Moroccan Journal of Chemistry

Comparative sorption isotherms of conserved Thymus satureioides

Lahnine L[1,*], Ouhammou M[1], Idlimam A[2], Mahrouz M[1], Lamharrar A[2], Hanine H[3], Bouchdoug M[1], Kouhila M[2]

[1] *Research Team of Innovation and Sustainable Development & Expertise in Green Chemistry ERIDDECV, Department of Chemistry, Cadi Ayyad University, B.P. 2390, Marrakesh 40000, Morocco*

[2] *Laboratory of Solar Energy and Medicinal Plants, Teacher's Training College, Cadi Ayyad University BP 2400 Marrakesh, Morocco*

[3] *Laboratoire de Valorisation et Hygiène des Produits Agro-Alimentaires, Université My Slimane, Beni mellal, Maroc*

** Corresponding author: lamyaelahnine@gmail.com , Tel: (+212659874992)*

Received 11 Nov 2015, Revised 10 Jan 2016, Accepted 17 Jan 2016

Abstract

Thymus satureioides is an endemic plant of Morocco used for its virtues in traditional medicine. This natural plant in comparison with the preserved one by gamma irradiation and thermal-biochemical treatment using a solar dryer, were investigated at various temperatures and water activity using a saturated salt solution method. Thus, it was necessary to study the effect of preservation processes on the storage conditions. The sorption curves of thyme decreased with the increase in temperature at constant relative humidity and the hysteresis effects were observed. The experimental data of sorption were described by six equations. The Enderby's model was found to be the most suitable for describing the sorption curves for untreated and irradiated thyme. In opposition, treated thyme was approached by Peleg's model. Moreover, the optimum water activity of storage was investigated.

***Keywords**: Thymus satureioides*, sorption isotherms, irradiation, solar energy.

1. Introduction

Thymus satureioides, endemic plant of Morocco, is categorized by its affluent composition in carvacrol (26.5 %), borneol (20.1 %), thymol (14.09 %), camphene (8 %) and p-cymene (5.4 %) [1]. Due to its chemical composition specially the borneol, this plant is used for its analgesic, antioxidation and anti-inflammation effect [2], [3].

Making plants suitable for human consumption and commercialization, acquire high physic-chemical and hygienic qualities of the plant material. The use of industrial preservation process was tool promising solutions to prevent the contamination and to facilitate storage and transportation [4]. Hence, enormous

conservation processes have been developed for plant materials among them: vapor treatments, ohmic heating, thermal shock (HTST: High Temperature Short Time), Controlled Sudden Decompression (DIC), pulsed light or microwave [5]–[7]. These processes may cause partial deterioration of organoleptic attributes of plant materials and food in general [8]–[10]. Drying and gamma irradiation are the most used processes for plants conservation [13-14]. Drying process provides valuable conservation. The industry used it in order to preserve and store the seasonal plants and make them available to consumers at low cost [15-16]. Beside, gamma irradiation improves the hygienic quality of food and various herbal materials and reduces losses due to microbial contamination. Furthermore, gamma irradiation has several advantages in increasing yield, improving the content of polyphenol compound and the antioxidant activity [17-18].

Consequently, our study's objectives are performed on thyme plants in order to delineate its endurance after irradiation and thermal-biochemical preservation process. Our study lies on the influence of food preservation process on the transfer of water in the Medicinal and Aromatic Plants (MAPs) "*Thymus satureioides*" and the explanation of these phenomena by characterization of thyme. The experimental sorption isotherms were identified and modelled for untreated and treated thyme by gamma irradiation (1KGy) and by thermobiochemical treatment using renewable energy. Three temperatures (30, 40, and 50 °C) were chosen to typify tropical storage conditions. Using the saturated salt solution method, the equilibrium curves are determined.

The experimental sorption curves are modelled by six different models to identify the most appropriate mathematical model for a better description of the product equilibrium state. Then, we investigate from the experimental data the optimal water activity for the storage of untreated and conserved thyme.

2. Materials and methods

2.1 Treatments & sorption study:

The plants *Thymus satureioides*, used in our study, were collected at Agounedis-Ijokak (High Atlas of Morocco). The untreated thyme was dried at room temperature. The samples of plant studied were treated by two preservation methods. The treated thyme was preserved by 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 [17]. The irradiated thyme was preserved by gamma irradiation at low dose 1KGy [15]. Fresh thyme was used in desorption experiments for untreated thyme. However, the irradiated and treated thyme were humidified at 25%.

