Thermodynamical properties of conserved thyme after gamma irradiation and thermal-biochemical treatment

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Abstract

Aromatic and medicinal plants are more asked in trading. Especially for some endemic plants as Thymus Satureioides, industrials are in need to keep it available to customers all the year. Thus, the conservation process allows the preservation and the storage of plants with great hygienic and safety quality. However, these processes can modify the food proprieties. Hence, we are interested in the effectiveness of preservation processes on the thermodynamic properties. The sorption isotherms were investigated using saturated salt solution method, at temperatures in the range of 30, 40 and 50°C and water activity ranging from 0.05 to 0.9. The experimental data of moisture sorption were exploited to identify the thermodynamic properties of the witness and conserved thyme such as differential enthalpy and entropy of sorption. The experimental data showed that enthalpy-entropy compensation theory was related to the moisture sorption conduct of thyme. Moreover, the conservative process influences the behavior of thermodynamic properties of thyme.

Key words: Irradiation, conserved thyme, food storage, sorption isotherms, solar energy, thermodynamic properties.

1. Introduction

The sorption isotherms determine the water activity that must be achieved during drying to approach a stable steady state, providing better storage [1-2]. The sorption isotherms are an excellent way to determine the thermodynamic properties and the distribution and intensity of the connections of water and its availability [3]. *Thymus satureioides* is an endemic plant of morocco and it is commonly used as spices and traditional medicine remedies. *Thymus satureioides* is a well used plant [4]. It is used not only as an infusion, but also for flavouring food. It has been reported to have multiple biological effects such as antispasmodic, antibacterial, anti-inflammatory, antioxidant and antihemolytic activities [5- 6]. Several studies involved the usefulness of *Thymus satureioides*, its compositions, uses, biologic and therapeutic effects [7-8]. Thus, highest quality and safety of the plant required the application of the conservation process. However, there is no scientific report about the conservation and its influence on the thermodynamic properties and the storage conditions. Hence, our study links the contrast between the transfer of water in the Medicinal and Aromatic Plants (MAPs) "*Thymus satureioides*" and the thermodynamic properties in hygroscopic equilibrium with the conservation process.

Our purpose is the experimental determination of thermodynamic properties for witness and conserved thyme by ionization and thermobiochemical treatment using renewable energy. The experimental sorption curves were identified and described by GAB's model.

Further analysis of sorption isotherm data by application of thermodynamic principals can provide information regarding differential enthalpy or the isosteric heat of sorption and differential entropy. Differential enthalpy of sorption gives an indication of the amount of bound water existing in the product. The differential entropy of material is proportional to the number of its available sorption sites at a specific energy level [9]. Subsequently

we determine the thermodynamic functions (differential enthalpy, differential entropy) and we describe the experimental data using selected mathematical models. Moreover, we recognized the linear relation exists between the enthalpy and entropy of the sorption reaction.

2. Materials and methods

2.1 The sorption isotherm :

The plants studied were treated by two preservation methods. The untreated thyme was dried at room temperature. The irradiated thyme was preserved by gamma irradiation at low dose 1KGy [10]. The treated thyme was preserved by combined thermo-biochemical treatment which is based on the pulverization of citric acid followed by drying at 80°C using a solar dryer with convective heat [11]. Fresh thyme was used in desorption experiments for untreated thyme. However, the irradiated and treated thyme were humidified at 25%. In our study, a static method is used to identify the hygroscopic equilibrium [12]. The method is based on the use of different saturated salt solutions KOH, ($MgCl_2$, $6H_2O$), K_2CO_3, $NaNO_3$, KCl and ($BaCl_2$, $2H_2O$) to maintain a fixed relative humidity Rh. The standard salts used and the corresponding water activities of their saturated solutions ($a_w = (Rh/100) \in [0.05 - 0.90]$) at different temperatures were based on the values given by Greenspan [13]. The atmosphere surrounding the product has a fixed relative humidity for every working temperature imposed on the system. The mass transfers between the product and the ambient air are assured by natural diffusion of the water vapor.

The experiment based on putting the samples in glass jar contains different saturated standards salts so as to have various water activities. It consists of six glass jars of 1 l each with an insulated lid. The sample holder containing 0.3 g (±0.001) of aromatic plant is placed in the glass jar.

