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Sorptions isotherms and isosteric heats of sorption of olive leaves (*Chemlali* variety): Experimental and mathematical investigations

Boudhrioua Nourhène[a,*], *Bahloul Neila*[a], *Kouhila Mohammed*[b], *Kechaou Nabil*[a]

[a] *Groupe de Recherche en Génie des Procédés Agroalimentaires de l'Unité de Recherche en Mécanique des Fluides Appliquée et Modélisation, Ecole Nationale d'Ingénieurs de Sfax, BP 'W' 3038, Sfax, Tunisia*

[b] *Laboratoire d'Energie Solaire et des Plantes Aromatiques et Médicinales, Ecole Normale Supérieure, BP 2400, Marrakech, Morocco*

ABSTRACT

Knowledge of sorption isotherms of agricultural products is necessary to control rehydration/dehydration and storage processes. The aim of this work is to determine moisture adsorption and desorption isotherms of Tunisian olive leaves of *Chemlali* variety (*Olea europea* L.). The static gravimetric method was used to determine sorption isotherms of olive leaves at three temperatures (40, 50 and 60 °C) and in the range of water activity varying from 0.0572 to 0.898. Eight models available in the literature were used to describe the experimental data. The agreement between experimental and calculated sorption isotherms was satisfactory (correlation coefficients and standard deviation values ranging, respectively, from 0.91 to 0.99 and from 5.6×10^{-5} to 0.23). GAB and Peleg models were found to be the best for describing the relationship between the equilibrium moisture content, the water activity and temperature. The isosteric heats of desorption and adsorption were determined from sorption isotherms and then correlated with corresponding equilibrium moisture contents. The isosteric heat of desorption was higher than the isosteric heat of adsorption and both decreased continuously with increasing of the equilibrium moisture content.

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Keywords: Olive leaves; Gravimetric method; Mathematical treatment; Adsorption; Desorption

1. Introduction

Olive leaves are known for their therapeutic and medicinal properties. They are used in both traditional and modern medicine. Olive leaves are a rich natural source of polyphenolic compounds which have biologic activities such as antioxidant, antibacterial and antifungal properties (Zarzuelo, 1991). These compounds could also be used for cosmetic and food preservation industries. Tunisian olive tree has always been explored for harvesting olive fruit and/or extracting olive oil. Olive leaves are not yet investigated at industrial scale. Fresh olives leaves evolve rapidly after falling in the ground and are always damaged by dust, insects and micro-organisms. Studies should be performed on fresh and/or processed olive leaves in order to define theirs optimal storage conditions required to increase their longevity before and after their processing. Consequently, knowledge of water sorption isotherms of olive leaves is necessary.

Moisture sorption isotherms could be defined as the curve presenting the variation of the equilibrium moisture content as a function of water activity (a_w) at a fixed temperature and appropriate environment of relative humidity. The concept of water activity is a measure of the availability of water in the product and could be further defined as the ratio of the water vapor pressure exerted by the food material to the vapor pressure of pure water, at the same temperature (Labuza, 1986). Desorption and adsorption isotherms could be established, respectively, by dehydration or rehydration of the product at fixed conditions of relative humidity and

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[*] *Corresponding author.*
E-mail addresses: nourhene.boudhrioua@fss.rnu.tn (B. Nourhène), neilabahloul@yahoo.com (B. Neila), kouhila@hotmail.com (K. Mohammed), nabil.kechaou@enis.rnu.tn (K. Nabil).

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Nomenclature

a_w	water activity
L_v	latent heat of vaporization of pure water (kJ mol^{-1})
Q_{st}	isosteric heat of sorption (kJ mol^{-1})
$q_{st,n}$	net isosteric heat of sorption (kJ mol^{-1})
R	universal gas constant (J mol^{-1} K^{-1})
r	correlation coefficient
S	standard error
T	absolute temperature (K, °C)
X_{eq}	equilibrium moisture content (kg/kg d.b.)
X_{opt}	optimum moisture content (kg/kg d.b.)

temperature. According to Brunauer et al. (1938) classification (BET classification) of sorption isotherms, based on Van der Waals adsorption of gases on solid substrates, five generalized sorption isotherms were distinguished. The sorption isotherm could be used for many technologic and scientific purposes. In fact, it allows the determination of optimal conditions (temperature and relative humidity) of packaging, storage and/or air drying of the material. The equilibrium moisture content of the product could be compared to the final moisture content of the product dried at the same condition to judge the stability of the processed material (Dumoulin et al., 2004). The relationship between water activity (a_w) and temperature (T) at constant equilibrium moisture content is described by moisture sorption isosters. Thermodynamic properties such as the isosteric heats of sorption could be deduced from sorption isotherms established at two or more temperatures. The energy required to dehydrate or rehydrate the product could thus be determined. The isosteric heats of sorption could be used to analyze, design different food processes and to study moisture–solid interactions.

