



Water sorption characteristics of six row barley malt (*Hordeum vulgare*)

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Abstract

The water sorption isotherms of six-row Belgian barley malt (Plaisant) were determined for a temperature range between 15°C and 35°C. The results obtained showed that for water activity (a_w) values below 0.5 the effect of temperature on the sorption isotherms was negligible. For a_w values above 0.5, an increase of the equilibrium moisture content with temperature at a given a_w was observed, as reported by other authors in products rich in sugars. Due to the malting process, barley malt presents a higher simple sugar content than nonmalting barley. The experimental data was fit to various sorption models. The constants obtained for each equation and the correlation coefficients were presented. From these, it was found that the Brunauer–Emmett–Teller (BET) and Kuhn models showed the highest correlation coefficients for a_w below 0.50. For a_w values above 0.50, the best fitting was obtained for the models of Harkins–Jura, Smith and Henderson, although other models such as Freundlich, Chung–Pfoest and Guggenheim–Anderson–De Boer (GAB) also showed good fitting. Due to the fact that the practical storage moisture content of barley malt is below 60 g/kg, the BET model (suitable for $a_w < 0.50$) would represent the best model to apply for this product. The monolayer water content calculated from the BET and GAB equations ranged between 42.5 and 43.9 g/kg (dry basis), and 46.0 and 51.0 g/kg (d.b.), respectively, for the range of temperature considered in this work (15–35°C). These values corresponded to a_w values ranging from 0.16 to 0.19, according to the BET equation and from 0.19 to 0.21 using the GAB equation. The average sorption enthalpy (Q_s) and the total heat requirement (ΔH_T) of this process (15–35°C) showed values of 7.787 ± 0.262 and 51.834 ± 0.275 kJ/gmol, respectively.

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1. Introduction

Barley is the cereal most frequently malted in the world. Barley malt has been an important trade commodity throughout the world, since it is used in the brewing making process in many countries. Two types of barley are frequently used for the malting process: six- and two-row. Two-row barley produces malt with a large extract, lighter color and less enzyme content than the six-row type (Broderick, 1977). Malting barley should have a minimum of broken kernels and must be free of mold growth, heat damage and foreign seeds.

The malting process includes cleaning of the barley to remove foreign seeds, broken kernels and other impu-

rities, followed by a steeping process consisting in soaking the grain in water in order to increase moisture to 420–440 g/kg. After steeping the grain is removed from water and allowed to germinate, with the formation of rootlets and acrospire, under controlled temperature and moisture conditions. The main objective is to produce the minimum amount of growth, maximum endosperm modification, yielding a grain with high enzyme activity. After germination malt is dried in a kilning process in order to halt growth and produce a storable product with low water activity (a_w) with the characteristic color and flavor of malt. During this process enzymes are synthesized and activated, among others α - and β -amylases. The drying process must be carefully controlled in order to avoid overheating and damage of the enzymatic system. Rootlets are removed during the malt cleaning process. Part of the endosperm is consumed to produce energy for the germination

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process. As a result of the malting process there is an increase in enzyme activity, soluble protein and, breakdown of starch into simple sugars, along with development of the typical color and flavor (Hoseney, 1994). The final moisture content of malt is about 35–40 g/kg, being a highly hygroscopic product.

Some problems of barley malt stability during storage and sea transportation have been found when the products is packaged in bags and carried as break bulk cargo in ship holds subjected to exposure and ventilation with hot moist tropical air. Ambient moisture can be adsorbed into the product due to mass transfer mechanisms through the packaging material or by entrance of condensation water in nonhermetic bags. Condensation over the bag material takes place when the cargo is loaded cool and the temperature of the surface is below the dew point of the surrounding air.

The knowledge of the water sorption isotherms is of prime importance to perform engineering calculations in order to predict the stability of malt during storage and transportation under humid conditions.

Limited water sorption data for barley malt could be found in the literature consulted. Coleman and Fellows (1955) presented sorption information for nonmalting barley at 25°C. Studies of moisture desorption isotherms of malt over temperature and relative humidity ranges from 15°C to 90°C and 20–910 g/kg were conducted by Tuerlinckx, Berckmans, and Goedseels (1982). The authors developed theoretical, empirical and semi-empirical models that could be used in the minimization of energy consumption during kilning of malt. The practical storage moisture content reported for barley malt for brewing purposes is below 60 g/kg, with a typical moisture content ranging from 35 and 40 g/kg (Broderick, 1977; Kirk-Othmer, 1982).