In order to determine the equilibrium moisture content EMC, Xeq, the samples are weighed every two days. As soon as the masses become stationary, the experiment is stopped and the samples are weighed and placed in a drying oven whose temperature is fixed at 105°C for 24 hours.

Consequently, the difference of mass before (M_w) and after (M_d) drying determines their equilibrium moisture content of the product at hygroscopic equilibrium EMC, Xeq:

$$EMC = X_{eq} = \frac{M_w - M_d}{M_d} \tag{1}$$

All the experimental treatments were conducted in three replicates. Statistical analyses were performed in order to estimate the effect of conservation conditions on the thermophysical properties of thyme by using OriginPro 8.1 for Windows to plot the curves and the ANOVA test was applied. The P<0.05 was considered significant.

2.2 Modelling by GAB equation:

The relationship between EMC, water activity, and temperature for Thyme was predicted in literature by several mathematical models [13-14]. In our study, we applied the GAB model (Guggenheim-Anderson-de Boer).

Van Den Berg and Bruin [15] stated that the "GAB" equation (Guggenheim-Anderson-de Boer) was the best theoretical model for foods. It is a three parameter equation [16]. The use of the Guggenheim-Anderson-de Boer model is generalized by its theoretical bases, mathematical simplicity, and ease to interpret; but it is not frequently used when the water activity greater than 0.93 [17-18]. The equation is:

$$X_{eq} = \frac{ABCa_w}{[1 - Ba_w][1 - Ba_w + BCa_w]} \tag{2}$$

The parameters B and C in the equation can be correlated with GAB temperatures using the Arrhenius equation [21]

$$B = B_0 \exp(\Delta H_1 / R.\theta) = B_0 \exp(h_1 / R.\theta) \tag{2.1}$$

$$C = C_0 \exp(\Delta H_2 / R.\theta) = C_0 \exp(h_2 / R.\theta) \tag{2.2}$$

Where:

A (or X_M): water content corresponding to the saturation of the monolayer Brunauer (% DM)

B (or K) : correction factor of the properties of adsorption of additional layers with respect to the liquid water

ΔH_1 (h_1): heat of condensation of the steam of pure water (kJ mol-1)

ΔH_2 (h_2): integral heat of sorption of additional water layers (kJ mol-1)

θ Absolute temperature (K)
R   gas constant (8,314 J.mol^{-1}.K^{-1})
B_0 and C_0 are constant

Levenberg-Marquardt nonlinear optimization method using appropriate software (CurveExpert and OriginPro 8.1) is used for the calculation of model coefficients and the statistical parameters: mean relative error (MRE) and the correlation coefficient (r).

Mean Relative Error:

$$MRE = \frac{100}{N} \sum_{i=1}^{N} \left| \frac{X_{eq_{i,exp}} - X_{eq_{i,pred}}}{X_{eq_{i,exp}}} \right| \tag{3}$$

Correlation coefficient:

$$r = \sqrt{\frac{\sum_{i=1}^{N} \left(Xeq_{i,pred} - \overline{Xeq}_{i,exp} \right)^2}{\sum_{i=1}^{N} \left(Xeq_{i,exp} - \overline{Xeq}_{i,exp} \right)^2}} \tag{4}$$

Where:

$X_{eq_{i,exp}}$ ith Experimental moisture content (%d.b),

$X_{eq_{i,pred}}$ ith Predicted moisture content (%d.b),

N   number of data points.

2.3   Differential enthalpy and differential entropy of sorption

The differential enthalpy or isosteric heat of sorption (ΔH_d) indicates the state of absorbed water by the solid material. The net isosteric heat of sorption (Δh_d) represents the quantity of energy exceeding the heat of vaporization of water (ΔH_{vap}) associated with the sorption process.

$$\Delta h_d = \Delta H_d - \Delta H_{vap} \tag{5}$$

The net isosteric heat of sorption can be calculated from the experimental data using the Clausius-Clapeyron equation [19]:

$$\left[\frac{d(\ln a_w)}{d(1/\theta)} \right]_{X_{eq}} = \frac{-\Delta h_d}{R} \tag{6}$$

