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Moisture Adsorption Characteristics of Lyophilized Algerian *Arbutus unedo* L. Fruit Powder

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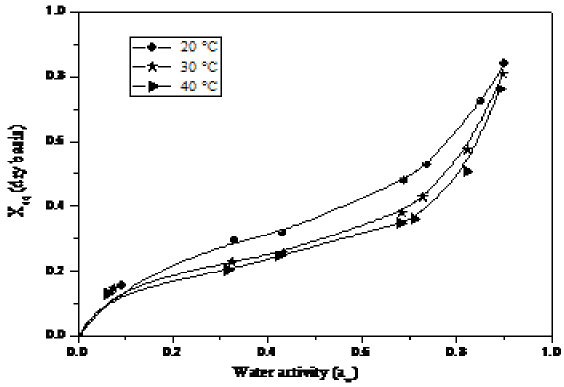
Abstract

The present work aims to investigate the moisture adsorption characteristics of Lyophilized Algerian *Arbutus unedo* L. fruit powder (LP). First, the LP was evaluated for some of its physicochemical parameters, including X-ray diffraction (XRD) properties, crude fiber, titrable acidity, etc. Second, the experimental sorption curves, determined at 20, 30 and 40°C with the standard static-gravimetric method, were fitted to six isotherm models (Kühn, Caurie, Smith, Halsey, Oswin and GAB). Based on XRD pattern, LP seemed to contain essentially amorphous sugar. Results showed also that the moisture adsorption isotherms of LP are of S-shaped profile (Type II), generally obtained for biomaterials. Among all tested models, those of Halsey and GAB (T=20 and 30°C) gave the best fits at 20 and 30°C, with the mean relative percentage deviation modulus (*E*%) less than 1%, $\chi^2 \leq 2.68\ 10^{-1}$ and a root mean square error (RMSE) ≤ 0.2808). The K parameter of GAB model was found to increase with increasing temperature, whereas the monolayer moisture content (X_0) decreased with increasing temperature. Such data are represent a useful tool for choose appropriate storage conditions of LP.

Graphical Abstract

(a)



(a) LP of *Arbutus unedo* L. fruit. (b) Adsorption isotherms of LP at different temperatures.

Keywords: Lyophilized powder; Sorption isotherm; Modeling; Gravimetric method; Water activity

Introduction

Strawberry tree (*Arbutus unedo* L.; Ericaceae family) is a typical Mediterranean wild tree, which is also cultivated in other regions of Eastern Europe [1]. Its fruit mature in autumn, at the same time as flowering [2]. It is fleshy and globular, from 1 to 1.7 cm in diameter. Its color changes from green to yellow, then to orange red and to bright red at maturity [3]. Berry fruits can be used for the fabrication of several industrialized products [4,5] since it is a rich numerous nutrinzents, especially Calcium, Phosphorus and Potassium [6]. Strawberry tree fruits are a good source of naturally occurring antioxidants [7,8]. Like other plants which are fitted with wonderful defense system assured by various biopharmaceuticals [9], the berries are also known to be used in the folk medicine as antiseptic, diuretic and laxative and against cardiovascular pathologies [10].

Establishing the relationship between equilibrium moisture content (EMC) and a_w, also known as sorption isotherm (adsorption or desorption), is one of the useful measurements to the stability, microbiological and the physicochemical deterioration reactions [11] of a food's, select formulations and storage conditions in new products and to improve drying process and equipment [12].

water activity (a_w) is an important concept and essential parameter which describes the water availability and mobility in foods [13].

The sorption isotherms show the amount of adsorbed water as a function of steady state water activity (a_w), at constant temperature [14], it can also be used to investigate the structural features such as the specific surface area, the pore volume, the pore size distribution

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and the crystallinity of food product [15]. Such data can be used for selecting the storage conditions and packaging systems [16] in order to prolong the shelf-life of food products. A number of equations allow the moisture content to be related to water activity [17,18].