The objectives of this research work were: to determine the water sorption isotherms for six-row barley malt at temperatures usually found in storage of this commodity in tropical regions (15°C, 25°C and 35°C); to establish a sorption model capable of fitting the data in order to predict the moisture sorption isotherms in the temperature range studied; and to calculate the monolayer moisture content and the sorption enthalpy.

2. Materials and methods

2.1. Sample collection

Six-row Belgian barley malt (Plaisant), of the 1996 harvest, intended for brewing purposes was used in this research work. The sample was drawn from a large lot of bags using a grain trier (25 cm long) following a statistical sampling plan for bagged commodities (Anonymous, 1982). More than 50 primary samples

(about 500 g each) were used to form a composite laboratory sample of about 5 kg, using a grain divider.

2.2. Moisture analysis

The moisture content was determined using a vacuum oven with a pressure of less than 25 mmHg, at 100°C for approximately 5 h, until constant weight was reached (AOAC, 1996). The analyses of the raw material were done in triplicate.

2.3. Water sorption isotherm

Primary samples had a moisture content ranging from 40 to 50 g/kg (wet basis). The grain used for the experiments was dehydrated in a forced convection oven at 70°C for enough time to reach a moisture content of 39.81 g/kg.

In order to determine the water sorption isotherms, the grain was ground in a motorized mill (Wiley No. 4) using a 20-mesh sieve. Sodium azide (5 g/kg) was added to the ground grain to inhibit mold growth in samples with $a_w > 0.8$ (KCl and K₂SO₄ saturated solutions).

A weight of about 1.5 grams of the ground samples with an initial moisture content of 40.14 g/kg (w.b.) and $a_w = 0.169$ at 22.9°C, was placed in open shallow plastic containers (3.8 cm diameter), filled to a height of about 0.6 cm. The a_w was measured using a Decagon CX-1 equipment, previously calibrated with a saturated salt solution of lithium chloride as salt standard. The containers were placed inside desiccators, each one containing saturated salt solutions of known equilibrium relative humidity at the temperatures studied. Ten saturated salt solutions were used in these experiments. Two desiccators were used for each salt solution in order to obtain duplicates for the results obtained. A range of a_w from 0.0673 to 0.9789 was studied, with the following salts (reagent grade) used for this purpose: KOH, C₂H₃KO₂, MgCl₂, K₂CO₃, Mg(NO₃)₂·6H₂O, NaNO₃, NaCl, KCl and K₂SO₄ (Anonymous, 1984). The a_w values for the saturated solutions of these salts at the temperatures studied (15°C, 25°C and 35°C) were obtained from Greenspan (1977). In this way, part of the data was obtained by adsorption (product containing final moisture equilibrium values above 40.14 g/kg) and the rest by desorption.

The desiccators with the samples were kept in temperature controlled incubators ($\pm 0.5^\circ\text{C}$) at 15°C, 25°C and 35°C. After equilibrium was reached, as evidenced when the sample reached constant weight, the moisture content of the equilibrated samples was determined by the vacuum oven method indicated above. It was experimentally shown that equilibrium was reached within a maximum of 24 days for the samples with the large a_w . Moisture determinations were done in duplicate and the averages calculated.

Table 1

Adjustment of the experimental data obtained for the sorption isotherm (15°C, 25°C and 35°C) of six-row Belgian barley malt to various sorption models