This relation requires determining the isotherms at various temperatures in order to calculate the variation logarithmic curve of the water activity according to the reverse of the temperature, for a water content fixed. However, starting from the modified equations, it is possible to obtain the analytical expression of the heat of sorption Δh_d. This approach assumes that isosteric heat ΔH_d does not vary with the temperature [19]. The relationship between the net isosteric heat Δh_d and the differential entropy ΔS_d of sorption is given by:

$$(-\ln a_w)_{X_{eq}} = \frac{\Delta h_d}{R.\theta} - \frac{\Delta S_d}{R} \tag{7}$$

The experimental sorption isotherm data was plotted in the form of $\ln(a_w)$ versus $1/\theta$ for specific moisture content (X_{eq}). We determined Δh_d from the slope $(\Delta h_d / R)$ and ΔS_d from the intercept $(\Delta S_d / R)$. Applying this at different moisture contents allowed determining the dependence on Δh_d and ΔS_d [20].

2.4   Enthalpy-Entropy Compensation Theory

According to the theory of compensation, the linear relationship between the enthalpy Δh_d and the entropy ΔS_d for a specific reaction are given by:

$$\Delta h_d = T_\beta . \Delta S_d + \Delta G_\beta \tag{8}$$

The isokinetic temperature (T_β) is a characteristic property of the food surface whose dimension is absolute temperature. It represents the temperature to which all the reactions of the series proceed to the same rate. The free energy (ΔG_β) provides a criterion to evaluate if the process of the water sorption is spontaneous ($-\Delta G_\beta$) or not ($+\Delta G_\beta$). This process is repeated for several values of equilibrium relative humidity with an aim of underlining the relation between the differential entropy of sorption and the equilibrium moisture content of the studied product [21]. The isokinetic temperature (T_β) and constant (ΔG_β) were calculated using linear regression. The isokinetic temperature T_β represents the slope of the linear function between the differential entropy and the differential enthalpy of sorption [9].

3. Results and discussion

3.1 Adsorption and desorption isotherms

The *Thymus satureioides* experimental data of desorption isotherms (X_{eq}=f (a_w)) are illustrated in Fig. 1 for 3 temperatures (30, 40 and 50 ° C). The isotherms have a sigmoid shape (type II), which is common for many hygroscopic products. From the figure, we observe the temperature dependence of the sorption behavior. For any constant water activity, an increase in temperature significantly decreases the equilibrium moisture content EMC. This effect is due to the activation of the water molecules by temperature that split them from water binding sites, which lowers the equilibrium moisture content [22-23]. This is in agreement with other results reported in the literature [24--25-26-27-28-29].
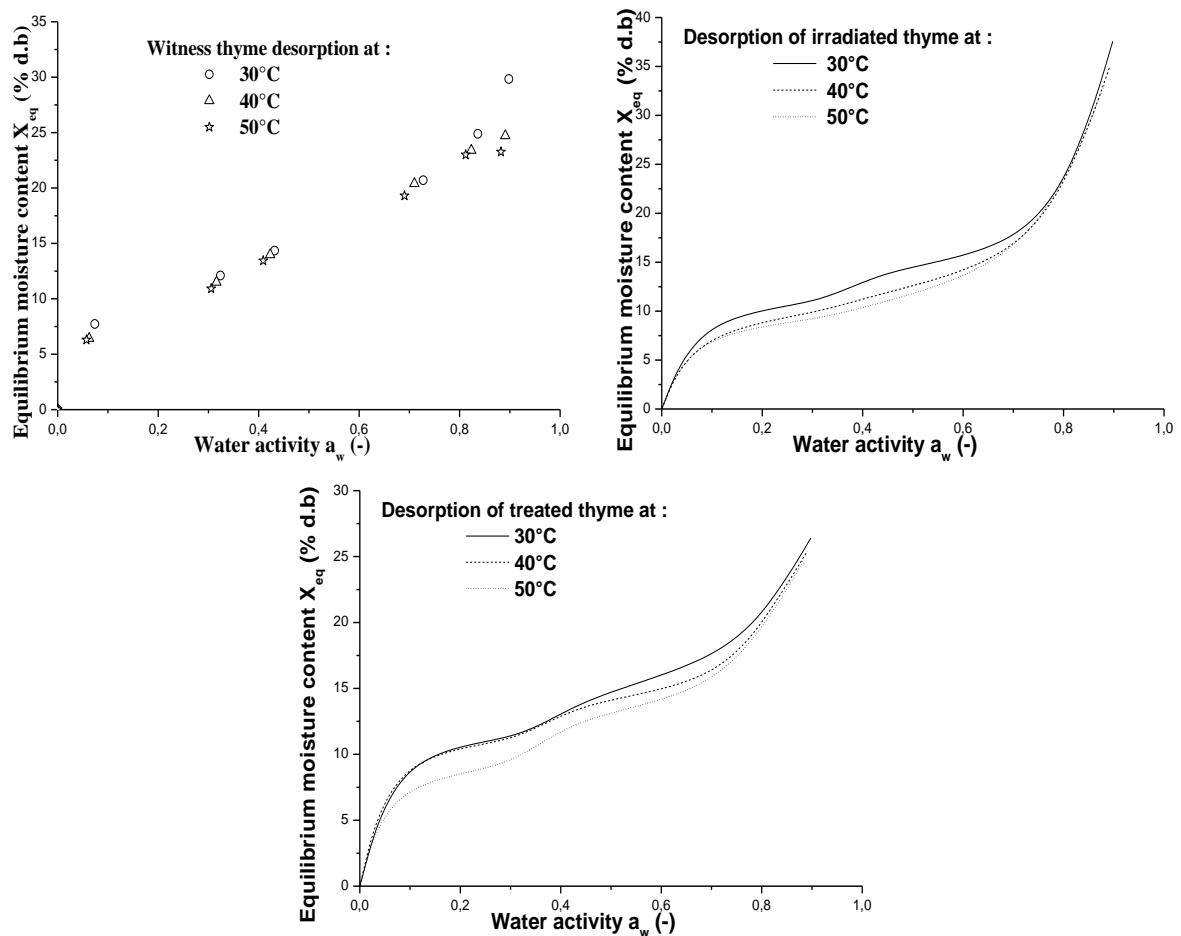