Some studies have been carried out on the sorption isotherm of various herbs, aromatical/medicinal plants [19-21] and Wild fruits. Bag et al. [22] reported the moisture desorption isotherm of bael (*Aegle marmelos)* pulp and adsorption isotherm of pulp powder while Alakali and Satimehin, [23] determined the adsorption equilibrium moisture content of Bambara groundnut (V*igna subterranea*) powders. Alexandre et al. [24] showed the moisture adsorption isotherms of red Brazilian cherry powder. Vega-Galvez et al. and Alcântara et al. [25,26] determined the adsorption isotherms of Blueberry powder and Dry cashew apple, respectively.

The lyophilized powder (LP) from Algerian arbutus wild berries (*Arbutus unedo* L.) has been used previously in the elaboration of tablets [27,28] but for storing the raw material (LP) and keep its nutritional quality causes a problem. Consequently the study of the moisture sorption characteristics of LP under various environmental conditions is imperative. There was no research report on the moisture sorption isotherms of arbutus berry powder; we sum interesed to establish the relationship between the equilibrium moisture content and water activity of LP powder at three different temperatures; (20, 30 and 40°C), to find the most and to evaluate the suitability of various models for fitting the isotherms.

Materials and Methods

Fruit and fruit powder

Fully ripe berries were randomly picked at various trees in the Kabylie region (North Algeria) during the winter 2016. The fruit was submitted to freeze drying at -64°C under vacuum (4.5 Pa) during 48 h, using lyophilizer Type (Christ Alpha1-4LD), provided with a vacuum pump (RZ 6, max pressure 0.04 Pa). The dried product is ground, sieved (sieve of type Euromatest-Sintoo, NFX11-501) to obtain powder with particle diameters (200 ≤ Ø ≤ 400 µm) and then kept in closed glass flask at 4°C. The general chemical parameters of LP *A. unedo* berries, namely; crude fiber [29], titrable acidity (measured by titration with NaOH, 0.1 N), pectin [30], ash and Acid-Insoluble Ash [31] were evaluated. The electrical conductivity of 20% LP solution in distilled water was measured at 20°C (mS cm^{-1}); the lipid was determined by extracting a known weight of powdered sample with petroleum ether, using a Soxhlet apparatus. The X-ray diffraction (XRD) of LP was investigated using diffractometer (Panalytical Xpert Pro ®).

Sorption isotherms

The sorption isotherms of LP were determined with the standard, static-gravimetric method [32] at 20, 30 and 40°C. Six saturated salt solutions were prepared corresponding to a range of water activities (0.0626-0.9200) (Table 1). These solutions were prepared in hermetic jars and maintained in a drying room regulated in desired temperature. Triplicate samples of around 0.5 g of LP were weighed into small glass receptacles and placed on tripods in jars, above salts. The required equilibration time was 15-20 days based on the change of the weight expressed on a dry basis, which did not exceed 0.1%. (0.001 gg^{-1} dry solids). The equilibrium moisture content was determined in a vacuum oven at 40°C for 24 h and calculated by:

$$X_{eq}=(M_f-M_d)/M_d \qquad \textbf{Eq. 1}$$

Where, X_{eq} is the equilibrium moisture content (g water g^{-1} dry matter), M_f is the final weight (g), M_d is the dry weight solid (g).

The isotherm models used to fit the data are presented in Table 2. These equations were chosen to fit the experimental sorption data because they are most widely used for several foods.

The statistical analysis of experimental data was performed with Origin software version 8. Goodness of fit of the selected models was evaluated by means of the coefficient of determination (R^2), the mean relative percentage deviation modulus ***(E%)***, the chi-squared error (**χ^2**) and the root mean square error (**RMSE**) [16].

$$RMSE=\left[\frac{1}{N}\sum_{i=1}^{N}(X_{exp}-X_{cal})^2\right]^{1/2} \qquad \textbf{Eq. 2}$$

$$X^2=\sum_{i=1}^{N}\left[(X_{exp}-X_{cal})^2/X_{cal}\right] \qquad \textbf{Eq. 3}$$

$$E\%=\frac{100}{N}\sum_{i=1}^{N}\frac{|X_{exp}-X_{cal}|}{X_{exp}} \qquad \textbf{Eq. 4}$$

Where, X_{exp} is the experimental value, X_{cal} the value predicted by the model and N the number of experimental measurements. It is generally assumed that a good fit is obtained when *E* lesser than 10% [41,42] and that is extremely good for values of ***E%*** lower than 5% [43].