Mathematical models are very useful for describing and predicting sorption isotherms at various experimental conditions and so to calculate all thermodynamic properties that could be deduced. More than 77 equations having two or more parameters have been suggested in the literature for mathematical investigation of experimental data of sorption isotherms (Oswin, 1946; Halsey, 1948; Henderson, 1952; Chung and Pfost, 1967; Chirife and Iglesias, 1978; Motarjemi, 1988; Costa et al., 1998). These equations are theoretical, semi-theoretical or empirical. Some equations have a dependent temperature term and were used to reflect the temperature dependency of moisture sorption isotherms. Each of the suggested equations has some success in describing sorption isotherms for a given type of product according to the Brunauer classification. The GAB equation is considered as the most suitable model to be applied to many products types and over a wide range of water activity. However, there is actually, no perfect equation for describing sorption isotherms for all biological materials.

The aim of this work is to establish sorption isotherms for Tunisian olive leaves of *Chemlali* variety (*Olea europea* L.) at three temperatures (40, 50 and 60 °C). Experiments were performed by using the static gravimetric method. Eight mathematical equations were chosen from the literature to fit the experimental data. The variations of the isosteric heats of sorption of olive leaves as a function of the equilibrium moisture contents were determined.

2. Material and methods

2.1. Raw material

Fresh olive leaves of *Chemlali* variety (*Olea europea* L.) were obtained from the farm of the Olive Institute of Sfax (a southern Tunisian country). Olive leaves were cut into parallelepiped samples for sorption experiments.

2.2. Global chemical analysis

In order to characterize the olive leaves, analysis were realized according to the Association of Official Analytical Chemists (AOAC, 1984): moisture content, by the gravimetric method at 105 °C up to constant weight (24 h). Total protein was determined by the Kjeldahl method. Protein was calculated using the general factor (6.25) (El-Shurafa et al., 1982). Fat content was determined by the Soxhlet method, using hexane as a solvent. Ash content was measured by using a muffle at 550 °C up to constant weight (4 h). Carbohydrate content was estimated by difference of mean values, i.e., 100 − {Sum of percentages of moisture, ash, protein and lipids} (Al-Hooti et al., 1998; Barminas et al., 1999).

The sample weight was measured by an analytical balance (METTLER-TOLEDO) having a precision of ±0.0001 g. Moisture content was expressed in both wet (g moisture/100 g fresh leaves) and dry basis (kg moisture/kg d.b). Protein, fat, carbohydrates and ash contents were expressed in wet basis (g/100 g fresh leaves). All analytical determinations were performed in triplicate. Values of different parameters were expressed as the mean ± standard deviation.

2.3. Sorption isotherms experiments

Sorption isotherms of olive leaves were determined by the gravimetric static method. Six saturated salt solutions were prepared by dissolving an appropriate quantity of salt in distilled water (Dumoulin et al., 2004). Each salt solution was prepared in a glass jar of 1 l with an insulated lid. The jars are immersed in a thermostated water bath adjusted to fixed temperature (T) for 24 h to be stabilized. Each jar provides a fixed relative humidity (RH) corresponding to a fixed water activity (a_w) for each temperature and salt concentration (Table 1).

Olive leaves samples used for both adsorption and desorption experiments were of 0.1 ± 0.0001 g. For adsorption experiments olive leaves samples were dried 24 h in an oven at 105 °C before putting them on a tripod in the glass jars. For desorption experiments, fresh olive leaves were used. The six jars containing olive leaves samples were put in thermostated water baths adjusted to fixed temperatures (40, 50 and 60 ± 1 °C) (Fig. 1). The samples were weighed at differ-

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

Salt	Water activity		
	40 °C	50 °C	60 °C
KOH	0.0626	0.0572	0.0549
$MgCl_2$	0.3159	0.3054	0.2926
K_2CO_3	0.4230	0.4091	0.3921
$NaNO_3$	0.7100	0.6904	0.6735
KCl	0.8232	0.8120	0.8025
$BaCl_2$	0.8910	0.8823	0.8728

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