Figure 1: Desorption isotherms of untreated, irradiated and treated Thyme at 30°, 40° and 50°C

The presentation of adsorption and desorption experimental data in the same plot show that the plant does not have the same hygroscopic equilibrium behavior (Fig. 2). For the same constant of water activity, the water content of desorption is higher than that of adsorption. Indeed, there exists a phenomenon of hysteresis. For the

reason that the bound fraction is always larger on desorption than on adsorption, hysteresis in the sorption isotherm is a consequence of variation in the fraction of bound water present in the adsorption and desorption processes [30-31].

The assessment of sorption isotherms for witness and conserved thyme by gamma irradiation and combined thermo-biochemical treatment illustrate that they have the same hygroscopic conduct.
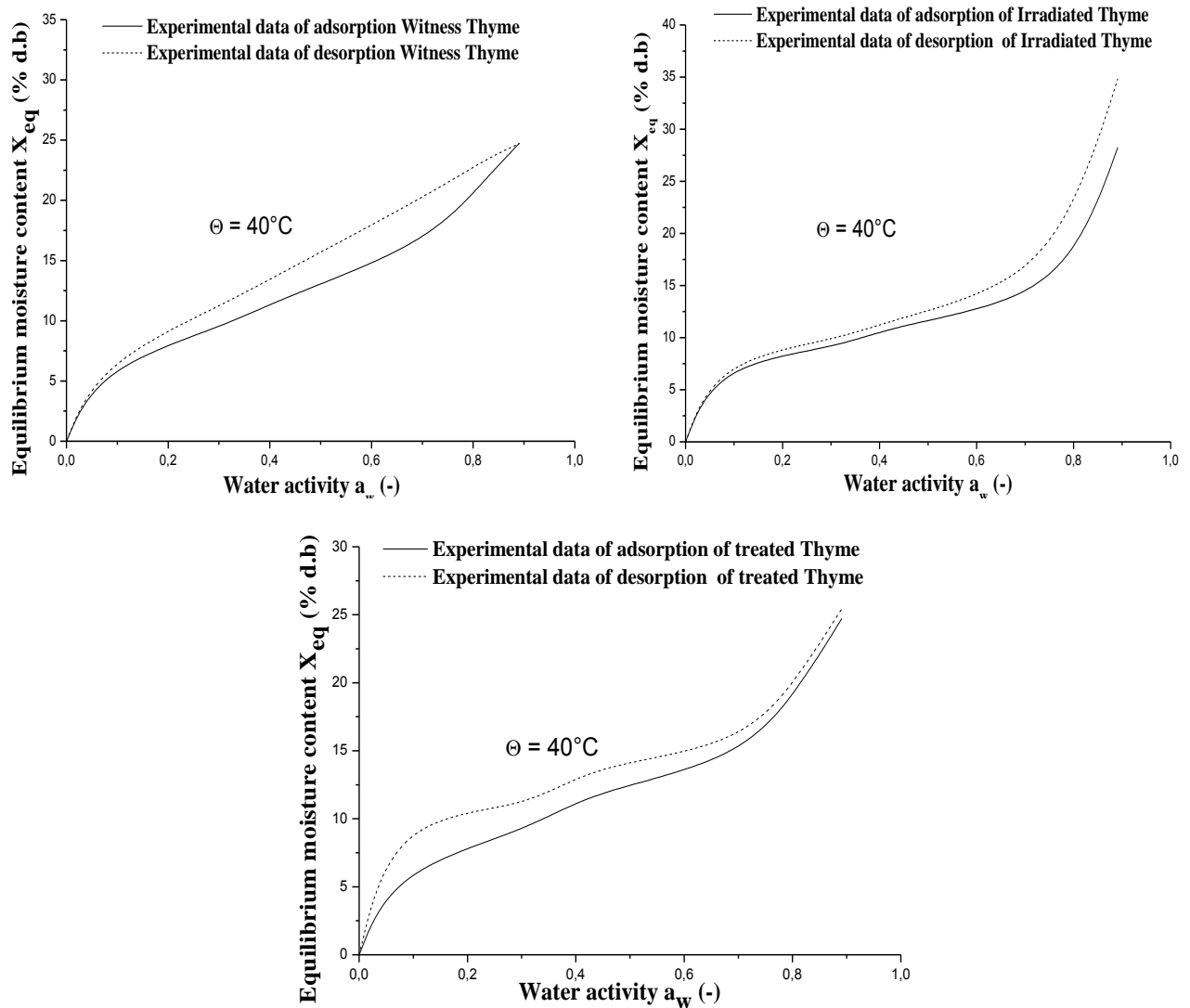

Figure 2: Sorption hysteresis phenomenon of *Thymus satureioides*

3.2 Modelling of sorption experimental data:

The experimental data of the adsorption and desorption curves of witness and preserved thyme were fitted to GAB sorption model (Tables 1).

The values of the monolayer moisture content at each temperature as calculated by the GAB equation are presented in Table 2. The results show that the monolayer moisture content X_m decreases with increasing temperature between 30-50°C. Except for witness thyme sorption and irradiated thyme (adsorption). This behaviour has also been reported for several foods and food systems by Iglesias and Chirife [23].

We can deal out the difference of sorption to the preservation process. The addition of citric acid to preserved thyme led to modification of absorption, due to the mechanical mixtures of plant molecules and citric acid. Also, the high drying temperatures used for the conservation, some water molecules are activated on energy levels that allow them to split from their sites [32]. However, the sorption behavior of irradiated thyme is due to the influence of gamma irradiation on bio-chemical and physical-chemical parameters [33].

Table 1: Estimated GAB model's coefficients, r, EMR fitted to adsorption and desorption isotherm