The temperature dependence of the GAB model constants was given by the Arrhenius equations [44]:

$$C=C_0 exp(\Delta Hc)/RT \qquad \textbf{Eq. 5}$$

$$K=K_0\exp(\Delta H_K)/RT \qquad \textbf{Eq. 6}$$

$$X=X_0\exp(-E_a/RT) \qquad \textbf{Eq. 7}$$

Where $\Delta H_K=h_w-H_m$ and $\Delta H_c=H_m-H_n$

C_0, K_0, X_0 are pre-exponential factors, (kjmol^{-1}), H_m, H_n and h_w are respectively; the sorption enthalpy of monolayer, multilayer and condensation of water. E_a is the activation energy (kJ mol^{-1}) for the monolayer moisture content. R is the gas constant and T is the absolute temperature. All constants were estemited by the regression analysis of Eqs. (5-7).

Results and Discussion

Physicochemical properties of LP

The different quality parameters of LP are summarized in Table 3.

Solutions	Water activity (a_w) at		
	20°C	30°C	40°C
KOH	0.0932	0.0738	0.0626
$MgCl_2$	0.3307	0.3244	0.3160
K_2CO_3	0.4316	0.4317	0.4230
$CuCl_2$	0.6880	0.6860	0.6800
$NaNO_3$	0.7536	0.7314	0.7100
KCl	0.8510	0.8360	0.8230
$BaCl_2$	0.9200	0.8980	0.8920

Table 1: Selected saturated salt solutions and corresponding water activity [30,33,34].

Model	Mathematical expression
GAB [35]	$X=(X_0CKa_w)/(1-Ka_w)(1-Ka_w+CKa_w)$
Halsey [36]	$X=[-A/(Lna_w)]^{1/B}$
Smith [37]	$X=A-B\ Ln(1-a_w)$
Oswin [38]	$X=A[a_w/(1-a_w)]^B$
Kühn [39]	$X=(B/Ln\ a_w)+A$
Caurie [40]	$X=exp(A+Ba_w)$

Table 2: Model equations fitted to the experimental sorption data of LP.

Crude fiber of LP is comparable to that reported by Ruiz-Rodríguez et al. [8] and is less than that reported by Özcan and Hacıseferogulları, [6] for fresh strawberry tree fruits (6.4 g/100 g of cellulose, 2.93 g/100 g soluble fibers respectively). The titratable acidity is close to that indicated in the literature 0.4% [6], On the other hand, it is less than that given by Sulusoglu et al. and Celikel et al. [2,45] (0.48-1.24 and 0.8-1.59% respectively) for the Turkish variety electric conductivity is greater than that calculated by Ulloa et al. [46] (0.643 mS cm^{-1}) for strawberry tree (*Arbutus unedo* L.) honey.

The XRD pattern of LP powder is presented in Figure 1. A broad band with very weak peaks, characteristic of amorphous forms, is observed in the pattern indicating the presence of amorphous sugar obtained by freeze-drying fruits berry. Furthermore, the amorphous characteristics are clearly reported on different dried mango powders [47] and fluidize-dried gum extracted from the fresh fruits of *Abelmoschus esculentus* [48]. However, Niimura et al. [49] have shown that strawberry flesh has low-crystallinity cellulose I.

