic compounds



(glucose, fructose, and sucrose) by the Norrish equation [16] and electrolytic compounds (sodium chloride) by the Pitzer equation [19]. Water activity of non-soluble compounds (fibre, proteins, starch) and non-solubilized fraction of soluble compounds (sugars and salt) was estimated from water desorption isotherms reported in literature. For non-soluble compounds, a modified Henderson equation was employed with the corresponding fitting parameters previously determined [24]. For the non-solubilized fraction of soluble compounds, the sorption isotherms were obtained in the literature at 25°C [13]. In order to simplify the prediction model, it was assumed that in the range of studied temperatures the sugars sorption isotherms were constant. Solubility of soluble compounds with temperature was estimated using the reported specific equations [22].

All equations, parameters, mathematical expressions and procedure are shown in detail in a previous paper [15]. The procedure of this prediction model consists on an iterative process in which a water activity value is fixed and, considering simultaneously the solubilized fraction of soluble compounds and water activity of non-soluble and soluble compounds, the corresponding equilibrium moisture content

are calculated at each step according to the following criteria of convergence: (a) a_w of soluble compounds $\geq a_w$ fixed for non-soluble compounds, and (b) if a_w of soluble compounds $< a_w$ fixed, the water amount must be increased. Finally, the outputs of the prediction model are the pairs of data constituted by water activity and corresponding equilibrium moisture content. The algorithm must be repeated as many times as pairs of data are considered necessary to obtain the sorption isotherm.

The algorithm was implemented in a worksheet (MO Excel 2007) to make systematic calculations. Goodness of fit was also evaluated through the parameters cited before.

RESULTS AND DISCUSSION

Osmotic Dehydration

Figure 1 shows the dehydration parameters of garlic samples after 2 hours of osmotic process. WR is practically the same for samples dehydrated with 10 and 26% NaCl solutions; after both treatments samples reduced their weight up to 55% of the initial value. WL is slightly higher (65% in comparison to 63%) in samples dehydrated

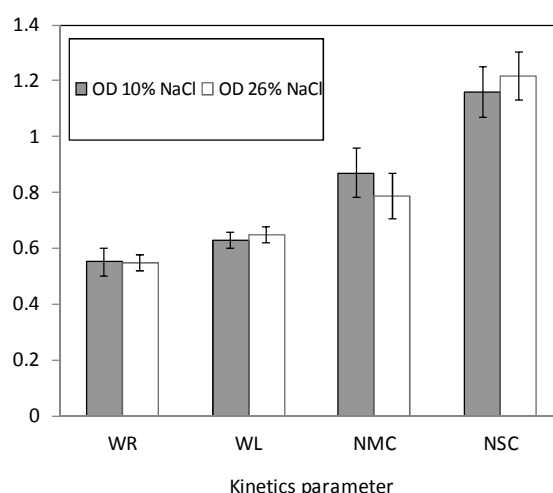


Figure 1. Osmotic dehydration kinetics parameters for garlic slices after two hours of dehydration with 10 and 26% NaCl solutions at 25°C. WR: Weight reduction; WL: Water loss; NMC: Normalized moisture content, NSC: Normalized solids content. Values are means and error bars are standard deviations (n= 3).

with 26% NaCl solutions, due to the higher osmotic pressure of this solution, resulting in a higher driving force for the process. The flux of NaCl from the solution to the garlic tissue is also higher in osmosed samples with 26% NaCl solutions. As a consequence of both water and salt fluxes, NMC is lower (0.79) and NSC is higher (1.22) when 26% NaCl solutions are used compared to osmotic dehydration with 10% NaCl solutions (0.87 and 1.16, respectively). The different composition of raw and osmosed samples leads to different water sorption properties in garlic slices, as observed and discussed in the next section.

Water Desorption Isotherms

Figure 2 shows the water desorption isotherms of raw and osmotically dehydrated garlic slices, that is, the plot of equilibrium moisture content versus water activity for all the tested conditions at the temperature range of 25-70°C. Equilibrium data for raw

garlic was in the same range as the reported data by other authors at different temperature intervals from 25 to 65°C [18] and from 50 to 70°C [14]. In all cases, moisture content increased with increasing water activity, leading to concave curves that can be classified as type III sorption isotherms [5].

