

Moisture sorption isotherms and thermodynamic properties of Oak wood (*Quercus robur* and *Quercus canariensis*): optimization of the processing parameters

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Abstract The aim of this paper was to determine the moisture desorption isotherms and essential thermodynamic properties of two Oak wood varieties. Desorption isotherms were measured using a static gravimetric method at 50, 60, 70 and 80 °C within the range of 5–90 % relative humidity. The equilibrium moisture content decreased with increasing temperature and decreased with decreasing relative humidity at a constant temperature. The ‘Thermodynamic’ sorption equation was found to be the best for describing the experimental moisture sorption isotherms of woods within the range of temperature and water activity investigated. The Fiber saturation point, deduced from the ‘Thermodynamic’ model parameters, depends on the temperature and varying from 22.6 to 54.4 (% kg water/kg dry matter). Isothermic heat of desorption and differential entropy were calculated by applying Clausius–Clapeyron equation to the desorption data fitted by the ‘Thermodynamic’ model. The isothermic heat of desorption and the differential entropy decreased with increasing moisture content according

to an exponential law equation and varying from 2.03 to 31.14 kJ/mol and from 73.98 to 4.34 J/(mol K), respectively. The linear relationship between differential enthalpy and entropy satisfied the enthalpy–entropy compensation theory. The sign of Gibbs free energy was found to be positive (+283 J/mol) and (+97 J/mol) for *Quercus robur* and *Quercus canariensis*, respectively. The isokinetic temperature was found to be greater than the harmonic temperature. Based on the enthalpy–entropy compensation theory, it could be concluded that the moisture desorption isotherm of Oak wood is a non-spontaneous and enthalpy-controlled process.

List of symbols

A, B and C	Model parameters
a_w	Water activity (–)
C_g	Constant related to the heat of sorption
cst	Constant
DM	Dry matter
K	Constant related to multilayer properties
L_v	Latent heat of vaporization (J/mol)
M_d	Dry mass (kg)
M_{eq}	Equilibrium mass (kg)
n	Total number of isotherms
q_0	Isothermic heat of sorption of the monolayer (kJ/mol)
q_{st}	Total isothermic heat of desorption (J/mol)
$Q_{st,n}$	Desorption isothermic heat (J/mol)
R	Perfect gas constant [8.315 J/(mol K)]
RH	Relative humidity (%)
T	Temperature (°C)
T_{hm}	Harmonic mean temperature (K)
T_β	Isokinetic temperature (K)
X_0	Water content of the monolayer [kg water/(kg DM)]

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$X_{eq\text{ cal},i}$	Equilibrium moisture content calculated
X_{eq}	Equilibrium moisture contents [kg water/(kg DM)]
X_{eqi}	Experimental value of equilibrium moisture content
X_{fsp}	Fiber saturation point [% kg water/(kg DM)]
n_{expdata}	Experimental point
n_{param}	Parameter number of the particular model
\bar{X}_{eq}	Arithmetic average value of the experimental equilibrium moisture content
X_{eqm}	Monolayer moisture content [kg/(kg DM)]
ΔS	Desorption entropy (J/mol K)
Φ	Thermodynamic parameter

1 Introduction

Wood is an important biodegradable and renewable material used all over the world as an energy source, building and packing material, furniture and tools, decoration purposes and artistic medium due to its high strength-to-weight ratio, unique porous structure, and aesthetic characteristics [1, 2]. Freshly cut wood contains large quantity of water in cell walls and liquid–vapour mixture or vapour in cell lumens [3]. Drying of wood stabilizes the dimensions and shape of wooden components and protect them from biological attack by reducing the water activity [4, 5]. The International Plant Protection Convention (IPPC) specifies heating process under controlled climate to sanitize wood packing material, until a well-known moisture content, in order to prevent the circulation of the forest pests around the world [6, 7]. Controlled, handling and storage of wood material minimize moisture content changes after drying when physical, chemical and microbiological change become undesirable [8]. Wood is a hygroscopic product, which tends towards equilibrium moisture content with its environment [4, 9–12]. Therefore, the heat supplied to wood accelerates the water migration and affects the dynamic equilibrium between the vapor and adsorbed phases during adsorption and desorption processes [13].

The better knowledge moisture desorption isotherms and essential thermodynamic properties (net isosteric heat, differential enthalpy, differential entropy) of wood under controlled climate is necessary for the prediction of the drying time, reduction of the heating energy coast and designing of drying apparatus [14, 15] and for calculating moisture changes that may occur during drying, storage and use under various climatic conditions [16].

Numerous reports in the literature demonstrated that the sorption isotherm was the most solution to determine the equilibrium moisture content of wood and various biological products under controlled operating conditions (temperature and relative humidity) by using

static-gravimetric method [10–12, 17–23]. The sorption isotherm was the curve representing the equilibrium moisture content of wood versus water activity at a constant temperature [10, 12]. According to [16], the concept of water activity quantifies the presence of water in the product and was commonly defined as the ratio of the water vapor pressure exerted by the product to the vapour pressure of pure water, at the same temperature. Rawat and Khali [24] demonstrate that the changes in free energy, enthalpy, and entropy during water sorption in Klinky Pine wood have a strong dependence on moisture content, and have a weak dependence on temperature. Themelin [17] demonstrates that the increase of temperature and the decrease of relative humidity decrease the equilibrium moisture content of tropical woods of angelica (*dicorynia guianensis*), iroko (*milicia excels*) and Eucalyptus (*Eucalyptus grandis*) and that the degradation of hemicelluloses in torrefied wood reduces the sorption sites of water vapour than in non torrefied wood. Nkolo Meze'e et al. [20] demonstrated that the thermodynamic properties (Gibbs energy, enthalpy and entropy) of tropical wood (*Bubinga*) have a great dependence to moisture content and no significant variation with temperature. Ouertani et al. [11] demonstrate that the increase of temperature decreases the moisture content of Jack pine (*pinus bankisiana*) and palm wood and that the corresponding thermodynamic properties depend on temperature and moisture content and that the moisture desorption reactions is a non-spontaneous and enthalpy controlled process.