Sorption isotherms

The adsorption isotherms of LP, at different temperatures, are shown in Figure 2. As it can be observed, at a constant water activity, the equilibrium moisture contents increase with decreasing temperature; similar trends were reported by Vega-Galvez et al. and Vaquiro et al. [50,51]. This trend can be explained by considering excitation states of molecules. At increased temperatures the molecules are in an increased excitation state, thus increasing their distance apart and decreasing the attractive forces between them [52]. This leads to a decrease in the degree of water sorption at a given relative humidity with increasing temperature [53,54]. According to Catelam et al. [55] the decrease in X was due a reduction in the number of active sites due to chemical and physical changes induced by temperature and then depend on the composition of foods [15,56]. Further, examination of the figure shows that the isotherms are S-shaped (Type II). This is a typical characteristic of many biomaterials [57-59] and of fruits rich in sugars [16,60].

Parameter	Value
Crude fiber (%)	4.440 ± 0.125
Titrable acidity (%)	0.210 ± 0.010
Pectin (%)	2.456 ± 0.034
Total ash	3.910 ± 0.030
Acid-Insoluble Ash (%)	0.510
Lipid (%)	0.801 ± 0.080
Electrical conductivity (mS.cm^{-1})	2.550 ± 0.050

Table 3: Physicochemical characterization of LP.

The average parameters related to various mathematical models, as well as the corresponding statistical data applied are recapitulated in Table 4. Graphical representation of the fit goodness of theoretical isothermsat 20, 30 and 40°C are shown in Figure 3. For all tested models, the parameters A, B and K are found to be temperature dependent and all models, with the exception of the Oswin over the used temperature range and GAB at 40°C, for values water activity greater than 71%, gave good fits to experimental data over the range of water activities employed, with *E* less than 10%. The Halsey and the GAB models (at T=20 and 30°C) gave the best fits (*E*<1%), and the lowest average values of χ^2 and RMSE.

These results are comparable to those recorded by others, Lamharrar et al. [59] have also reported that the GAB model was the best model describing the equilibrium moisture data for desorption, and the modified Halsey model was the most suitable to estimate adsorption isotherms of *Artemisia herba-alba*, while Lavoyer et al. [61] found very good adjustment of the GAB model to adsorption isotherms of green coconut pulp. According to Kohayakawa et al. [62], the GAB modelhas been extensively used for foodstuffs, mainly for fruits. Chukwu, [57] showed that the Oswin and the Bradley models gave better fits for the adsorptive mode than for the desorptive mode for the two varieties of dates (Khalas and Handal variety). In this work, the Oswin model gave a poor fit over the entire range of equilibrium moisture contents (*E*>10% at 30 and 40°C).

The the monolayer moisture content (X_0) is of particular interest; it is considered as the optimum value to assure the food stability [15] and it measures number sorbing sites [63]. Below it, the rates of deteriorative reactions, except for oxidation for unsaturated fats, are minimized [64]. Monolayer moisture contents obtained from GAB