The highest increase in moisture content was given at water activity values higher than 0.6, whereas at lower values the moisture content increased very slightly, being practically constant at the lowest water activity values. The effect of the osmotic treatment on water desorption isotherms is clearly observed in Figure 2. Sodium chloride has a water activity depressing effect; in this way, water desorption isotherms of osmosed garlic present higher moisture content at the same water activity when compared with raw garlic isotherms, and also osmosed samples treated with 26% NaCl solutions present higher moisture content than those treated with 10% NaCl solutions. This effect is

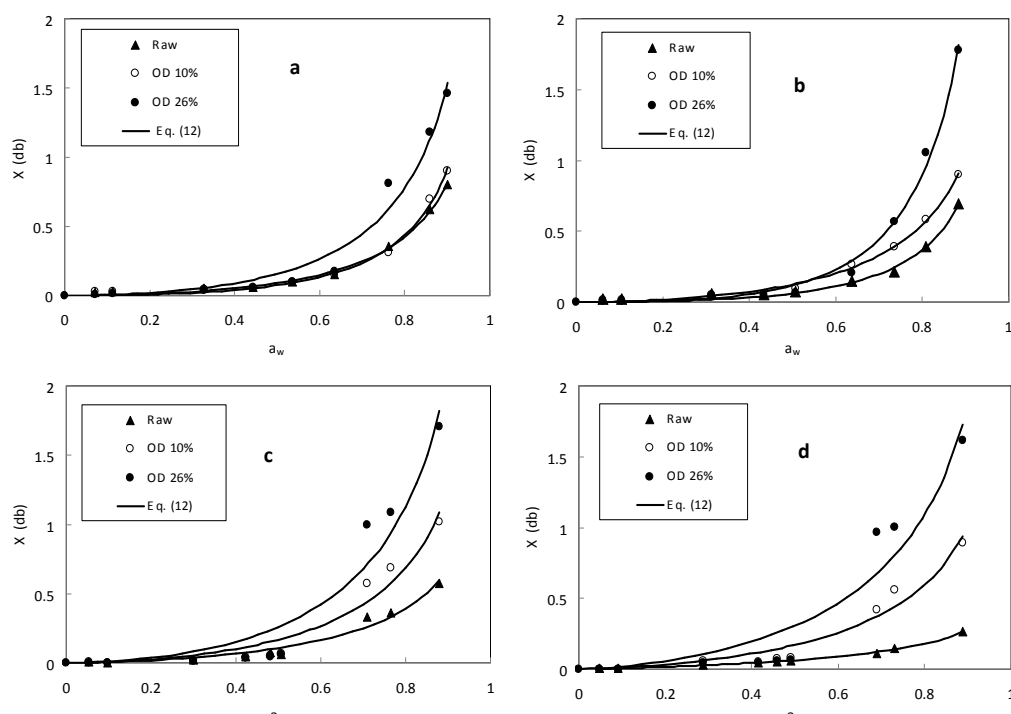


Figure 2. Water desorption isotherms of raw and osmotically dehydrated garlic with NaCl solutions. Dots are experimental data and lines are values obtained by Equation (12). (a) 25; (b) 40; (c) 55, and (d) 70°C.



observed at any water activity although it is more accentuated at high water activities. Water activity depressing effect increases with the temperature, as evidenced by the gap increase among isotherms of raw and osmosed samples with temperature (Figures 2a-d). Similar results were reported for osmotically treated carrots with sodium chloride in which the acquisition of sodium chloride led to food products being more hygroscopic [28].

Temperature has a relevant effect on water desorption isotherms, since an increase in temperature favors the detachment of water molecules from the solid matrix and water evaporation [17]. This effect of temperature on moisture sorption isotherms has also been reported in many studies [20], [28] and [31]. Water activity of garlic increased with increasing temperature at the same moisture content (see sorption isotherms for raw garlic at different temperatures, Figure 2a-d), and this effect was more accentuated at water activity values higher than 0.6. In the case of osmosed samples, the effect of temperature was also clear at low water activity (< 0.5), but above this value, an inversion of the sorption isotherms was observed. This fact can be explained by the relevant increase of sodium chloride content by the osmotic treatment in garlic that promoted the existence of a fraction of non-solubilized sodium chloride even at high water activity values that depend on temperature (at these conditions, the liquid phase is saturated sodium chloride solution). When liquid phase starts to become unsaturated, the amount of water necessary to achieve high water activity interval increases sharply with increasing temperature and, consequently, equilibrium moisture content increases. This fact can be explained by two simultaneous physical phenomena. Sodium chloride solubility varies in a restricted range in the studied interval of temperatures, but water activity of sodium chloride solutions decreases with temperature and, consequently, at constant water activity value, more diluted systems at higher temperatures are necessary.