The hygroscopic equilibrium is achieved by a static method [18]. The method is based on the use of saturated salt solutions to maintain a fixed relative humidity Rh. The mass transfers between the product and the ambient air are assured by natural diffusion of the water vapor. The experiment consists in putting the samples in glass jar contain different standards saturated salts (KOH, $MgCl_2$, $6H_2O$, K_2CO_3, $NaNO_3$, KCl, $BaCl_2$, $2H_2O$) so as to have a relative humidity which varies from 5% to 90%. The samples are weighed every two days in order to determine their equilibrium moisture content EMC, Xeq. The experiment is stopped when the six masses become stationary. Thus, the difference of mass before (M_w) and after (M_d) drying, in a drying oven whose temperature is fixed at 105°C for 24 hours, determines their equilibrium moisture content EMC:

$$EMC = X_{eq} = \frac{M_w - M_d}{M_d} \quad (1)$$

2.2 Modelling equations

The relationship between water activity, temperature and EMC, was envisaged in literature by several mathematical models [21-22]. In our study, six models have been applied: modified Henderson's model, modified Oswin's model, modified Halsey, model Enderby's model, LESPAM's model and Peleg's model. Table 1shows the models equations.

Table 1: Mathematical models used in our study

Model's name	Expression of the model	References
Enderby	$X_{eq} = \left[\frac{A}{1-B\,a_w} + \frac{C}{1-D\,a_w}\right] a_w$	[21]
Modified Halsey	$X_{eq} = \left[\frac{(-\exp(A+B.\theta)}{\ln(a_w)}\right]^{(1/c)}$	[20]
Modified Henderson	$X_{eq} = \left[\frac{-\ln(1-a_w)}{A(\theta+B)}\right]^{1/c}$	[22]
Modified Oswin	$X_{eq} = (A+B.\theta)\left[\frac{a_w}{1-a_w}\right]^{C}$	[23]
LESPAM	$X_{eq} = A.\exp\left(B\,a_w/\theta\right) + C$	[24]
Peleg	$X_{eq} = A(a_w)^C + B(a_w)^D$	[25]

The statistical parameters mean relative error (MRE) as a percentage was used to determine the quality of the fit. In addition, the correlation coefficient (r) was one of the primary criteria for selecting the best equation to fit the six models to the experimental data [26]. Levenberg-Marquardt nonlinear optimization method using appropriate software is used for the calculation of model coefficients that describe the equilibrium curves and their statistical parameters: mean relative error (MRE) and the correlation coefficient (r).

Mean Relative Error:

$$MRE = \frac{100}{N}\sum_{i=1}^{N}\left|\frac{Xeq_{i,exp} - Xeq_{i,pred}}{Xeq_{i,exp}}\right| \quad (3)$$

Correlation coefficient:

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

3. Results and discussion

3.1 Adsorption and desorption isotherms

The hygroscopic equilibrium of desorption for fresh, irradiated and treated Thyme was accomplished in 15 days. The hygroscopic equilibrium of adsorption for witness Thyme (dried), irradiated and treated thyme was carried out respectively in 17, 18 and 15 days. Experimental data of sorption isotherms (X_{eq}=f (a_w)) of *Thymus satureioides* for 3 temperatures (30, 40 and 50 °C) are illustrated in Fig. 1. The isotherms have a sigmoid curve (type II), which is common for many hygroscopic products [30-31].