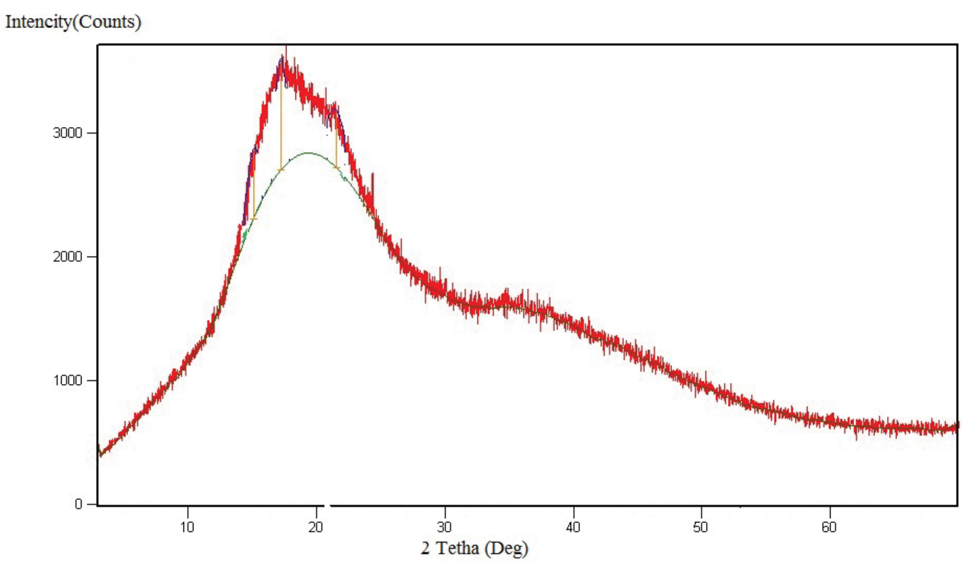

Figure 1: X-ray diffraction patterns of powders freeze dried arbutus berries.

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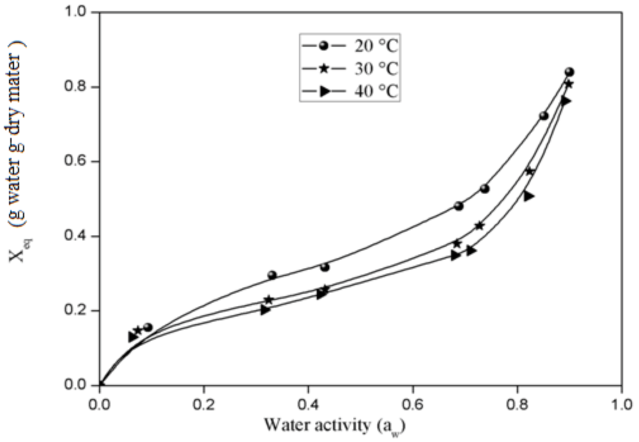

Figure 2: Adsorption isotherms of LP at different temperatures.

Model		Temperature (°C)		
		20	30	40
GAB	R^2	0.966	0.976	0.944
	X_0 (g g^{-1})	0.223	0.157	0.143
	C	25.417	266.100	262.698
	K	0.813	0.900	1.065
	RMSE	4.625×10^{-3}	8.118×10^{-3}	2.680×10^{-1}
	χ^2	4.556×10^{-4}	1.040×10^{-3}	2.808×10^{-1}
	E (%)	0.453	0.573	10.670
Smith	R^2	0.998	0.994	0.965
	A	0.102	-0.064	-0.111
	B	0.322	0.378	0.384
	RMSE	1.573×10^{-2}	1.537×10^{-2}	1.714×10^{-2}
	χ^2	1.568×10^{-2}	1.559×10^{-2}	1.610×10^{-2}
	E (%)	1.064	1.898	2.346
Oswin	R^2	0.996	0.956	0.942
	A	0.094	0.041	0.028
	B	0.694	0.358	0.354
	RMSE	8.845×10^{-2}	1.323×10^{-1}	1.253×10^{-1}
	χ^2	2.710×10^{-1}	2.476	3.328
	E (%)	7.28	12.44	12.86
Caurie	R^2	0.978	0.950	0.939
	A	-1.989	-2.122	-2.219
	B	1.939	1.899	1.914
	RMSE	2.025×10^{-2}	2.806×10^{-2}	2.800×10^{-2}
	χ^2	1.920×10^{-2}	2.00×10^{-2}	2.300×10^{-2}
	E (%)	2.23	2.5	2.527
Halsey	R^2	0.978	0.997	0.990
	A	0.087	0.070	0.059
	B	1.910	1.891	1.859
	RMSE	1.081×10^{-2}	5.723×10^{-3}	1.063×10^{-2}
	χ^2	1.680×10^{-3}	5.421×10^{-4}	1.340×10^{-3}
	E (%)	0.828	0.414	0.599
Khun	R^2	0.915	0.977	0.990
	A	0.231	0.169	0.140
	B	-0.071	-0.072	-0.073
	RMSE	1.994×10^{-2}	9.750×10^{-3}	5.960×10^{-3}
	χ^2	9.740×10^{-3}	2.810×10^{-3}	1.600×10^{-3}
	E (%)	2.20	1.204	0.771

Table 4: Isotherm models used for experimental data fitting.