Water Desorption Isotherms Modeling

Correlation Models

Halsey [9], Equation (8), and Henderson [10], Equation (9), models were selected to correlate water desorption experimental data, since both models are quite simple (only two correlation parameters) and are appropriated for type III sorption isotherms.

$$X = \left(\frac{-a}{\ln a_w} \right)^{\frac{1}{r}} \quad (8)$$

$$X = \left(\frac{-\ln(1-a_w)}{A} \right)^B \quad (9)$$

Where, a , r , A and B are fitting parameters. Henderson model led to better results for tested fits, with $r^2 > 0.97$, $MSE < 0.051$ and $P (\%) < 14.39$ versus $r^2 > 0.85$, $MSE < 0.085$ and $P (\%) < 21.32$ of the Halsey model. Consequently, Henderson model was selected for experimental data modeling and Table 1 shows A and B parameters of Equation (9) at different temperatures and dehydration conditions of garlic.

Parameter B shows restricted and random variations at each temperature for raw and osmosed garlic. An average B value was calculated at each temperature, and was correlated ($r^2 = 0.99$) with absolute temperature (K) through a linear equation:

$$B = 0.0035T - 0.52 \quad (10)$$

Parameter A was correlated ($r^2 = 0.97$) with absolute temperature and the osmotic solution concentration (% w/w, considering 0 for raw garlic) through the following relationship:

$$A = \frac{2.4893 - 0.0069T}{1 - 0.0028T + 0.0022C} \quad (11)$$

By introducing Equations 10) and (11) in Henderson model, it is possible to obtain water desorption isotherms of garlic with 6 fit parameters, in the range of the tested temperatures and osmotic solution concentrations

Figure 2 shows the results of the application of Equation (12) to desorption data. Table 1 shows the goodness of fit of Henderson and Equation (12) models at several temperatures for raw and osmosed garlic. It is assumed that the fit is good when P and MSE are as low as possible. In view of MSE and P values, Henderson model offers a good fit of experimental sorption data, being the better fit at low temperatures and when the gained solids are lower (raw and 10% NaCl solutions). For Equation (12), the fit is not as good as that obtained with Henderson model, but looking at P values, the fit is acceptable. Furthermore, Equation (12) is very useful from a practical point of view since it is an equation valid for all tested temperatures and osmotic treatments.

$$X = \left[\frac{(0.0028T - 0.0022C - 1) \ln(1 - a_w)}{2.4893 - 0.0069T} \right]^{(0.0035T - 0.52)} \quad (12)$$

Prediction Model

Chemical composition data of raw garlic were obtained from literature [29]. Table 2 shows the composition of raw and osmosed garlic with NaCl solutions. Chemical compositions of osmotically dehydrated garlic (with 10 and 26% NaCl solutions)

were evaluated from data of water loss and NaCl gain obtained in this work. Non-soluble component amounts were unvariant and soluble compounds (sugars with low initial content) were evaluated by difference between experimental total dry basis and salt+non-soluble amounts. Results showed that sugars were leached during osmosis and sugar composition of osmosed sample was $< 0.003 \text{ g g}^{-1}$ sample. Furthermore, it was assumed that raw garlic sugars were leached with water flux from garlic slabs to osmotic solution, leaving behind only 10% of the initial sugar content. Data are presented as non-soluble and soluble compounds, since, depending on the component, state water activity calculations are different.

Figure 3 shows, as example, experimental data and predicted water sorption isotherms for raw and osmotically dehydrated garlic with 26% NaCl solution at 25 and 55°C. As observed, the prediction model adequately fits to experimental data for raw and osmotically dehydrated garlic. Table 3 shows the goodness of fit of the prediction model for the different tested process conditions and isotherm temperatures.