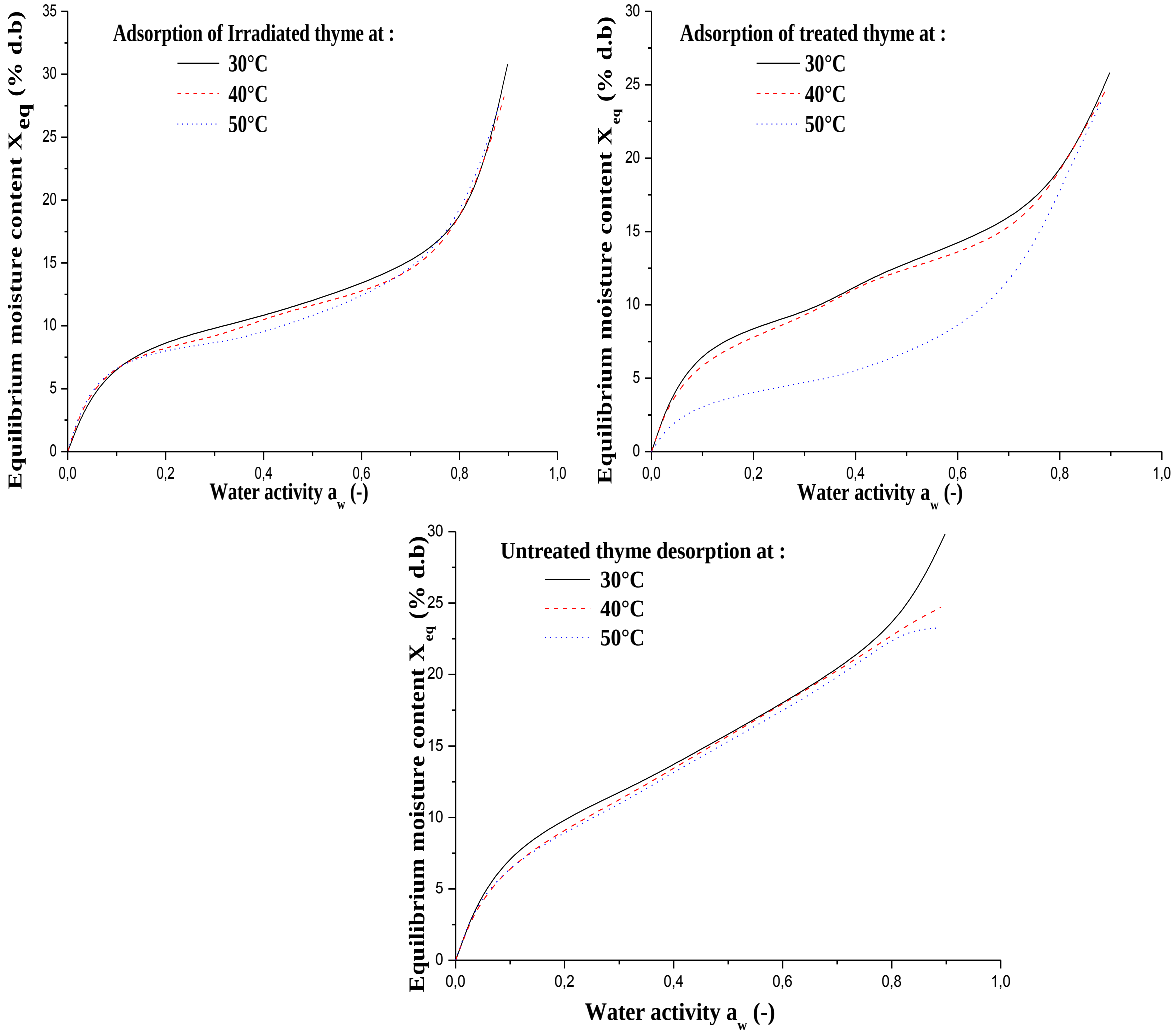

Figure 1: Sorption isotherms of irradiated, treated and untreated thyme at 30, 40 and 50 °C.

The equilibrium moisture contents results of untreated thyme, irradiated thyme by gamma irradiation at 1 kGy and treated thyme by thermal-biochemical process at six water activities for three different temperatures are given in tables 2 and 3.

It can be outstanding from the figures the temperature dependence for the sorption behaviour. An increase in temperature significantly decreases the equilibrium moisture content EMC for any constant water activity. The increase in temperature activates the water molecules and causes them to split from water binding sites, thus lowering the equilibrium moisture content. This is in agreement with other results reported in the literature in case of *Thymus satureioides* [29] as well as other plants, such as *Mentha crispa* L., *Opuntia ficus indica*, coffee fruits and *Gelidium sesquipedale* [30]–[33].

Table 2: The equilibrium moisture content Xeq of untreated thyme for various water activity and temperature

Isotherm at 30 °C			Isotherm at 40 °C			Isotherm at 50 °C		
a_w	X_{eq} (% d.b)		a_w	X_{eq} (% d.b)		a_w	X_{eq} (% d.b)	
	Ads	Des		Ads	Des		Ads	Des
0.0738	7.1838	7.7314	0.0626	6.0332	6.4168	0.0572	5.7399	6.2997
0.3238	10.2868	12.0996	0.3159	9.6447	11.4989	0.3054	9.5666	10.9173
0.4317	12.3304	14.3549	0.4230	11.8873	13.9766	0.4091	11.4220	13.4385
0.7275	16.5457	20.7042	0.7100	16.4048	20.3971	0.6904	14.9452	19.3057
0.8362	22.3118	24.8881	0.8232	21.7375	23.3980	0.8120	20.5019	22.9994
0.8980	29.2664	29.8333	0.8910	24.7593	24.7167	0.8823	23.3927	23.2588

Table 3: The equilibrium moisture content Xeq (%d.b) of irradiated and treated thyme for various water activity and temperature

Isotherm at 30 °C					Isotherm at 40 °C					Isotherm at 50 °C				
a_w	X_{eq} of Irradiated		X_{eq} of Treated		a_w	X_{eq} of Irradiated		X_{eq} of Treated		a_w	X_{eq} of Irradiated		X_{eq} of Treated	
	Ads	Des	Ads	Des		Ads	Des	Ads	Des		Ads	Des	Ads	Des
0.07	7.42	9.64	7.46	10.43	0.06	7.37	7.76	6.23	10.18	0.06	7.33	7.54	3.14	8.08
0.32	10.09	10.65	9.46	10.94	0.32	9.07	9.87	9.19	10.72	0.31	8.63	9.12	4.75	8.78
0.43	11.09	14.29	12.19	14.23	0.42	11.08	11.68	11.96	13.97	0.41	9.49	10.43	5.41	12.75
0.73	14.94	16.32	15.60	17.18	0.71	13.48	15.23	14.31	15.25	0.69	13.44	14.53	9.48	14.47
0.84	19.96	26.77	20.88	22.45	0.82	19.56	24.88	20.32	21.17	0.81	19.02	23.92	18.73	20.02
0.89	30.80	37.58	25.82	26.42	0.89	28.25	34.85	27.29	25.43	0.88	27.80	33.27	23.92	24.64