		T (°C)	Coefficients			r	EMR
			a	b	c		
Witness thyme	Adsorption	30	0.841	$7.220 \ 10^9$	6.930	0.991	6.310
		40	0.803	$2.260 \ 10^{10}$	7.150	0.992	6.712
		50	0.803	$1.970 \ 10^{11}$	6.912	0.990	8.111
	Desorption	30	0.749	55.101	9.681	0.997	6.750
		40	0.635	26.129	11.333	0.997	3.278
		50	0.634	29.071	10.985	0.992	4.537
Irradiated thyme	Adsorption	30	0.888	$6.150 \ 10^{11}$	5.870	0.971	13.161
		40	0.874	$1.930 \ 10^{11}$	5.942	0.977	11.421
		50	0.915	$1.370 \ 10^{11}$	5.208	0.988	11.363
	Desorption	30	0.900	$8.130 \ 10^{10}$	6.918	0.973	6.750
		40	0.920	$3.640 \ 10^{11}$	6.154	0.989	3.278
		50	0.928	$6.250 \ 10^{10}$	5.941	0.993	4.537
Treated thyme	Adsorption	30	0.803	$4.210 \ 10^{10}$	7.006	0.991	4.674
		40	0.803	136.100	6.930	0.988	5.248
		50	0.966	57.614	3.632	0.991	7.498
	Desorption	30	0.724	$1.310 \ 10^{11}$	8.955	0.984	4.674
		40	0.725	$5.590 \ 10^{10}$	8.624	0.968	5.248
		50	0.790	$6.070 \ 10^9$	7.278	0.982	7.498

Table 2: GAB's estimated coefficients of sorption isotherm for witness, irradiated and treated thyme

		Witness thyme		Irradiated thyme		Treated thyme	
		Adsorption	Desorption	Adsorption	Desorption	Adsorption	Desorption
Co	Co	$7.921 \ 10^{32}$	$1.361 \ 10^{-3}$	$4.051 \ 10^{16}$	$3.422 \ 10^9$	$6.288 \ 10^{-136}$	$6.048 \ 10^{-11}$
h2	hc	$-1.341 \ 10^5$	$2.640 \ 10^4$	$-3.317 \ 10^4$	$9.311 \ 10^3$	$8.389 \ 10^5$	$1.244 \ 10^5$
Bo	ko	0.393	0.048	1.418	1.477	14.854	2.94
h1	hk	1896.742	6848	-1206.524	-1243.475	-7430.684	-3568.964
A	Xm	6.997	10.666	5.673	6.337	5.856	8.285

3.3 Enthalpy and entropy of sorption