Mathematical models that describe the experimental sorption data aid to determine the thermodynamic interactions of wood-water system, the useful equilibrium moisture content at the end of drying operation and were important tools in modeling approach of heat and mass transfer operation [5, 25]. Thus, thermodynamic properties are readily deduced from data fitted by the sorption isotherm equations. There are more than 77 mathematical equations with two or more parameters, having a physical meaning, were proposed in the literature to fit the sorption isotherms in a wide range of temperature and relative humidity. The most used equation for lingo-cellulosic products, in particular wood, were Thermodynamic model [26], Dent model [27], GAB and Henderson models [28].

Thermodynamic properties such as isosteric heat of desorption, differential enthalpy, Gibbs free energy and differential entropy may be calculated by applying Clausius–Clapeyron equation to fitted sorption data [22, 29]. The isosteric heat of desorption, measure the energy required to break the intermolecular forces between the water molecules and the surface of adsorbent. The better knowledge of the isosteric heat of desorption has more accurate information when designing drying apparatuses [12]. Enthalpy variation measures the energy changes that occur upon mixing

of water molecules with wood during sorption and may be associated with binding forces of the water-wood system [20]. The free energy variation may be indicative of the sorbent's affinity for water based on its sign and provide a criterion whether water sorption is a spontaneous process or not [30]. Spontaneous reaction occurs naturally without continuous external source energy. However, non-spontaneous reaction takes place as a result of continuous external intervention [31]. The concept of enthalpy controlled desorption process means that the main driving force is the release of heat [30]. The entropy changes during sorption process may be associated with the spatial arrangements occurring in the water–wood system in a defined state [32]. The enthalpy–entropy compensation theory has been widely investigated in different physical and chemical processes, in particular, in water desorption reactions under controlled temperature and relative humidity [11, 33, 34]. The theory specifies that in order to minimize free energy variation due to these phenomena, compensation arises from the nature of the interaction between the solute and solvent causing the reaction and that the relationship between the enthalpy and entropy for a specific reaction is linear [20].

Oak wood (*Quercus robur* and *Quercus canariensis*) is an important crop grown in the North-East of France and in the Northern part of Tunisia, respectively. This wood crop plays a very important role in the socioeconomic and ecological levels of France and Tunisia. It used in various specific industries, such as plywood, decorative veneers, furniture, crafts and packaging. However, moisture desorption isotherms and thermodynamic properties of *Quercus robur* and *Quercus canariensis* wood under controlled climate stills remains unclear. Therefore, the objective of this work was (1) to determine the desorption isotherms of two Oak wood varieties, (2) to determine the suitable model fitting the desorption isotherms, (3) to determine the dependence of thermodynamic properties with temperature and moisture content and to check the existence of the enthalpy–entropy compensation theory (4) and to verify if the desorption isotherm reaction in this wood materials is spontaneous or non-spontaneous and enthalpy-driven or entropy-controlled process.

2 Materials and methods

2.1 Wood sample preparation

Oak wood samples of *Quercus robur* and *Quercus canariensis* are freshly harvested from the North-East of France (Vosges) and from the Northern part of Tunisia, respectively [35, 36]. To focus the attention on the influence of temperature and relative humidity on the water desorption

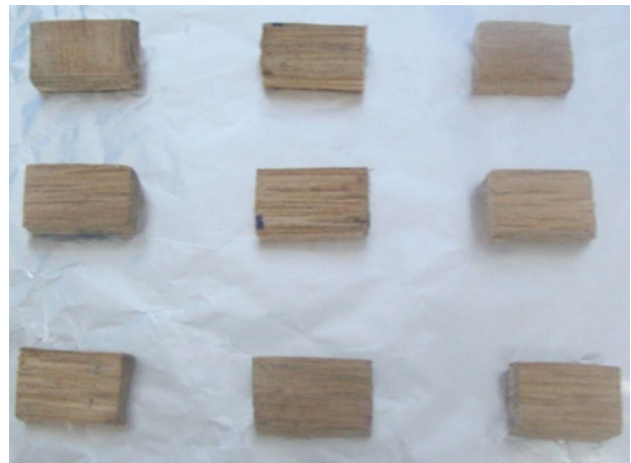


Fig. 1 Wood samples used for desorption isotherms experiments

Table 1 Selected salt used for preparing saturated salt solutions and their corresponding water activities

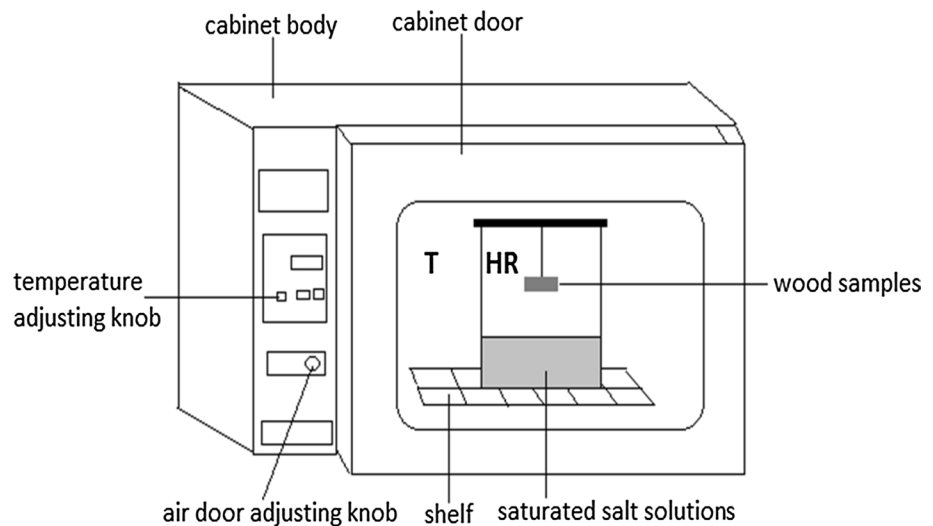
Salt	Water activity (a_w)	r	Authors
LiCl	$\ln(a_w) = \frac{500.95}{T} - 3.85$	0.976	[11, 16, 37]
MgCl ₂	$\ln(a_w) = \frac{303.35}{T} - 2.13$	0.995	
K ₂ CO ₃	$\ln(a_w) = \frac{145}{T} - 1.3$	0.967	
NaCl	$\ln(a_w) = \frac{228.92}{T} - 1.04$	0.961	
KCl	$\ln(a_w) = \frac{367.58}{T} - 1.39$	0.967	
KOH, NaBr	a_w values at 50, 60, 70 and 80 °C are available		[11, 39]
BaCl ₂	in the following references		[11, 38]