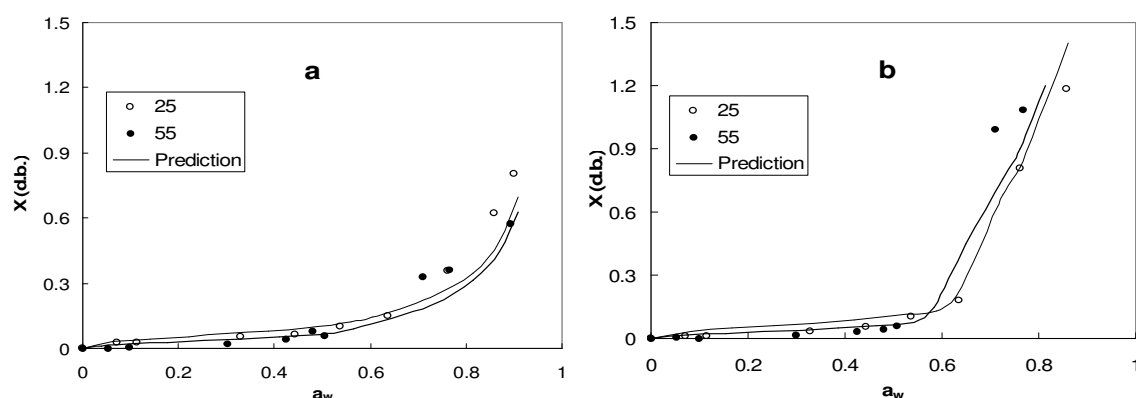
Statistical parameters are satisfactory for all process conditions, although at 70°C the model presents higher deviations to experimental data. This fact can be related to gelatinization process of starch (usually initial temperature of gelatinization is

Table 1. Parameters of Henderson model, Equation (9), and statistical parameters for fit goodness of Equations (9) and (12) at different temperatures for garlic without and with NaCl osmotic treatment.

T (°C)	[NaCl] (%)	Henderson model, Equation (9)					Equation (12)		
		A (db) ⁻¹	B (-)	MSE	P (%)	r ²	MSE	P (%)	r ²
25	0	2.582	0.548	0.001	7.32	0.99	0.009	12.03	0.98
	10	2.401	0.480	0.001	7.50	0.99	0.009	10.92	0.98
	26	1.841	0.528	0.011	9.65	0.98	0.031	10.04	0.98
40	0	2.561	0.466	0.001	8.92	0.98	0.002	10.95	0.98
	10	2.267	0.567	0.001	1.11	0.99	0.007	10.01	0.98
	26	1.683	0.411	0.003	5.53	0.99	0.015	10.06	0.98
55	0	2.948	0.648	0.002	9.32	0.98	0.017	10.17	0.98
	10	2.011	0.597	0.009	11.49	0.98	0.035	9.89	0.98
	26	1.503	0.573	0.051	13.47	0.97	0.062	10.16	0.98
70	0	6.300	0.795	0.001	11.90	0.98	0.001	14.42	0.97
	10	2.293	0.679	0.005	13.68	0.97	0.006	11.24	0.97
	26	1.524	0.668	0.044	15.39	0.97	0.008	8.68	0.98

**Table 2.** Proximal composition of raw and osmotically dehydrated garlic with NaCl solutions.

Component		Raw garlic ^a (g g ⁻¹ dry solid)	Osmosed garlic, [NaCl] 10% (g g ⁻¹ dry solid)	Osmosed garlic, [NaCl] 26% (g g ⁻¹ dry solid)
Water		2.516	2.187	1.978
Non-soluble	Protein	0.213	0.209	0.185
	Fibre	0.060	0.059	0.052
	Starch	0.563	0.553	0.489
	Fat	0.004	0.004	0.004
	Other	0.002	0.002	0.002
Soluble	Salt	0.064	0.163	0.260
	Glucose	0.025	0.002	0.002
	Fructose	0.035	0.003	0.003
	Sucrose	0.035	0.003	0.003

^aAdapted from [29].**Figure 3.** Water desorption isotherms of raw (a) and osmotically dehydrated garlic with 26% NaCl solutions (b) at 25 and 55°C. Dots are experimental values and lines are values obtained by prediction model (20).**Table 3.** Goodness of the prediction model fitting at different temperatures for garlic without and with NaCl osmotic treatment.