The differential enthalpy of witness and preserved thyme was calculated by applying the Clausius-clapeyron equation (6) to the experimental equilibrium isotherm data. The variation of the heats of adsorption and desorption of the three plants is shown in figure 3. The isosteric heats Δh_d and entropy ΔS_d are sufficiently represented by polynomial functions (table 3). As seen in this figure, at low equilibrium moisture contents, the heat of sorption is high at equilibrium moisture contents (6-15%). This suggested that the high values of heats of sorption at low equilibrium moisture contents were due to the existence of highly active polar sites on the surface of the product, which are covered with water molecules forming a mono-molecular layer. In addition, the isosteric heat of desorption is greater than of adsorption at low moisture contents for witness and irradiated thyme in opposition to treated thyme. Moreover, the witness thyme heat is higher than irradiated thyme heat, which is followed by the treated thyme. The variation of isosteric heat of sorption was similar to those observed by other researchers [34-35-36-37-38]. The differential entropy is plotted as a function of equilibrium moisture content in figure 4. The entropy data put on view a strong dependence on moisture content. This reflects the differing strength of water binding function of the conservation process.
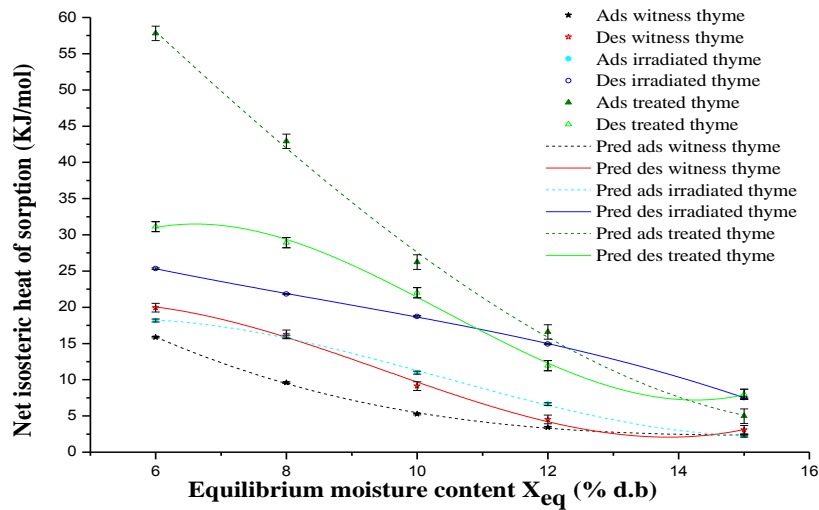
Figure 3: Net isosteric heat of sorption for witness, irradiated and treated thyme as a function of equilibrium moisture content
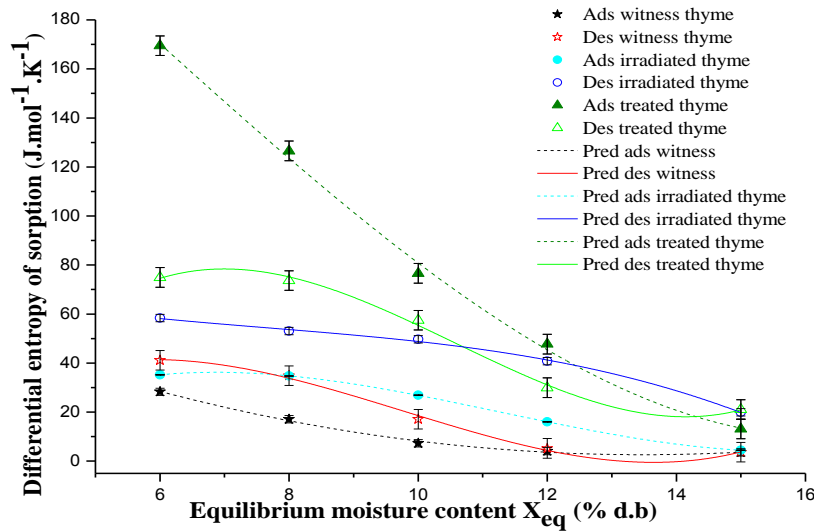