process, wood samples were sawn from a one green tree in order to limit the wood variability [18]. The dimensions of the test pieces were 15 mm long, 10 mm wide and 5 mm thick in longitudinal (L), radial (R) and Tangential (T) directions, respectively. The size of the wood samples was small enough to obtain uniform equilibrium moisture content (Fig. 1).

2.2 Experimental procedure: moisture desorption isotherms and data analysis

The equilibrium moisture content of wood at 50, 60, 70 and 80 °C was measured using the static gravimetric method. Standard saturated salt solutions (KOH, LiCl, MgCl₂, K₂CO₃, NaBr, NaCl, KCl and BaCl₂) were used to control the relative humidity in a closed space. In this method, diffusion is the only mass transfer mechanism inside the product. Values of relative humidity of saturated salt solutions are obtained from the works of [11, 16, 37–39] and presented in Table 1. These solutions are prepared by dissolving the

Fig. 2 Experimental apparatus for determination of sorption isotherms (DHG-9053A)



necessary quantity of salt in distilled water up to super-saturation, at a given temperature, covering a water activity range of 0.058–0.891. These solutions were placed inside hermetic jars with insulated lids. Wood samples are suspended to jars lids without touching the solutions. Finally, hermetic jars were placed inside heating oven (DHG-9053A) with a temperature range of +10 to 250 °C and temperature accuracy of ± 1 °C (Fig. 2). The thermodynamic equilibrium of wood was considered when the mass of the samples showed a difference less than 1 mg after three consecutive measurements at intervals of 24 h [11, 40–42]. Wood samples were weighed on a balance (Mettler Toledo, Mississauga, Canada) at 0.001 g precision [43]. When equilibrium was reached and based on standard test method to obtain dry matter content, the wood samples were dried using oven method at 103 ± 2 °C for 24 h [44]. The equilibrium moisture content is given by the following Eq. (1):

$$X_{\text{eq}} = \frac{M_{\text{eq}} - M_{\text{d}}}{M_{\text{d}}} = \frac{M_{\text{water}}}{M_{\text{d}}} \text{ (kg water/kg DM)} \quad (1)$$

2.3 Moisture desorption isotherms modeling

The sorption isotherm equations used in the present study for fitting the experimental data are presented in Table 2. The four selected sorption models are mostly used to describe the moisture sorption of wood [11, 17, 18, 27]. To select the most appropriate equation that adequately describes the sorption data of two varieties of Oak wood, models parameters were identified by non-linear least square regression analysis, using CurvExpert 1.3 software with two statistical criteria, namely the standard error (s) and the correlation coefficient (r), defined in the “Appendix” [45].

3 Thermodynamic properties of desorption

3.1 Net isosteric heat of desorption-differential enthalpy

The isosteric heat of desorption (differential enthalpy) was determined by applying the Clausius–Clapeyron equation (Eq. 2), that relates water activity and temperature at a constant moisture content to equilibrium data obtained from the best fitting desorption equation [11, 21, 38].

$$\frac{d \ln(a_w)}{d(1/T)|_X} = \frac{Q_{\text{st},n}}{R} = \frac{q_{\text{st}} - L_v}{R} \quad (2)$$

Assuming that the isosteric heat of desorption is invariant with temperature [46], the integration of equation (Eq. 2) gives the following equation (Eq. 3):

$$\ln(a_w) = - \left[\frac{Q_{\text{st},n}}{R} \right] \left[\frac{1}{T} \right] + \text{cte} \quad (3)$$

The net isosteric heat of desorption was determined from the slope of the curve $\ln(a_w)$ versus $1/T$ (K^{-1}). This approach was repeated for several equilibrium moisture content values obtained from the best fitting equation. This procedure determines the evolution of isosteric heat of desorption with equilibrium moisture content. Therefore, this procedure was recommended for sorption isotherms measured at more than two temperatures [11, 21, 42].

To describe the relationship between the isosteric heat of desorption and the equilibrium moisture content, Tsami [21] proposed the following empirical exponential expression (Eq. 4):

$$Q_{\text{st},n} = q_0 \exp \left(- \frac{X_{\text{eq}}}{X_0} \right) \quad (4)$$

3.2 Differential entropy

Differential entropy of desorption (ΔS) changes may be associated with the binding or repulsive forces of water molecules in the product and is proportional to the number of available sorption sites at a specific energy level. It defines the degree of order or randomness existing in the water-sorbent system and can help interpreting processes such as dissolution, crystallization and swelling [47, 48]. Gibbs free energy (ΔG) sign may be indicative of the affinity of sorbents for water, and provides a criterion to determine whether water sorption occurs as a spontaneous ($\Delta G < 0$) or non-spontaneous ($\Delta G > 0$) process [49]. The differential entropy of desorption is calculated using the equation of Gibbs–Helmholtz [50]:

$$\Delta S = \frac{Q_{st,n} - \Delta G}{T} \quad (5)$$

where ΔG is the Gibbs free energy, calculated from Eq. (6):

$$\Delta G = -RT \ln(a_w) \quad (6)$$

The relationship between the differential enthalpy ($Q_{st,n}$) and entropy of desorption (ΔS) is given by Eq. (7), which was deduced from Eqs. (5) and (6):

$$-\ln(a_w) = \frac{Q_{st,n}}{RT} - \frac{\Delta S}{R} \quad (7)$$

For given moisture content, differential desorption entropy (ΔS) is determined from the intercept of the line of $\ln(a_w)$ versus $1/T$ (K)⁻¹ [34].