The presentation of adsorption and desorption experimental data in the same plot show that the plant does not have the same hygroscopic equilibrium behaviour (Fig. 2).

The phenomenon of desorption-adsorption is irreversible. For the same constant relative moisture, the water content of desorption is higher than that of adsorption. Indeed, there exists a phenomenon of hysteresis. This characteristic is visible for *Thymus satureioides* plants. Several assumptions have been made to explain the hysteresis. One of it is an analogy of the plant with a sponge. When the pores do not contain any more water, adsorption does not make it possible to recover the totality of initial water since this one imprisons air in the pores. Another assumption states that hysteresis in the sorption isotherm is a consequence of variation in the fraction of bound water present in the adsorption and desorption processes. The bound fraction being always larger on desorption than on adsorption [34].

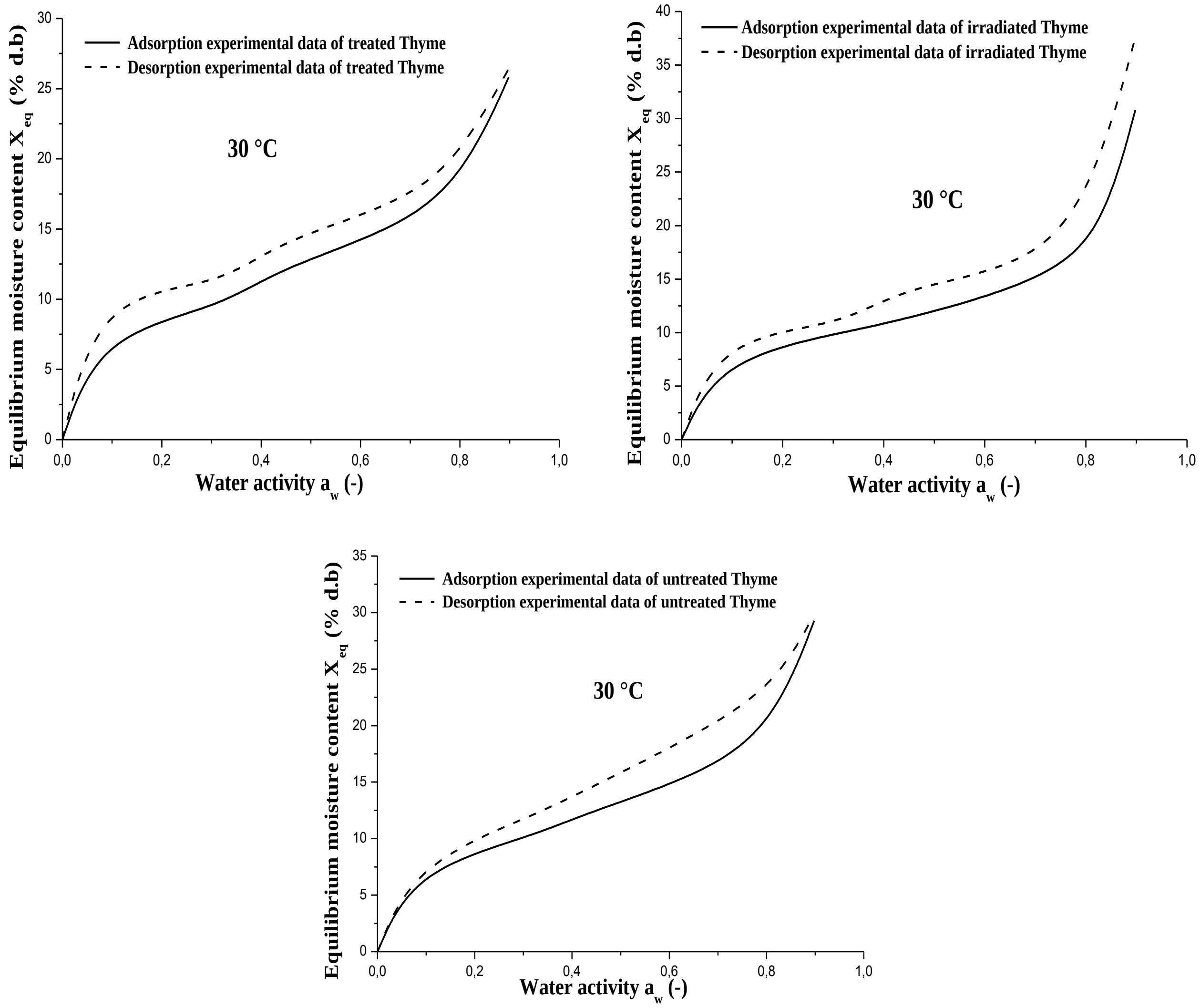

Figure 2: Sorption hysteresis phenomenon of untreated, irradiated and treated *Thymus satureioides*