Isotherm	Model	Range of a_w	Temperature (°C)					
			15°C		25°C		35°C	
			Constant values	R^2	Constant values	R^2	Constant values	R^2
Freundlich (1926)	$V = A(a_w)^{1/B}$	$a_w < 0.90$	$A = 150.803$ $B = 1.3904$	0.965	$A = 159.401$ $B = 1.3029$	0.961	$A = 170.471$ $B = 1.3114$	0.925
BET (1938)	$a_w / [(1 - a_w)V] = A + Ba_w$ $A = 1/V_m C$ $B = (C - 1)/V_m C$	$a_w < 0.50$	$V_m = 43.346$ $C = 29.351$	0.999	$V_m = 43.883$ $C = 18.724$	0.998	$V_m = 42.488$ $C = 30.344$	0.998
Harkins-Jura (1944)	$\ln a_w = A + B(1/V^2)$	$a_w > 0.5$	$A = 0.0519$ $B = -4946.315$	0.999	$A = 0.0034$ $B = -4684.56$	0.996	$A = 0.0123$ $B = -6354.86$	0.997
Smith (1947)	$V = A + B \ln(1 - a_w)$	$0.5 < a_w < 0.95$	$A = 35.627$ $B = -62.763$	0.999	$A = 23.224$ $B = -78.681$	0.996	$A = 17.748$ $B = -92.773$	0.997
Hasley (1948)	$a_w = \exp[A(1/V)^B]$	$0.10 < a_w < 0.80$	$A = -1682.79$ $B = 1.7959$	0.996	$A = -862.47$ $B = 1.6372$	0.995	$A = -395.62$ $B = 1.4436$	0.999
Henderson (1952)	$(1 - a_w) = \exp(AV^B)$	$0.5 < a_w < 0.95$	$A = -0.0011$ $B = 1.4758$	0.997	$A = -0.0025$ $B = 1.2900$	0.994	$A = -0.0055$ $B = 1.1093$	0.996
Chung-Pfost (1967)	$\ln a_w = A \exp(BV)$	$0.20 < a_w < 0.90$	$A = -3.8897$ $B = -0.0209$	0.997	$A = -3.0466$ $B = -0.0176$	0.994	$A = -2.6977$ $B = -0.0152$	0.994
Kuhn (1967)	$V = A / \ln a_w + B$	$a_w < 0.5$	$A = -47.120$ $B = -17.530$	0.999	$A = -52.926$ $B = -10.487$	0.997	$A = -45.880$ $B = -17.101$	0.998
GAB	$a_w/V = \alpha a_w^2 + \beta a_w + \gamma$ $\alpha = K/V_m(1/C' - 1)$ $\beta = 1/V_m(1 - 2/C')$ $\gamma = 1/(V_m K C')$	$a_w < 0.95$	$V_m = 51.030$ $C' = 24.558$ $K = 0.804$	0.993	$V_m = 50.770$ $C' = 23.205$ $K = 0.850$	0.987	$V_m = 46.030$ $C' = 25.181$ $K = 0.904$	0.978

A: regression constant for the isotherm models, K: constant of the GAB model (dimensionless), a_w : water activity (dimensionless), V: moisture content-dry basis (g/kg), B: regression constant for the isotherm models, V_m : water content of the monolayer-dry basis (g/kg), C: constant of the BET equation (dimensionless), C': constant of the GAB model (dimensionless).

The data obtained corresponding to the a_w and moisture content at the temperatures studied were adjusted to several regression models in order to determine the best fit. The following equations for sorption isotherms (Table 1) were studied: Freundlich (1926), BET (Brunauer, Emmett, and Teller 1938), Harkins and Jura (1944), Smith (1947), Hasley (1948), Henderson (1952), Chung and Pfost (1967), Kuhn (1967) and GAB model (Anderson, 1946; de Boer, 1953; Guggenheim, 1966). Linear and polynomial regression statistical analyses were done, depending on the equation, using Microsoft Excel 97.

The water content of the monolayer was determined from the BET and GAB equations, and the sorption enthalpy from the BET equation.

3. Results and discussion

The experimental data obtained relating the moisture content of the malt with a_w for the temperatures studied (15°C, 25°C and 35°C) are presented in Fig. 1. The isotherms obtained had the typical shape of type II isotherms characteristic of cereals grains.