3.3 Enthalpy–entropy compensation theory

Enthalpy–entropy compensation theory is used to evaluate physical and chemical phenomena such as moisture sorption reactions. Sharp [51] explained that the entropy–enthalpy compensation term was applied in literature and different usages are discussed. However, the most precise and interesting case was a linear relationship between enthalpy and entropy for some series of perturbations or changes in experimental variables. In a true equilibrium, assumed in the Clausius–Clapeyron equation, entropy is maximized, hence the differential entropy is zero and the differential Gibbs free energy is equal to the isosteric heat of adsorption [52]. For a specific thermodynamic reaction, the enthalpy–entropy compensation theory proposes a linear relationship between the differential enthalpy ($Q_{st,n}$) and entropy of desorption (ΔS) [Eq. (8)] [30, 53]:

$$Q_{st,n} = T_\beta \Delta S + \Delta G_\beta \quad (8)$$

In a true equilibrium, assumed in the Clausius–Clapeyron equation, entropy is maximized, hence the differential entropy is zero and the differential Gibbs free energy is equal to the isosteric heat of adsorption [52].

The isokinetic temperature T_β represents the temperature at which all reactions in the series proceed at the same rate. ΔG_β represent the Gibbs free energy at the specific temperature T_β . The sign of ΔG_β indicates whether water desorption reaction is a spontaneous (negative) or a non-spontaneous process (positive).

To confirm the enthalpy–entropy compensation theory a statistical analysis test was proposed by Krug et al. [54]. The isokinetic temperature should be compared with the harmonic mean temperature (T_{hm}) [Eq. (9)]. The enthalpy–entropy compensation theory applies if $T_\beta \neq T_{hm}$. Moreover, if $T_\beta > T_{hm}$ the process is enthalpy driven, while if $T_\beta < T_{hm}$, the process is entropy-controlled [30, 48, 53].

$$T_{hm} = \frac{n}{\sum_{i=1}^n (1/T(K))} \quad (9)$$

Based on [34, 55] works, parameter (ΔG_β) can be neglected for its small contribution in the enthalpy change. The enthalpy–entropy compensation theory was further used to evaluate the impact of temperature on the sorption behaviour by rearranging Eqs. (7) and (8) to obtain the following Eq. (10).

$$-\ln(a_w) = \frac{Q_{st,n}}{R} \left(\frac{1}{T} - \frac{1}{T_\beta} \right) \quad (10)$$

The influence of temperature on moisture equilibrium properties can be considered by re-expressing Eq. (10) in the following form (Eq. 11):

$$\Psi_T \ln(a_w) = K \Phi(W) \quad (11)$$

Where $\Psi_T = ((1/T) - (1/T_\beta))^{-1}$ is the temperature correction factor in the desorption isotherms and $\Phi(X)$ is an empirical function of the equilibrium moisture content. The equilibrium data can be represented by plotting $\ln(\Psi_T \ln(a_w))$ as a function of the equilibrium moisture content.

Aguerre et al. [34] asserted that if this correction factor is adequate, the equilibrium data of a given product, at different temperatures, present the same mathematical form when plotted in accordance with Eq. (10). The dependence of $\Psi_T \ln(a_w)$ with moisture content (X) follows an exponential law (Eq. 12):

$$\Psi_T \ln(a_w) = K_1 K_2^X \quad (12)$$

After plotting $\ln(\Psi_T \ln(a_w))$ versus equilibrium moisture content, the constants K_1 and K_2 were calculated by linear regression.

4 Results and discussion

4.1 Moisture desorption isotherms

The experimental moisture desorption isotherms for the Oak wood of *Quercus canariensis* and *Quercus robur* at 50, 60, 70 and 80 °C are shown in Figs. 3 and 4, respectively. Curves analysis demonstrates that the equilibrium moisture content increases with the water activity at constant temperature. Moisture desorption isotherms behaviour of two wood varieties showed the typical sigmoid shape of type II, according to the BET classification [56]. This shape is typical of tropical wood [10, 57], Jack pine and palm wood [11], Maritime pine wood [18] and Japanese cypress wood (hinoki: *Chamaecyparis obtusa*) [58]. The curves shape informs on the nature of the water-wood sorption process. The Type II isotherms correspond to process which initially follows 'Type I' form but then it shows a buildup of

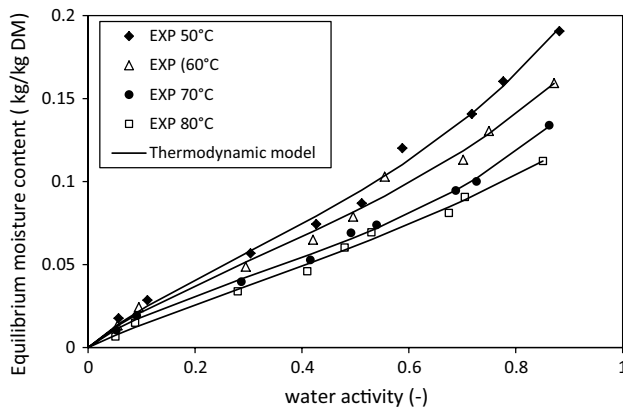


Fig. 3 Desorption isotherms for Tunisian Oak wood (*Quercus canariensis*) at various temperatures