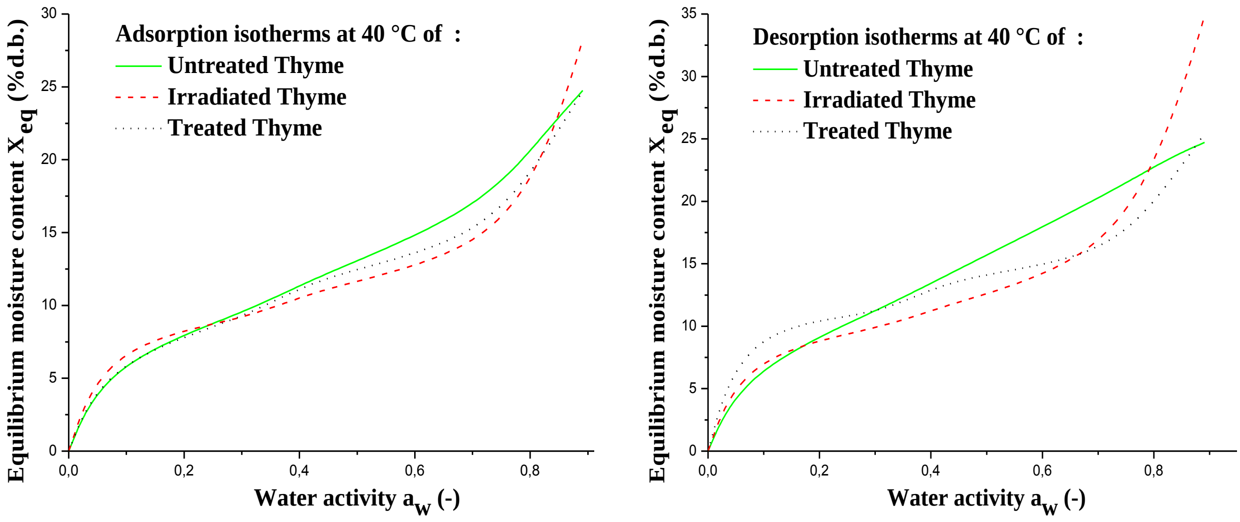

Figure 3: Sorption isotherms of untreated, irradiated and treated *Thymus satureioides* at 40°C

For a constant water activity at 40°C (Fig. 3), the adsorption equilibrium moisture contents of the three plants are almost similar. In the same way, for a constant water activity at 50°C, the adsorption equilibrium moisture contents of the three plants are almost similar at a water activity lower than 0.7. However, for desorption at 40 and 50°C, we notice the presence of a majority phase of water activity a_w ranging between [0.2 -0.7]. Accordingly, the equilibrium moisture content of untreated thyme is higher than the treated thyme, which has equilibrium moisture content higher than the irradiated thyme.

It can be seen that the three samples have the same hygroscopic behaviour. However, the curves show different sorption activities function of the applied preservation technology.

For this study, we can allocate the difference of sorption to the preservation process. The preserved thyme is influenced by drying temperature and the pretreatment by citric acid. Addition of citric acid to preserved thyme led to the modification of thyme desorption and adsorption. Due to the mechanical mixtures of plant macromolecules and simple solutes "citric acid" outline environments which undergo many physical and chemical changes accompanying water sorption. In addition, it is likely that the adsorption of citric acid on the macromolecules of plant occurs via hydrogen bonding, reducing hence the plant electrical surface charge, and making it less hydrophilic, as compared to the witness or to the irradiated sample. On the second hand, the decrease of equilibrium moisture content is due to the use of 80 °C for the conservation of treated thyme. Because of drying, some water molecules are activated to energy levels that allow them to break away from their sorption sites [35],

The difference of sorption of irradiated thyme can be explained by the influence of gamma irradiation on the cellular and subcellular structures [36]. Moreover, the gamma irradiation causes an effect on bio-chemical and physical-chemical parameters ether at lower doses [18-40]. Furthermore, the difference of sorption observed independence of the water activity range, for the three categories of plants, is in accordance with the assumption that the mixture components adsorb water independently of each other at low water activities where monolayer adsorption prevails [38]. Otherwise, the first part of the isotherm "a_w [0-0.2]" is the monolayer where water molecules are strongly fixed by several hydrogen routes with high energy on primary adsorption sites (polar groups). These water molecules characterized by a high heat of sorption, have virtually zero mobility and can participate in any interaction or reaction. These water molecules are considered as an integral part of the solid phase [39]. Only some studies related conservation process to sorption isotherms. Karel and Nickerson (1964) studies juice powder produced by freeze drying [40]. Udomkun et al. (2015) illustrated the influence of osmotic pretreatment and drying on sorption behaviour of papayas [28]. Besides, the conservation by instant controlled pressure drop (DIC) and / or freezing modified the sorption isotherms of apple [41]. However, Edrisi Sormoli and Langrish (2015) illustrated the effect of spray-drying on moisture sorption of orange juice powder [42].