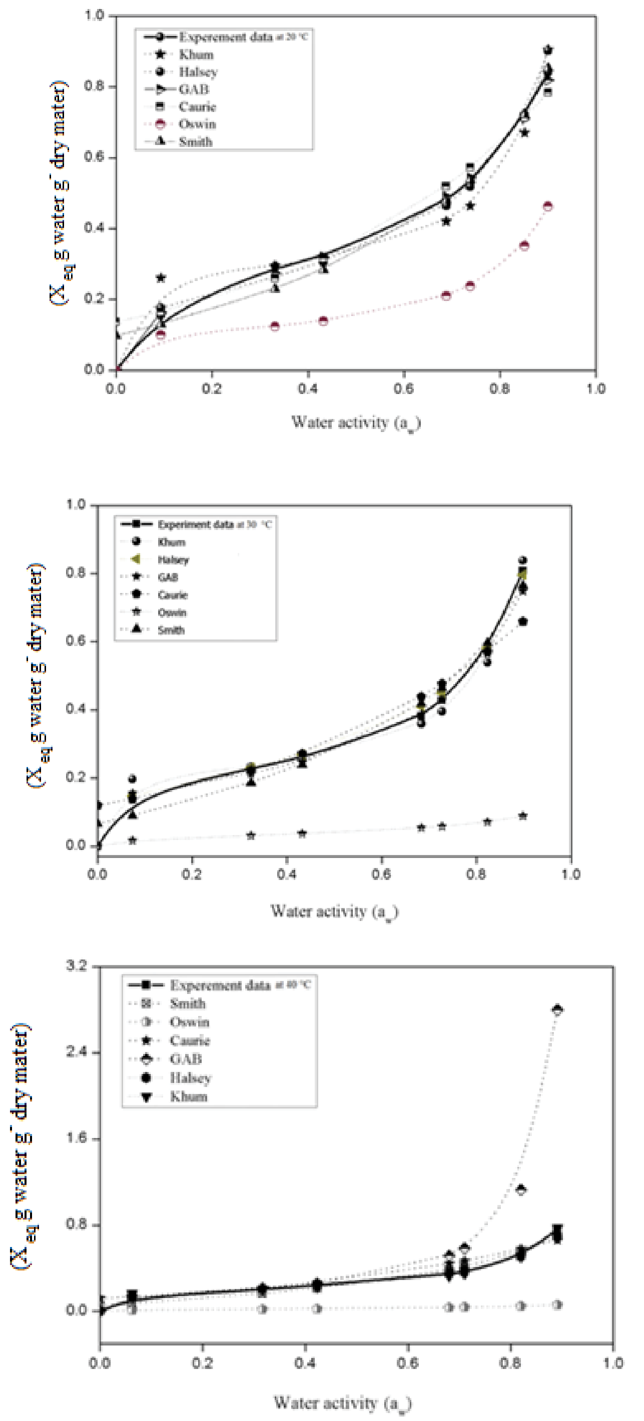

Figure 3: Adsorption isotherms of LP compared to predicted isotherms at: 20, 30 and 40°C.

model (Table 4) were found to decrease with increasing temperature. This can be explained by the reduction of some bonds (hydrogen bonding, ...) by raising the temperature thus makin the less stable water molecules thereby, favoring its break away from the binding sites of the food materials. Palipane and Driscoll [65] suggested that at increased temperature, some water molecules are activated to energy levels that allow them to break away from their sorption sites, thus decreasing in the monolayer moisture content. Estimated values for X_0 were found to be within the range reported values for agro-food products [66]. Aroldo et al. and Bag et al. [67,68] have reported similar effect of temperature on monolayer moisture content on Murici, Inga fruit and Bael pulp respectevely.