For a_w below 0.50 the three curves were practically coincident, with the effect of temperature on the water

sorption capacity of the grain being negligible. For a_w values above 0.50 a clear differentiation in the water sorption isotherms could be observed. The typical behavior of these curves indicates that for a given moisture content, a_w increases with temperature. In the same way, at constant a_w , temperature and equilibrium moisture content are inversely related. (Jayas & Mazza, 1991). This typical behavior was not observed in this case for barley malt, where the equilibrium moisture content increased with temperature at a given a_w . This behavior has been observed in products such as glucose (Loncin, Bimbenet, & Lenges, 1968), maltose and fructose (Audu, Loncin, & Weisser, 1978), and foods rich in carbohydrates (Iglesias & Chirife, 1982; Chuzel & Zakhia, 1991) such as beets (Iglesias, Chirife, & Lombardi, 1975), banana (Wolf, Spiess, & Jung, 1973), potato slices (Mazza, 1982), carrots (Mazza, 1983), Jerusalem artichokes (Mazza, 1984) and sultana raisins (Saravacos, Tsiourvas, & Tsami, 1986). Barley malt is subjected to a process of malting, which results in elevated enzyme activity and breakdown of complex sugars into simple sugars. Typical composition of barley malt (dry basis) showed a sugar and protein content ranging from 84 to 105 g/kg and from 108 to 138 g/kg, respectively (Kirk-Othmer, 1982). Barley malt has a sugar content significantly higher than in nonmalting

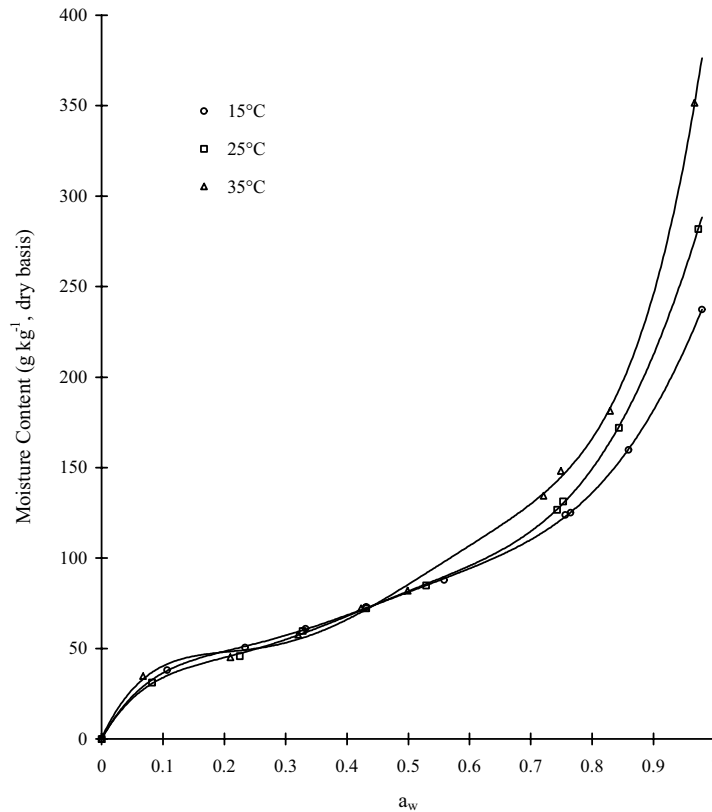


Fig. 1. Sorption isotherm of six-row Belgian barley malt for a temperature range from 15°C to 35°C.

barley. On the other hand, enzyme activity increases with increased a_w and substrate mobility. It is also possible that the endothermic dissolution of sugars at elevated a_w values could occur with increased substrate mobility along with an increase of active sites for the sorption of water by proteins (Sá & Sereno, 1993; Hermansson, 1977). The increase in simple sugars in barley malt due to the malting process can explain the behavior observed in this research work for the sorption isotherm, in contrast with the normal behavior found in nonmalting cereals, according to the sorption data for cereal grains reported by Brooker, Bakker-Arkema, and Hall (1974) and Iglesias and Chirife (1982).

The data obtained were adjusted to various sorption models at the temperature studied (15°C, 25°C and 35°C), in order to determine the best model to predict the sorption isotherm for the product studied. The results obtained are presented in Table 1. The constants obtained for each linearized equation and the correlation coefficients (R^2) are presented in this table. All models showed correlation coefficients above 0.919.

The models of BET and Kuhn ($a_w < 0.50$) showed the highest correlation coefficients ($R^2 > 0.997$). For the range of a_w above 0.50, the isotherms of Harkins–Jura, Smith and Henderson showed excellent fittings ($R^2 > 0.994$). On the other hand, long-range models such as Freundlich ($a_w < 0.90$), Chung–Pfoest ($0.20 < a_w < 0.90$)

and GAB ($a_w < 0.95$) also showed good fittings ($R^2 > 0.919$).