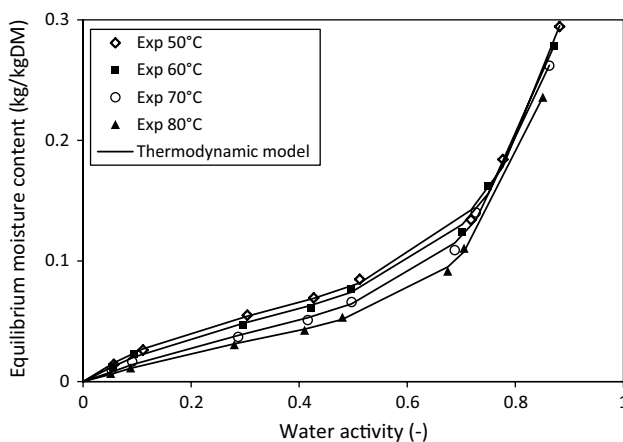


Fig. 4 Desorption isotherms for French Oak wood (*Quercus robur*) at various temperatures

a monolayer. After the monolayer region, the increase of vapour pressure results in a successive multilayer adsorption and a strong interaction between the molecules of adsorbate [59]. Desorption isotherms curves analysis lead to the identification of two different regions. At low and intermediate water activities, multilayer sorption region is localized. It is characterized by linear increases of moisture content with water activity. While, at high water activities, capillary condensation region is localized. It is characterized by rapid increasing of water content with water activity [60].

At constant water activity, the equilibrium moisture content of two types of wood decreases as the temperature increases. This behavior explains that the increase of temperature can reduce the total number of active sites for water binding as a result of physical and/or chemical changes in the product [61]. The increase of temperature promotes the migration of water molecules from the water binding sites [11]. When temperature increases the water molecules become activated due to an increase in their energy level, causing them to become less stable and to break away from the water-binding sites of the material, which consequently decreases the monolayer moisture content [62].

Comparative aspect between moisture desorption isotherms of Figs. 3 and 4 shows that *Quercus canariensis* wood is less hygroscopic at all operating conditions than *Quercus robur*. The difference in adsorption capacity, under same controlled climate, between two Oak varieties becomes from various parameters that affect the physical, anatomical structure and tissue of wood such as species, growth conditions, site and climate [9, 63, 64]. The Kelvin equation gives the relationship between the radius of the cell and the equilibrium relative vapor pressure [65]. According to the distribution of the cells size of the product, the capillary condensation can change the part of the sorption isotherm at the high humidity. So we could assume that the samples selected for this work for *Quercus canariensis* and *Quercus robur* have a contrasted structure. On the other hand, the average density of dried wood of *Quercus robur* and *Quercus canariensis* was equal to 584.3 kg m^{-3} [66] and 708.5 kg m^{-3} (determined experimentally), respectively. However, we could assume that *Quercus robur* wood have a large cells (vessels and fibers) than *Quercus canariensis* which favorite the capacity of water adsorption under the same operating conditions (Table 3).

4.2 Isotherm predictive models

The experimental desorption data were fitted with GAB, Dent, Henderson, and Thermodynamic models. The results of non-linear regression of desorption isotherms of wood,

Table 2 Mathematical models used to feet moisture sorption isotherms

Model name	Model equation	Domain of validity
GAB	$X_{eq} = \frac{X_{eqm} C_g K a_w}{(1 - K a_w)(1 - K a_w + K C a_w)}$	$0 < RH < 0.9$
Henderson	$X_{eq} = \frac{-\ln(1 - a_w)}{B}$	Complete curve
Dent	$X_{eq} = \frac{a_w}{A a_w^2 + B a_w + C}$	$0.1 < RH < 0.95$
Thermodynamic	$X_{eq} = X_{PSF} \exp(\Phi \ln a_w \exp(a a_w))$	$0 < RH < 1$

at various temperatures, are shown in Table 3. According to our results, the ‘Thermodynamic’ model presents the lowest values of standard error (equal to 2.61×10^{-3} and 6.1×10^{-3}) and the highest value of correlation coefficients (equal to 0.994 and 0.999), respectively, and gives the best fit as shown in Figs. 3 and 4. Based on these results, Thermodynamic model was selected to describe the desorption isotherms of *Quercus robur* and *Quercus canariensis* wood. The ‘Thermodynamic’ equation is based on thermodynamic approach of heat sorption of bound water which depends on the moisture content state, the superficial layer energy state and the environment properties [26]. A plot of predicted (using Thermodynamic model) versus measured equilibrium moisture content, between 50 and 80 °C was presented in Figs. 3 and 4. According to [17] the better knowledge of the desorption data and corresponding mathematical models under controlled operating conditions can be integrated directly in drying schedules and industrial kilns supervision software.

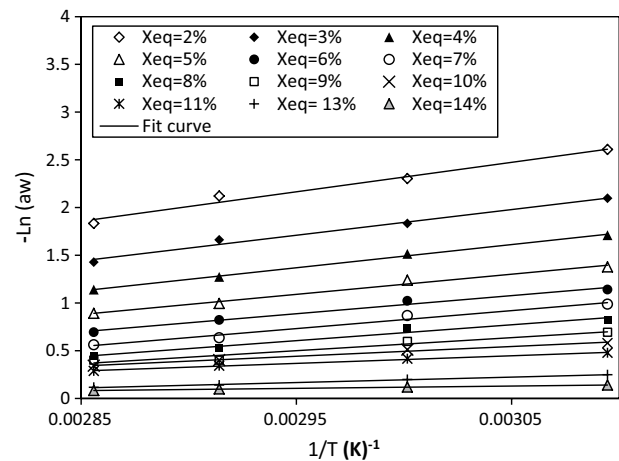
The fiber saturation point (FSP) of *Quercus robur* and *Quercus canariensis* wood varying from 54.4 to 45.1 and 34.1 to 22.6 % kg/(kg DM), respectively. Results shows that an increase in temperature decreases the FSP. This result illustrates the impact of temperature on the water adsorption–desorption capacity of Oak wood and gives information about the best fitting of desorption data. Similar results were obtained by [26] for Douglas fir, [18] for Maritime pine and [11] for Jack pine and Palm wood. Fundamentally,

the fiber saturation point is the amount of water that the composite polymers of the cell wall can hold at a particular temperature and pressure [67]. Varying bound water bellow FSP causes dimensional changes (shrinkage or swelling) of wood. Therefore, the knowledge of the FSP has many practical or industrial applications, it gives crucial information on the beginning of the shrinkage mechanism during drying and it can aid to optimize the processing parameters, to avoid the cracking, collapse, twist, etc.

