Estimation of Moisture Sorption Isotherms of Mango Pulp Freeze- dried.

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***Abstract*—**The static gravimetric method, along with different saturated salt solutions, was used in a range of water activity (a_w) of 0.11–0.88, and at 288.15, 298.15 y 308.15 K, to obtain sorption isotherms of freeze-dried pulp of mango. At a given a_w the results show that the moisture content decreases with increasing temperature. BET, G.A.B, Halsey, Henderson, Oswin and Smith were evaluated to determine the best fit for the experimental data. A nonlinear regression–analysis method was used to evaluate the constants of equations. The BET and GAB models were the most suitable to described the sorption curves; the monolayer-content values for the sorption at different temperatures are calculated. The isosteric sorption heat was determines for adsorption of freeze-dried pulp of mango using the relevant thermodynamic relations (i.e., Clausius Clapeyron equation).

***Keywords*—** Mango, sorption isotherm, sorption models, isosteric heat of sorption, water activity.

I. Introduction

The production of mango ranks third among the tropical fruits, the seventh largest in the whole fruit, but in terms of consumption, mango ranks first worldwide [1] derived from fresh consumption. This fruit is an excellent source of vitamin A and C, as the consumption of 100 g of ripe mango will cover more than 50% of the recommended daily amount of each of these vitamins [2]. This fruit provides a certain amount of other vitamins and minerals such as riboflavin, niacin, calcium, phosphorus and iron [3].

Fresh fruits and vegetables are important components of human food, occupying the second place in the food pyramid [4]. Today, consumers demand products to market that are identical to the fresh, to preserve their own physicochemical properties and durability, without reducing the properties of food. Most of foods have a substantial quantity of water, which is critical for the disqualification or dissemination of the different reactions. Too High moisture content lead to micro organism growth and low quality [5] The food industry have developed a number of techniques for conservation, the mango is consumed in many forms and products, has a shelf life of approximately 16 weeks, is subject to various diseases in all stages of development, consequently is the interest to maintain its organoleptic properties as long as possible [6, 7, 8]. Changes in surface and flesh colours during maturation seem useful as a good index of fruit maturity and determination factors of optimal harvesting time. Mango fruits ripen unevenly on the tree and fruits are picked by hand at an average maturity. Tree ripe fruits show bright skin colour with uniformly softened flesh and developed flavor, but those fruits have a very short shelf life [9]. It was used freeze-drying technology for the production of mango pulp that keep the most organoleptic characteristics of fresh product, easy distribution and storage.

The water content in a food is particularly important from a nutritional standpoint. Also, is interesting how the water is present. The availability of water, will determine the shelf life. In fact, foods with the same water content may have different changes because they have a different availability [10]. The extent to which the water interacts with the components contributes to the stability of a food is determined by the amount that is present and is defined by the activity of water and its thermodynamic state [11].

The values of equilibrium moisture content of biological products depend mainly on the temperature and relative humidity of the species or variety of the product. Physiological maturity and history of the product, and how was the equilibrium obtained (adsorption or desorption), also influence the equilibrium moisture content [12].

Water activity is the main factor of numerous important food processing operations, such as microbial growth, toxin formation, enzymatic and non-enzymatic reactions. It is the availability of water for microbial, enzymatic or chemical activity that determines the shelf life of Food, also is a function of moisture content in food and temperature [13].The relation-ship between total moisture content and a_w for a meal in a range of values at a constant temperature, produce a moisture sorption isotherm when expressed in graphical form [14]. They

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relate the equilibrium moisture content (g water / g dry matter) at a constant temperature with the thermodynamic activity of water in the product at a given range of moisture or activity [15]. To determine, more quickly, the optimum moisture content required for the stability of the dehydrated product, it is useful for producers to decrease microbial growth, enzymatic reactions, growth and non-enzymatic oxidation of lipids [16].