Considering that the practical storage moisture content of barley malt is below 60 g/kg, with a typical moisture content ranging from 35 and 40 g/kg (Broderick, 1977), the BET model ($a_w < 0.5$) would represent the best one to apply from the practical viewpoint. Malt with higher moisture content is not suitable for brewing purposes.

The monolayer water content was calculated from the linearized BET equation and the nonlinear GAB model presented in Table 1. The calculated monolayer moisture content was between 42.5 and 43.9 g/kg (d.b.), using the BET model and between 46 and 51 g/kg (d.b.), using the GAB model, for the range of temperature considered in this work (15–35°C). These values corresponded to a_w values ranging from 0.16 to 0.19, according to the BET equation and from 0.19 to 0.21 using the GAB equation.

The sorption enthalpy (Q_s) was calculated by the equation presented by Chirife, Suárez, and Iglesias (1986) that relates the constant C with this parameter and the absolute temperature (T).

$$Q_s = RT \ln C \quad (1)$$

The value of C was calculated from the BET equation adjusting all the sorption data for the three temperatures

studied (15°C, 25°C and 35°C) to a single isotherm. Excellent fitting was found, with a correlation coefficient $R^2 = 0.997$. The monolayer value obtained was 43.5 g/kg (d.b.) and $C = 23.1718$. The value of C calculated was introduced into Eq. (1) to calculate the sorption enthalpy. Sorption enthalpy values of 7.526, 7.787 and 8.049 kJ/gmol were obtained for the temperatures of 15°C, 25°C and 35°C, respectively.

In the same way, the total heat requirement in this process (ΔH_T) was calculated with the following equation (Labuza, Kaanane, & Chen, 1985):

$$\Delta H_T = Q_s + \Delta H_v \quad (2)$$

The latent heat of vaporization ΔH_v was determined from the standard steam tables (Keenan & Keyes, 1965). The calculated values ranged from 51.599 to 52.136 kJ/gmol with an average value of 51.834 ± 0.275 kJ/gmol.

4. Conclusions

Sorption isotherms of six-row Belgian barley malt (Plaisant) were determined for a temperature range between 15°C and 35°C. Experimental data indicated that the effect of temperature on the sorption characteristic of this material was negligible for a_w values below 0.5. For a_w values greater than 0.5, an increase of the equilibrium moisture content with temperature at a given a_w was observed. This behavior has been found by other authors in products rich in carbohydrates. Due to the malting process, barley malt presents elevated enzyme activity that results in the breakdown of complex carbohydrates into simple sugars. For this reason, the sugar content of barley malt is significantly higher than that of nonmalting barley. Also, it is known that enzyme activity increases with increased a_w due to substrate mobility. In this manner, an increased sugar content and enzyme activity in barley malt could explain the sorption behavior observed in this research work for a_w values above 0.50.

Fitting of the experimental data to different sorption models at the temperatures studied (15°C, 25°C and 35°C) was carried out. Although all models showed good fitting to the experimental data, the BET and Kuhn models presented the highest correlation coefficients for a_w below 0.50. For the range of a_w above 0.50, the isotherms of Harkins–Jura, Smith and Henderson showed excellent fittings, while other wide a_w range models such as Freundlich, Chung–Pfoest and GAB also showed good fittings. For practical purposes any of them could be used satisfactorily. However, since the practical storage moisture content of barley malt is below 60 g/kg, the BET model would represent the best one to apply from the practical viewpoint.

The monolayer water content calculated from the BET and GAB equations ranged between 42.5 and

43.9 g/kg (d.b.), and 46 and 51 g/kg (d.b.), respectively for the range of temperature considered in this work (15–35°C). These values corresponded to a_w values ranging from 0.16 to 0.19, according to the BET equation and from 0.19 to 0.21 using the GAB equation.

The average sorption enthalpy (Q_s) and the total heat requirement (ΔH_T) of this process for the temperature range studied (15–35°C) showed values of 7.787 ± 0.262 and 51.834 ± 0.275 kJ/gmol, respectively.

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