Figure 4: Differential entropy of adsorption and desorption of witness, irradiated and treated thyme as a function of equilibrium moisture content

Table 3: Empiric expressions of net isosteric heat Δh_d and differential entropy ΔS_d of adsorption and desorption for untreated, irradiated and treated thyme

	Δh_d (KJ.mol-1)	ΔS_d (J.mol-1.K-1)
Witness thyme (ads)	$55.48-9.70X_{eq}+0.59X_{eq}^2-0.01X_{eq}^3$ r = 0.998	$86.59 -12.48X_{eq}+0.47X_{eq}^2-10^{-4}X_{eq}^3$ r = 0.987
Witness thyme (des)	$-5.87+11.80X_{eq}-1.58X_{eq}^2+0.06X_{eq}^3$ r =0.989	$-67.46+43.22X_{eq}-5.26X_{eq}^2+0.18X_{eq}^3$ r =0.986
Irradiated thyme (ads)	$-2.73+8.71X_{eq}-1.08X_{eq}^2+0.03X_{eq}^3$ r = 0.996	$-53.89+30.75X_{eq}-3.22X_{eq}^2+0.10X_{eq}^3$ r = 1.000
Irradiated thyme (des)	$42.11-4.18X_{eq}+0.30X_{eq}^2-0.01X_{eq}^3$ r=1.000	$92.75 -10.72X_{eq}+1.12X_{eq}^2-0.05X_{eq}^3$ r=0.994
Treated thyme (ads)	$108.25 -7.73X_{eq}-0.21X_{eq}^2+0.02X_{eq}^3$ r = 0.992	$299.37 -16.77X_{eq}-1.22X_{eq}^2+0.07X_{eq}^3$ r = 0.992
Treated thyme (des)	$-53.96+30.61X_{eq}-3.39X_{eq}^2+0.11X_{eq}^3$ r=0.993	$-208.11+98.08X_{eq}-10.48X_{eq}^2+0.33X_{eq}^3$ r=0.987

3.4 Compensation theory of sorption

The Δh_d and ΔS_d values for adsorption and desorption, at given moisture contents, were calculated by linear regression equation using Equation 7. It was assumed that, at specific moisture content, Δh_d and ΔS_d did not vary with temperature [21]. Figure 5, shows a linear relation, with a correlation of determination (r =1). This indicates that the compensation exists. The isokinetic temperature (T_β) and the free energy (ΔG_β) parameters (Equation 8) were calculated from the data by linear regression, and the values are detailed in Table 4. The desorption data of isokinetic temperature gave lower values than those for adsorption for witness and irradiated thyme opposed to treated thyme.