4.3 Isosteric heat and entropy of desorption

The isosteric heat of desorption or differential enthalpy and entropy change were calculated from the desorption data determined throughout ‘Thermodynamic’ model. The desorption isosters of *Quercus robur* and *Quercus canariensis* wood are presented in Figs. 5 and 6.

Isosteric heat of desorption was calculated from the slope of the plot of $\ln(a_w)$ versus $1/T$ (K) $^{-1}$ (Eq. 3) for each equilibrium moisture content. Figure 7 shows that the isosteric heat of desorption ($Q_{st,n}$) of Oak wood, determined by applying Clausius–Clapeyron equation, decreases

**Fig. 5** Desorption isosteric curves of Tunisian Oak wood (*Quercus canariensis*)**Table 3** Results of fitting of the desorption isotherms of two Oak wood varieties by the thermodynamic model

Product	T (°C)	Thermodynamic model parameters	s	r
<i>Quercus canariensis</i>	50	$X_{psf} = 0.341, \Phi = 0.95, a = 0.771$	6.1×10^{-3}	0.996
	60	$X_{psf} = 0.281, \Phi = 0.9, a = 0.699$	6.11×10^{-3}	0.994
	70	$X_{psf} = 0.246, \Phi = 0.9, a = 0.922$	2.61×10^{-3}	0.998
	80	$X_{psf} = 0.226, \Phi = 0.96, a = 0.433$	3.28×10^{-3}	0.996
<i>Quercus robur</i>	50	$X_{psf} = 0.544, \Phi = 1.17, a = 1.898$	4.8×10^{-3}	0.999
	60	$X_{psf} = 0.520, \Phi = 1.23, a = 1.867$	4.3×10^{-3}	0.999
	70	$X_{psf} = 0.484, \Phi = 1.37, a = 1.815$	4.2×10^{-3}	0.999
	80	$X_{psf} = 0.451, \Phi = 1.49, a = 1.916$	2.25×10^{-3}	0.999

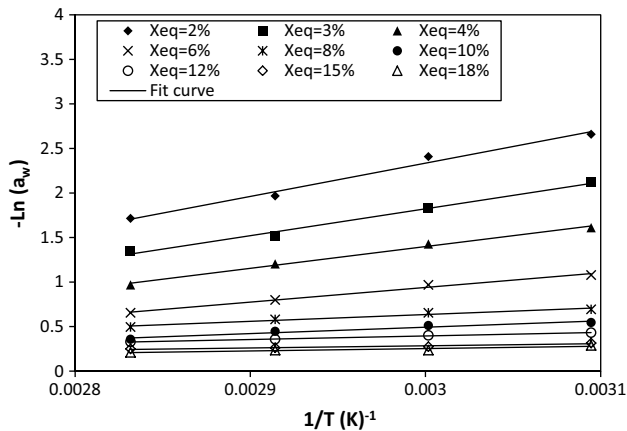


Fig. 6 Desorption isotherm curves of French Oak wood (*Quercus robur*)

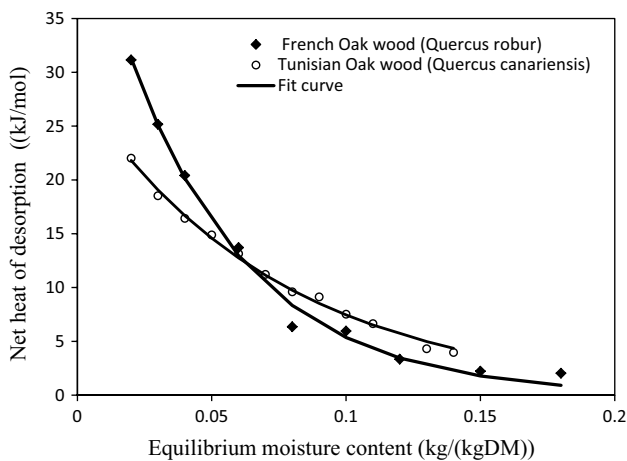


Fig. 7 Isothermic heat of desorption versus moisture content

continuously with the increase of the equilibrium moisture content. At high moisture content, isothermic heat of desorption is close to the mean value of latent heat of vaporization of pure water (L_v) [12, 21]. As seen from Fig. 7, the isothermic heat of desorption ($Q_{st,n}$) decreased with the increase of the moisture content from 31.15 to 2.03 kJ/mol and from 22.02 to 3.96 kJ/mol with a variation of equilibrium moisture content ranging from 0.02 to 0.18 kg/(kg DM) and from 0.02 to 0.14 kg/(kg DM) for *Quercus robur* and *Quercus canariensis*, respectively. The high value of isothermic heat of desorption ($Q_{st,n}$) showed at low moisture content is due to the existence of highly active polar sites on the surface on the product, which are covered with water molecules forming a mono-molecular layer [68]. According to [69] at increasing moisture content, the most active sites become occupied and sorption occurs on the less active site giving lower heats of sorption. Similar results were