3.2 Modelling of sorption experimental data:

Several models have been used for the correlation of the moisture content with the equilibrium relative humidity of the air surrounding. The results of nonlinear regression analysis of fitting the sorption equations to the experimental data are shown in figure 4. The obtained statistical information is summarized in Table 4. The models were compared for their highest correlation coefficient (r) and lowest mean relative error (MRE). The analysis of the six statistical models shows that the Enderby model, based on physical considerations by 4 parameters is the most suitable to describe the sorption isotherms of untreated and

irradiated thyme in the range of water activity ranging from 0.05-0.90. Beside, the Peleg model is the most appropriate model to illustrate desorption and adsorption isotherms for preserved thyme.

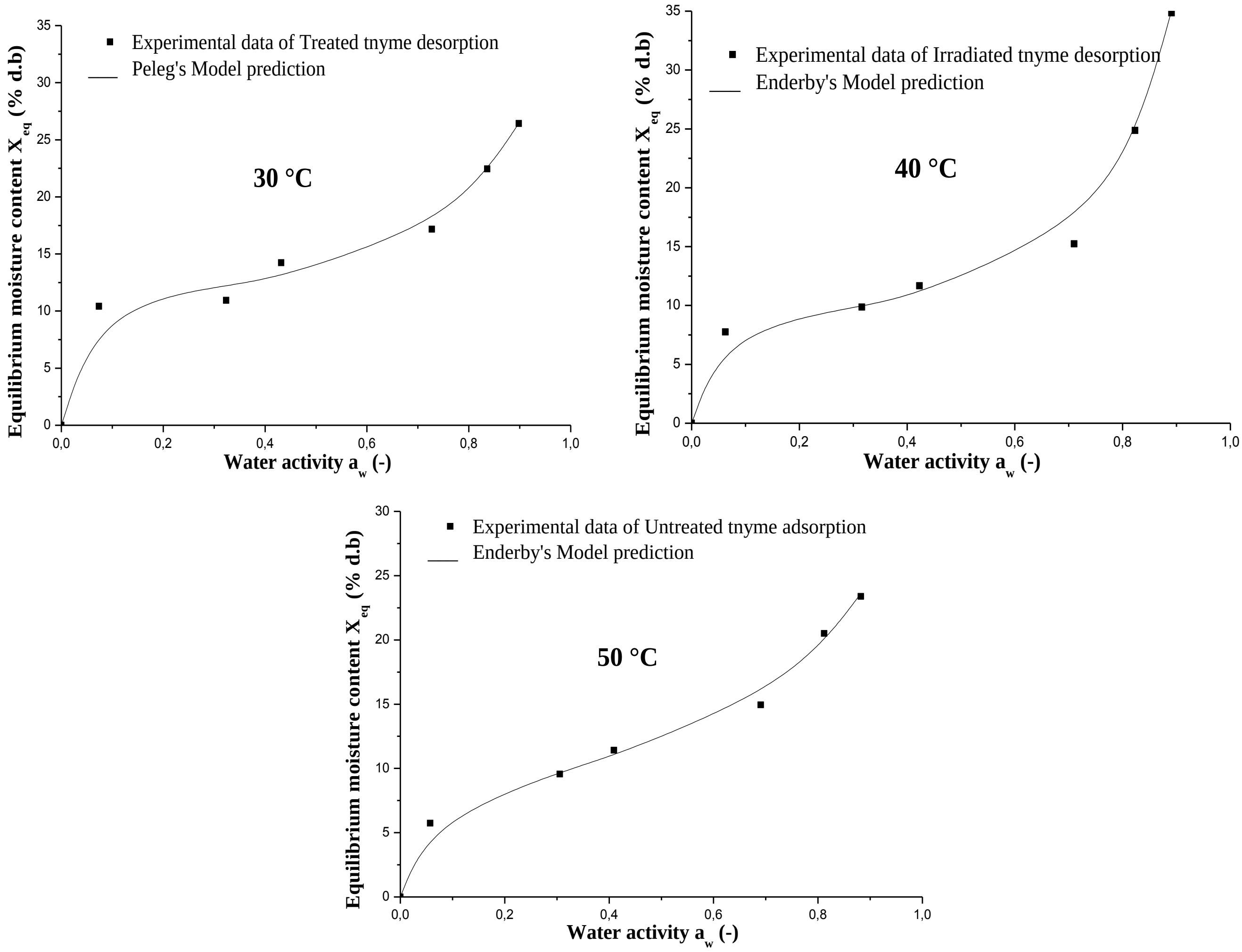

Figure 4: Sorption isotherms of untreated, irradiated and untreated thyme described by Enderby and Peleg models.