Table 4: Characteristic parameters for $\Delta h_d / \Delta S_d$ Relationship

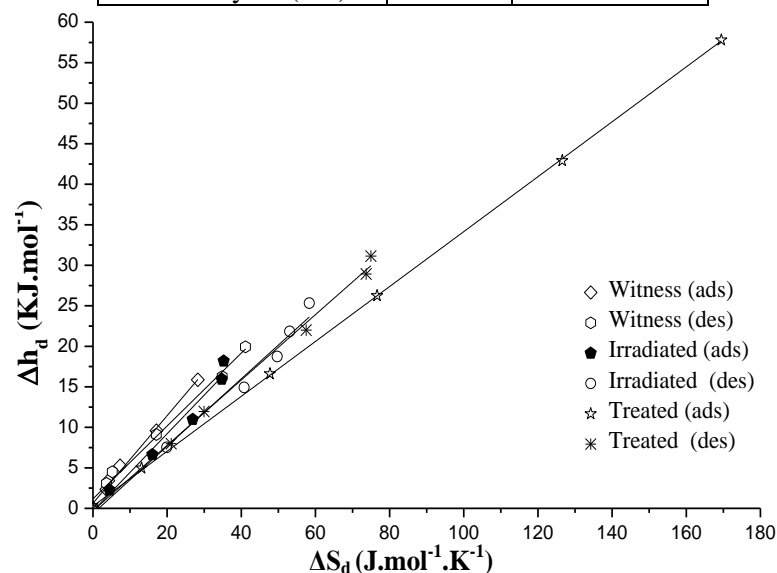|	T_β (K)	ΔG_β (J.mol^{-1})
Witness thyme (ads)	540.220	638.020
Witness thyme (des)	449.800	1175.760
Irradiated thyme (ads)	476.840	-324.890
Irradiated thyme (des)	415.920	-642.740
Treated thyme (ads)	338.820	274.220
Treated thyme (des)	403.390	-290.520

Figure 5: Compensation theory of enthalpy/entropy

Conclusion

1. The identification of the hygroscopic and thermodynamic characteristics of natural and the preserved thyme by gamma irradiation and thermal- biochemical treatment using solar energy.
2. The sorption isotherms of untreated and conserved thyme have been determined by experiment and then modelled by GAB model's.
3. The temperature effect on the sorption isotherms.
4. The decreased of net isosteric heat of sorption and differential entropy values of thyme with increasing moisture content.
5. The isosteric heat of sorption versus entropy data satisfies the enthalpy-entropy compensation theory.
6. The methods applied to the plant conservations change its thermodynamic properties.
7. The influence of the gamma radiation on the molecules stability of the water incorporated in the product.
8. The thermobiochemical treatment influences the sorption and the thermodynamic properties of thyme due to the influence of temperature on water structure and also to the citric acid used.

Acknowledgements

Notations

A, B and C model coefficients
Ads   adsorption
a_w water activity
d.b.   Dry weight basis
Des desorption
$EMC = X_{eq}$ Equilibrium moisture content (Kg water/(kg d.b.))

GAB Guggenheim, Anderson and De Boer model
M_d mass of dry matter (kg)
MRE mean relative error (%)
M_w mass of wet matter (kg)
N   number of data points
Pred predicted

r correlation coefficient
R universal gas constant (8.3145 J.mol-1.K-1)
Rh equilibrium relative air humidity (%)
T Absolute temperature (K)
T_β isokinetic temperature (K)

$Xeq_{i,exp}$ experiment EMC (% d.b)
$X\,eq_{i,pred}$ predicted EMC (% d.b)
ΔG_β free enthalpy at the isokinetic temperature, J.mol-1
Δh_d isosteric heat of sorption, J. mol-1
ΔS_d entropy of sorption, J.mol-1.K-1
θ Temperature (°C)

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