T(°C)	[NaCl](%)	MSE	P (%)	r ²
25	0	0.001	8.85	0.98
	10	0.001	10.13	0.98
	26	0.014	9.65	0.98
40	0	0.001	8.77	0.99
	10	0.009	11.01	0.98
	26	0.029	11.45	0.98
55	0	0.001	9.04	0.99
	10	0.004	10.90	0.98
	26	0.012	11.32	0.98
70	0	0.018	16.61	0.97
	10	0.013	18.04	0.96
	26	0.059	19.92	0.95

between 60 and 70°C) that modifies the hygroscopicity of samples. Gelatinization temperature range of starch depends on the starch source, water content, and the presence of other compounds like sugars and salts also modify the temperature of gelatinization interval. Although the model employs water sorption isotherms of isolated starch in the range of tested temperatures, the composition of garlic can modify the gelatinization process and also the hygroscopic characteristics of the food material, especially in samples with high salt content.

The results of the prediction model can be considered satisfactory, taking into account that experimental water desorption isotherms of food materials present important variations even for the same material. Furthermore, an accurate water activity prediction is very complex in multicomponent and multiphasic systems in which highly sophisticated computational models must be implemented. In this sense, this prediction model can be considered as a good approximation to the real food isotherm.

CONCLUSIONS

Osmotic treatment significantly affected the composition of garlic, decreasing water content and increasing NaCl content with the increase of NaCl concentration of the osmotic solution. Garlic isotherms can be classified as type III. The increase of NaCl content in garlic samples led to a decrease in water activity at the same moisture content, and this effect was more accentuated at high temperatures. Water activity increased with increasing temperature at constant moisture content, but at water activity values higher than 0.6, the salt acquired during osmotic treatment inverts this behavior. Nevertheless, below 0.4 of water activity (usual range of relative humidity employed in storage) osmosed and non-osmosed samples show similar equilibrium data. Consequently, all samples can be stored and

handled at the same environmental conditions.

Water desorption isotherms of garlic were satisfactorily modeled by the Henderson model. A six parameters equation based on Henderson model, which correlates moisture content with water activity, temperature, and concentration of the osmotic solution was also satisfactorily fitted to experimental data.

The prediction model based on concentration and physical state of garlic components successfully reproduced water desorption isotherms of garlic obtained experimentally at temperatures less than 55°C. This model can be very useful when it is not possible to obtain sorption isotherms experimentally, or when equilibrium data are necessary in a very short time.

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همدماهای آب زدایی سیر طبیعی و سیر خشک شده با روش اسمزی

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چکیده

در این پژوهش، همدماهای آب زدایی برای برش هایی از سیر طبیعی و سیر خشک شده با روش اسمزی (در حرارت ۲۵ درجه سانتی گراد به مدت ۲ ساعت با محلول ۱۰٪ و ۲۶٪ نمک طعام NaCl) در حرارت های ۲۵، ۴۰، ۵۵ و ۷۰ درجه سانتی گراد و در محدوده ی فعالیت آب بین ۰/۹ و ۰/۰۵ با استفاده از روش وزن سنجی ایستا (static gravimetric method) تعیین شد. همدماهای آب زدایی را می توان در گروه ۳ (Type III) دسته بندی کرد. همدماهای سیر طبیعی به خوبی اثر درجه حرارت را به صورت زیاد شدن فعالیت آب با افزایش حرارت در یک مقدار رطوبت ثابت نشان دادند درحالیکه برای سیر خشک شده با اسمزی اثر درجه حرارت اهمیت کمتری داشت. در مورد سیر خشک شده با اسمزی، زیاد شدن مقدار درصد نمک طعام منجر به کاهش فعالیت آب در همان مقدار رطوبت شد و این اثر در حرارت های بالا تشدید شد. داده های رطوبت در حالت تعادل در هر درجه حرارت به صورت مناسبی با مدل هندرسن برازش شدند و بر مبنای این مدل و به منظور ایجاد همبستگی همزمان بین مقدار رطوبت موجود با فعالیت آب، درجه حرارت، و غلظت محلول اسمزی، معادله ای با شش پارامتر به دست آمد. نیز مدلی پیشگو بر پایه غلظت و حالت فیزیکی ترکیبات سیر با موفقیت به کار رفت تا بتوان داده های آزمایشی را بازتولید کرد.