Table 4: Parameter estimation, r and MRE of equations fitted to the sorption isotherms of Thyme

Model	Untreated Thyme				Irradiated Thyme				Treated Thyme			
	Adsorption		Desorption		Adsorption		Desorption		Adsorption		Desorption	
	r	MRE	r	MRE	r	MRE	r	MRE	r	MRE	r	MRE
Modified Halsey	0.994	4.888	0.976	9.328	0.979	11.147	0.982	9.328	0.993	6.059	0.982	6.059
Modified Henderson	0.977	9.853	0.993	4.866	0.917	20.520	0.925	4.866	0.967	17.149	0.924	17.149
Modified Oswin	0.993	5.152	0.991	5.387	0.957	15.326	0.961	5.387	0.986	11.248	0.961	11.248
LESPAM	0.991	6.953	0.997	3.712	0.990	7.539	0.995	3.712	0.989	8.313	0.985	8.313
Enderby	**0.998**	**2.405**	**0.999**	**2.380**	**0.998**	**2.926**	**0.996**	**2.380**	0.994	5.010	0.988	5.010
Peleg	0.998	2.437	0.999	2.411	0.996	3.621	0.998	2.411	**0.996**	**4.314**	**0.988**	**4.314**

The experimental data of adsorption and desorption curves of untreated and preserved thyme were fitted to six sorption models. The results of nonlinear regression analysis of fitting the sorption equations to the experimental data are used to terminate the residual error between the predicted and measured EMC (Fig. 5).