There are comparable to values reported by other authors; Kaymak-Ertekin and Gedik, [69] have obtained values in the range (0.067-0.220 gg^{-1}) dry solids for grapes, apricots and apples 30, 45 and 60°C; Talla et al. [70] found values between 0.080 and 0.185 gg^{-1} dry solids Vega-Galvez et al. [54] reported monolayer moisture contents of 0.044-0.075 g g^{-1} dry solids for Cape Gooseberry (*Physalis peruviana* L.) in the temperature 20, 40 and 60°C.

The constant (C) has an enthalpic nature and is a measurement how strong the water molecules are bound to the primary sorption sites [71]. The parameter C showed no temperature dependence but is within the ranges ($5.67 \le C \le \infty$) as indicated by Lewicki, [72] and they is in the same extent as that reported by Alakali and Satimehin, [73] for ginger (*Zingiber officinale*) powders. Iglesias and Chirife, [36] studied more than 30 different foods and found that in 74% of them, *C* increases as temperature increases; they have explained it by irreversible changes associated with increasing temperature, such as enzymatic reactions and protein denaturation. Martínez et al. [74] showed that, the isotherms are classified as type II for C>2. According to Quirijns et al. [71] (very) high banana, mango and pine apples in the temperature range (40-60°C). For pure pineapple pulp, Gabas et al. [75] showed X_0 values in the range (14.6-16.6%) (dry basis) with a decreasing tendency of X_0 with increasing temperature from 20 to 50°C.

Values and K, approaching 1 indicate that the multilayer molecules have properties comparable with those of bulk liquid molecules. K values increase with increasing temperature. According to Cano-Higuita et al. [76] the K value provides a measure of the interactions between the molecules of vapor water in the multilayers with the adsorbent, and tends to decrease between the energy of molecules in the monolayer and those of liquid water and also observed for K close to 1.

Estimated value for K are greater than 1 at 40°C; Quirijns et al. [71] suggests that the high K values (>0.9) indicate that the monolayer and multilayer molecules are not so different and that the multilayer molecules behave more like liquid molecules. The ΔH_c values obtained in this work are not significantly affected by the temperature. The temperature dependence of GAB constants calculated by using the linear regression are summarized in Table 5. The negative value of ΔHk and E_a indicate that the moisture adsorption process is endothermic.

The negative value for ΔHk suggest that stronger bonds exist between monolayer and subsequent multilayer water molecules than the solid and monolayer water molecules. On the other hand, the estimated value indicate that water molecules are less bonded in multilayer [52] and the energy required to release the sorption energy from the multi-layers is higher than that of pure water. These results are similar to those reported by Das et al. [77].

The activation energy deduced in this study is in the same order of that found by Vega-Gálvez et al. (14.48 kJ mol^{-1}) [78] related to adsorption isotherms of Chilean papaya.

X(T)		K(T)	
X_0	2.00×10^{-4}	K_0	54.326
E_a (kJmol^{-1})	-17.026	ΔH_K (kJ mol^{-1})	-10.272
R^2	0.910	R^2	0.949

Table 5: Temperature Dependencies of GAB Constants.

Conclusion

The study of lyophilized powder (LP) (*Arbutus unedo* L.) from Algerian Arbutus berries was undertaken. The XRD pattern of LP indicates the presence of amorphous sugar obtained by freeze-drying fruits berry.

The sorption isotherms constitute an important source of information for the stability products food and its storage conditions.

For the first time, the water adsorption by LP was studied giving the following results: The moisture sorption isotherms of LP exhibited S shape described as type II which is common for many hygroscopic products. The equilibrium moisture content of LP increased with increasing water activity and decreased with increasing temperature. Among all tested models, those of Halsey and GAB (T=20 and 30°C) gave the best fits at 20 and 30°C, with the mean relative percentage deviation modulus (*E*%) less than 1%. PL showed higher monolayer moisture content at 20°C and was found to be less shelf-stable. For LP berry, the monolayer moisture content can be used to evaluate the shelf stability and efficient use of energy in the drying process.

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