reported for wood of Klinky Pine [24], Western hemlock, Douglas fir, Western red cedar, Sitka spruce and Lodgepole pine [19], tropical wood (Bubinga) [20] and Jack pine and Palm wood [11]. The high value of isothermic heat of desorption ($Q_{st,n}$) at low moisture content indicates the strong link between water molecules and the solid matrix of wood [20, 70]. This requires supplementary heat to the latent heat of vaporization of pure water to evaporate the bound water molecules in the hygroscopic region. This trend supported the fact that as moisture content increases, the forces of water molecules attraction by wood decrease [71]. The water sorption mechanism occurs initially in the most active sites giving rise to the greatest interaction energy [72]. When the moisture content increases the sorption occurs on the less active sites resulting in lower heats of sorption and a negligible influence of solid matrix of wood on adsorbed water. The variation of the isothermic heat of desorption with moisture content was fitted by an exponential function, according to the typical Tsami [21] equation, for *Quercus robur* and *Quercus canariensis* as following:

$$Q_{st,n} = 48.65 \exp\left(\frac{-X_{eq}}{0.045}\right) \quad (r = 0.996) \quad (Quercus\ robur) \quad (13)$$

$$Q_{st,n} = 28.53 \exp\left(\frac{-X_{eq}}{0.074}\right) \quad (r = 0.997) \quad (Quercus\ canariensis) \quad (14)$$

The parameters of Tsami [21] equation (q_0 ; X_0) for desorption data were (48.65 kJ/mol; 0.045 kg/kg DM) and (28.53 kJ/mol; 0.074 kg/kg DM) for *Quercus robur* and *Quercus canariensis*, respectively. The q_0 and X_0 parameters defines the necessary isothermic heat to evaporate the monolayer equilibrium water content of the product. Similar results were reported for Jack pine and Palm wood [11] and for prickly pear seeds [42]. However, power law equation has been proposed to describe the isothermic desorption heat with moisture content of olive leaves (*Olea europaea* L.) [22] and Polynomial law were also proposed for peppermint tea (*Mentha piperita*) [68].

At low moisture content (<7 %), the isothermic heat of desorption is higher in *Quercus robur* than in *Quercus canariensis*. However, at high moisture content (>7 %) it is higher in *Quercus canariensis* than in *Quercus robur*. This behaviour can be related to the variability between Oak varieties in anatomical structure, growth conditions, site and climate of the North-East of France and the Northern part of Tunisia [9].

The variation of the differential entropy of desorption of *Quercus robur* and *Quercus canariensis* versus equilibrium moisture content is shown in Fig. 8. The curve shows a continuous decrease of the value of differential entropy of desorption with the increase of equilibrium moisture content. It ranged from 73.98 to 4.345 J/(mol K) and from

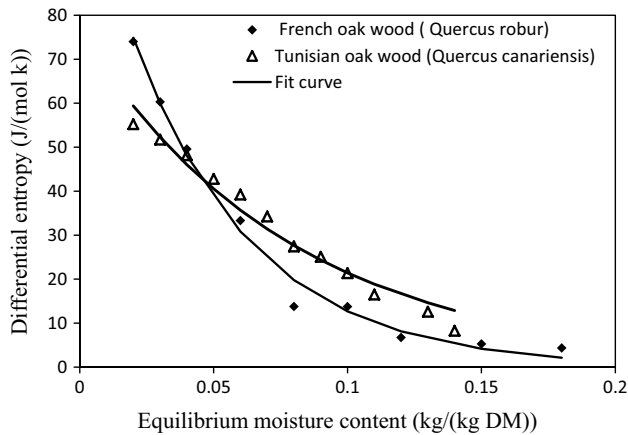


Fig. 8 Differential entropy versus moisture content

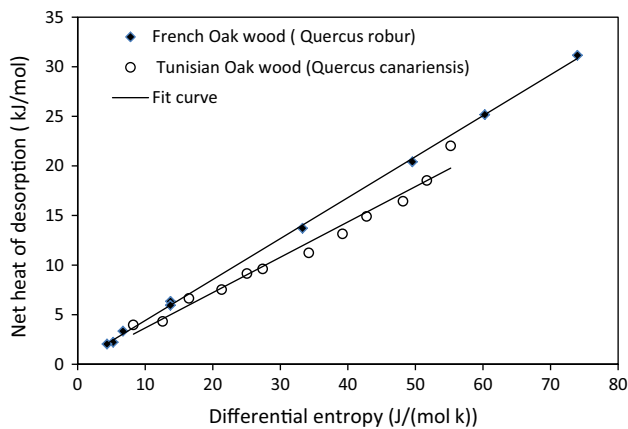


Fig. 9 Enthalpy–entropy relationship for water desorption processes in two types of wood

Table 4 Characteristic parameters for enthalpy–entropy relationship

Product	T_{β} (K)	ΔG_{β} (J/mol)	r
<i>Quercus canariensis</i>	356	97	0.973
<i>Quercus robur</i>	413	283	0.999

55.23 to 8.25 J/(mol K) with moisture content ranging from 0.02 to 0.18 kg/kg DM and from 0.02 to 0.14 kg/(kg DM), for *Quercus robur* and *Quercus canariensis*, respectively. Similar trends of desorption entropy were found for tropical wood and Klinky Pine wood [20, 24]. This results is expected because the differential entropy of desorption measures the molecule ordering change. It is lower when water molecular movement is more restricted at high moisture content [42, 72].

The variation of desorption entropy (ΔS) with moisture content for *Quercus robur* and *Quercus canariensis* was

fitted by an exponential function similar to the behaviour exhibited for the isosteric heat of desorption:

$$\Delta S = 116.738 \exp\left(\frac{-X_{eq}}{0.045}\right) \quad (r = 0.995) \quad (Quercus\ robur) \quad (15)$$

$$\Delta S = 76.62 \exp\left(\frac{-X_{eq}}{0.0785}\right) \quad (r = 0.985) \quad (Quercus\ canariensis) \quad (16)$$

Similar exponential trend for desorption entropy was reported for Palm and Jack pine wood [11]. However, McMinn et al. [55] Reported power law for potato.