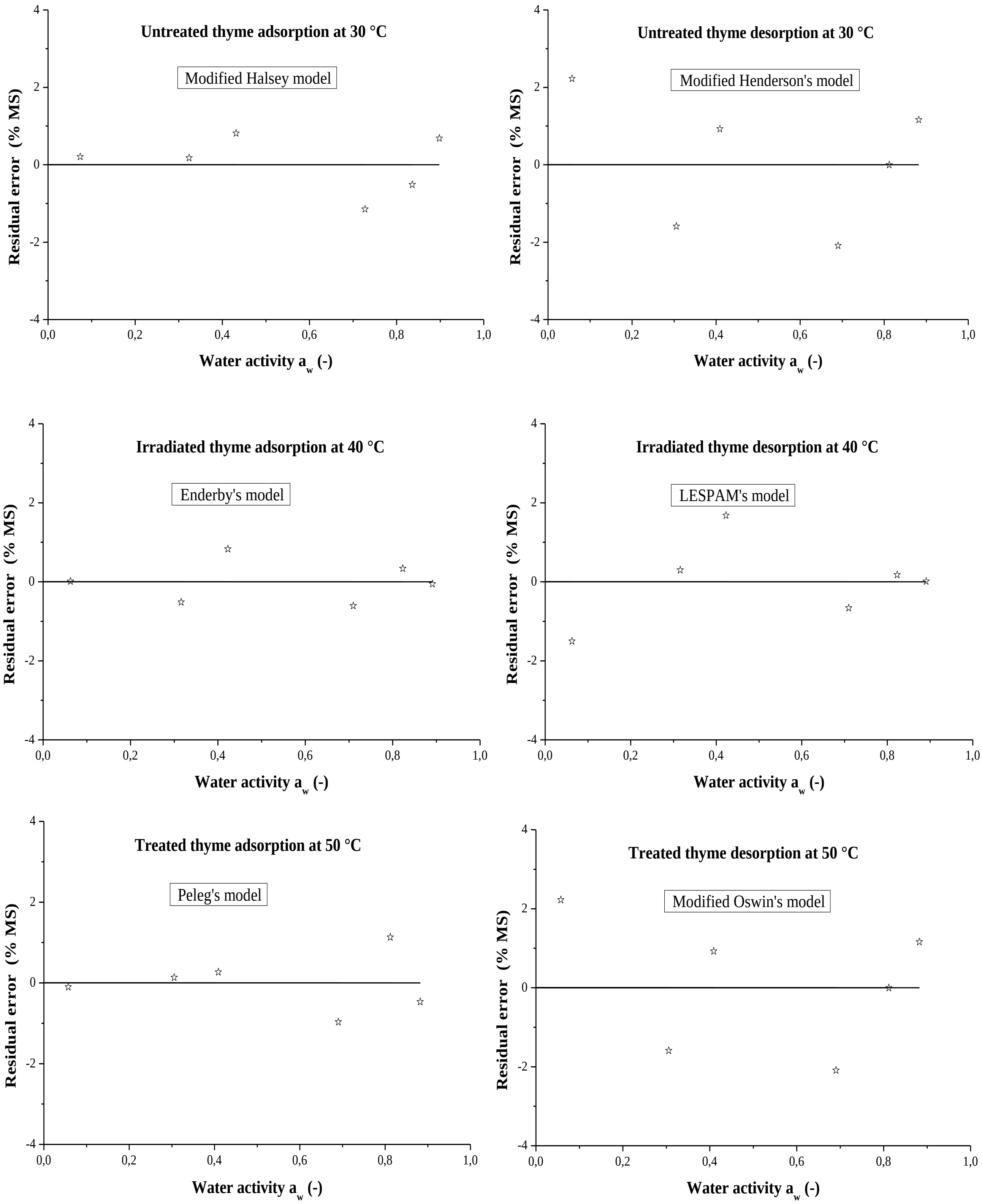

Figure 5: Residual error between predicted and measured EMC for three samples at different temperature

3.3 Determination of the optimum conditions for storage

The conservation and storage of plants require the management of the relative humidity surrounding the product. Hence, the study of sorption isotherms enables us to recognize the optimal relative equilibrium moisture for the conservation of a product as well as the equilibrium water content to reach at the end of drying. Also, it affords users with accurate information on how to handle a product during its shelf life [43].

Table 4: Optimal water activity for conservation of untreated, irradiated and treated thyme

Samples	Polynomial equation	$a_{w(op)}$
Untreated	$X_{eq} = 3.09 + 46.32\, a_w - 86.50\, a_w^2 + 71.37\, a_w^3$	0.37
Irradiated	$X_{eq} = 2.86 + 77.24\, a_w - 224.56\, a_w^2 + 195.33\, a_w^3$	0.38
Treated	$X_{eq} = 3.76 + 49.78\, a_w - 125.51\, a_w^2 + 108.67\, a_w^3$	0.39

For this purpose, the optimal water activities for conservation $a_{w(op)}$ were determined. The total experimental points are assembled on the same graph. Then, the sorption isotherm was fitted by a polynomial equation of the third degree. A polynomial decomposition of the equilibrium moisture content Xeq according to the water activity was carried out and the central part, the best area of product stability, was identified. This makes it possible to calculate the value for which the second derivative of Xeq is cancelled "inflection point" and consequently optimal relative humidity for conservation (Table 4).

The optimal water activities are ranged in 0.3-0.4. The witness thyme presents less optimal water activity (0.37) than irradiated one (0.38). The treated thyme provides the highest value of optimal water activity (0.39). The values of the optimal water activity conservation are in agreement with other product results [43].

4. Conclusion

It's now widely accepted that the sorption data provide valuable information for industrial drying in order to preserve and store aromatic plants. The purpose of our study is to identify the hygroscopic characteristics of natural thyme "traditional drying" in comparison with the industrial preserved thyme by two preservation methods "gamma irradiation and thermal-biochemical treatment using renewable energy". The experimental results show that the equilibrium moisture content increases with decreasing temperature at constant water activity. Furthermore, Enderby's moel proved to be satisfactory for the prediction of both desorption and adsorption isotherms obtained for untreated and irradiated thyme in the range of relative humidity 5 to 90%. However, treated thyme was best presented by Peleg's model for desorption and adsorption. It can be concluded that the *Thymus satureioides* plant has the same hygroscopic behaviour of the most AMP. However, the methods applied to the plant conservations change its sorption behaviour and the optimal water activity of storage. Finally, our combined treatment using solar energy is promising.

Acknowledgements

A thank to the Ministry of Higher Education, Scientific Research and Professional Training of Morocco, Priority Research program (PPR-B-Mahrouz-FS-UCA- Marrakesh) and the Regional Centre for Agricultural Research CRRA, Ionization Station Boukhalef Tanger.

Nomenclatures

A, B, C and D model coefficients ads adsorption

Ads adsorption

a_w water activity

$a_{w(op)}$ water activity optimal

d.b. Dry weight basis

Des desorption

EMC=X_{eq} Equilibrium moisture content (Kg water/(kg d.b.))

LESPAM Laboratory of Solar Energy and Medicinal Plants

M_d mass of dry matter (kg)

M_w mass of wet matter (kg)

MRE mean relative error (%)

N number of data points

Pred predicted

r correlation coefficient

Rh equilibrium relative air humidity (%)

T Absolute temperature (K)

θ Temperature (°C)

$Xeq_{i,exp}$ experiment EMC (% d.b)

$X\ eq_{i,pred}$ predicted EMC (% d.b)

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