4.4 Enthalpy–entropy compensation theory

A linear relationship related the isosteric heat of desorption to differential entropy of desorption for two wood varieties was shown in Fig. 9. The results confirm the enthalpy–entropy compensation theory. The isokinetic temperature (T_{β}) and free energy (ΔG_{β}) were determined by linear regression of Eq. (8). The characteristic parameters for enthalpy–entropy relationship for selected wood varieties were presented by the following fit curves:

$$Q_{st,n} = 0.413 \Delta S + 0.283 \quad (r = 0.999) \quad (Quercus\ robur) \quad (17)$$

$$Q_{st,n} = 0.356 \Delta S + 0.097 \quad (r = 0.973) \quad (Quercus\ canariensis) \quad (18)$$

The sign of (ΔG_{β}) was found to be positive (+283 J/mol) and (+97 J/mol) for *Quercus robur* and *Quercus canariensis*, respectively, indicating a non-spontaneous desorption process (Table 4). These positive values of Gibbs free energy (ΔG_{β}) is totally in agreement with thermodynamic laws because the desorption is a non-spontaneous process. The *Quercus robur* and *Quercus canariensis* samples are exposed to high relative humidity degree, then these samples are placed at low relative humidity level, until thermodynamic equilibrium. According to [30, 31] the non-spontaneous reaction requires input energy from the surrounding environment to the subjected product. Therefore, Gibbs free energy is tied to work necessary to make the sorption sites available to the swelling of the wood structure; the higher the moisture content, the lower the number of available sites [20].

The isokinetic temperature (T_{β}) value of *Quercus robur* and *Quercus canariensis* is 413 and 356 K, respectively. The harmonic temperatures (T_{hm}) was found to be $T_{hm} = 337.78$ K (calculated by applying Eq. 9). Based on our results, it is observed that $T_{\beta} \neq T_{hm}$ for two products. Furthermore, our result shows that $T_{\beta} > T_{hm}$ indicating an enthalpy controlled desorption process. This result indicates the stability of the microstructure of two types of wood during the water desorption reaction in the

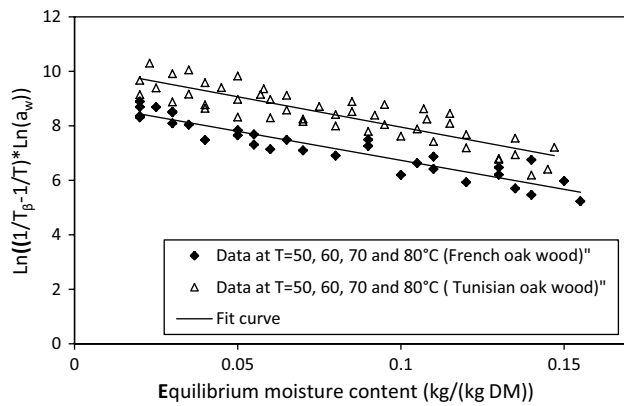


Fig. 10 Equilibrium moisture data of Oak wood plotted according to Eq. 12

Table 5 Values of parameters relative to Eq. (12)

Wood species	K_1 (K)	K_2	r
<i>Quercus canariensis</i>	2579.293	0.000637	0.924
<i>Quercus robur</i>	5639.5	0.000964	0.972

temperature range from 50 to 80 °C [42]. These observations of enthalpy–entropy compensation are obtained for other woods such as Jack pine and palm wood [11], Douglas fir heartwood [19] and quince [73].

The enthalpy–entropy compensation theory was also applied to model the effect of temperature on moisture desorption. Figure 10 shows the typical evolution of $\ln((1/T_\beta - 1/T) \ln(a_w))$ with moisture content. A straight line was obtained for the two types of Oak wood. This behaviour confirms that the temperature effect on moisture desorption follows a power law [34, 42]. The values of K_1 and K_2 parameters of two products were calculated using linear regression and are presented in Table 5. According to [11], the values of K_1 and K_2 were found to be, respectively, 5506.6 K and 3.69×10^{-9} for water desorption of Jack pine and 5859.38 K and 0.00018 for Palm wood.

5 Conclusions

The desorption isotherms of *Quercus robur* and *Quercus canariensis* wood have determined at 50, 60, 70 and 80 °C and over range of relative humidity by using the static gravimetric method. Based on experimental methods and physical laws, the predictive correlations of essential thermodynamic properties of water desorption isotherms of products, were established and compared. Results from this study led to the following conclusions:

- The obtained desorption isotherms of Oak woods at various temperature presented sigmoid forms of type II. The equilibrium moisture content of wood decreases with increasing temperature at a given water activity and decreases with decreasing water activity at a constant temperature.
- From four sorption models tested, the ‘Thermodynamic’ equation was selected to predict the equilibrium moisture content of Oak wood in the whole range of temperature and relative humidity investigated.
- The isosteric heat and differential entropy of desorption decreases with increasing moisture content in an exponential law.
- Differential enthalpy versus desorption entropy data satisfies the enthalpy–entropy compensation theory. According to the positive sign of the Gibbs free energy, desorption is a spontaneous process. It requires continuously energy from external environment to maintain the desorption process.
- As the isokinetic temperature is greater than the harmonic mean temperature, the desorption process is enthalpy-controlled mechanism. Heat was the responsible energy to active the desorption process.

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Appendix

$$s = \sqrt{\frac{\sum_{i=1}^{n_{\text{expdata}}} (X_{\text{eq}_i} - X_{\text{eq}_{\text{cal},i}})^2}{n_{\text{exp.data}} - n_{\text{param}}}}$$

$$r = \sqrt{1 - \frac{\sum_{i=1}^{n_{\text{expdata}}} (X_{\text{eq}_i} - X_{\text{eq}_{\text{cal},i}})^2}{\sum_{i=1}^{n_{\text{expdata}}} (\bar{X}_{\text{eq}} - X_{\text{eq}_i})^2}}$$

$$\bar{X}_{\text{eq}} = \frac{1}{n_{\text{exp.data}}} \sum_{i=1}^{n_{\text{expdata}}} X_{\text{eq}_i}$$

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