A number of models have been suggested in the literature for the dependence between the equilibrium moisture content and the water activity (a_w) [17]. Some of them take into account the effect of temperature. The Oswin (1946), Smith (1947), Halsey (1948), Henderson (1952) and the Guggeheim-Ander-son de Boer (GAB) equations have been adopted as standard equations by The American Society of Agricultural Engineers (ASAE) for describing sorption isotherms [17]. Among the sorption models, the GAB [18] equation has been applied successfully to various food materials. The heat of sorption is the amount of energy given when sorption occurs indicates the strength of bond between sorbate and sorbent [19]. This heat can be determined from sorption isotherms determined at different temperatures. During an adsorption process this heat is called the isosteric heat of sorption (Q_S) and is a direct function of temperature and moisture adsorption capacity and represents the heat involved in the phenomenon of adsorption and the temperature change (heat transfer system) [18, 20].

The aim of this study was to determine the sorption isotherms of mango pulp freeze-dried, the type of isotherm between a_w values of 0.1 to 0.89 to determine the effect at 288.15, 298.15 and 308.15 K, and determined the isosteric heat for each temperature range storage. The experimental data set were determined and correlations were calculated applying different theoretical models or semi-theoretical to predict the appropriate conditions for storage and greater stability.

II. Materials and Methods

A. Raw Material

It was used the variety of Ataulfo mango (*Mangifera Indica L.*), bought in a supermarket in Puebla city. The Pulp was separated from the bone and shell, and then was liquefied in a food processor BRAUN brand. Total acidity (%), total soluble solids (°Brix), pH Value, were determined by methods reported in the literature as initial analysis of mango pulp [21]. The results are as follows: 0.3479% ± 0.0115 acidity expressed as citric acid, 20.60 ° Bx, pH: 4.42 ± 0.070, then stored in petri dishes of 20 cm x 1 cm for subsequent freezing cabinet at 233.15 K (REVCO, Model ULT 2090-3 ABA, USA). Frozen mango pulp was lyophilized in a 51-SRC team sublimator VirTis (New York) with a gap of 0.1-0.2mm Hg and a condenser temperature of 218.15 K.

B. Sorption Isotherm

The initial moisture content of mango pulp freeze-dried was determined according to 22.013 AOAC [21] obtaining 0.2950 ± 0.0246 Kg water / Kg dry solid. The equilibrium moisture content of freeze-dried mango pulp at different temperatures

TABLE I
Mathematical models applied to sorption isotherms of mango pulp freeze-dried

Reference	Model	Equation No
B. E. T. (1938) [24]	$M_w = \frac{ABa_w}{(1-a_w)[1+(B-1)a_w]}$ $A = m_0$ $a_w < 0.5$	(1)
G. A. B. (1966) [18]	$M_w = \frac{A\ B\ C\ a_w}{(1-C)(1-C\,a_w + B\ Ca_w)}$ $0.10 < a_w < 0.90$	(2)
Halsey (1948) [25]	$M_w = \left(\frac{-A}{\ln a_w}\right)^{1/B}$ $0.10 < a_w < 0.80$	(3)
Henderson (1952) [26]	$M_w = \left[-\frac{\ln(1-a_w)}{A}\right]^{1/B}$ $0.5 < a_w < 0.95$	(4)
Oswin (1946) [27]	$M_w = \exp\left(A + B\ln\left[a_w/1-a_w\right]\right)$ $a_w < 0.57$	(5)
Smith (1947) [28]	$M_w = A + B\ln(-\ln a_w)$ $0.06 < a_w < 0.495$	(6)

a_w: Water Activity $A = m_0$, $B = c_{constant}$, $C = c_{constant}$, M_w =: Equilibrium moisture content (Kg water / Kg dry solid)

was determined by the gravimetric - static method. The method is based on measuring triplicate samples 1g (±0.1 g); they were placed in glass jar inside a constant relative humidity [22] Figure 1. Ten reagent saturated salt solutions were prepared according to the values of water activity from 0.11 to 0.89; the method of preparation was the same as that adopted in the COST 90 [23].

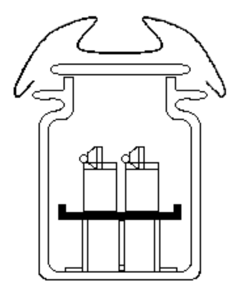

Fig. 1 System used for the determination of Sorption Isotherms [22].

C. Modelling

The experimental data were modeled with the equations of BET (Brunauer, Emmett and Teller), GAB (Guggenheim, Anderson and de Boer), Halsey, Henderson, Oswin and Smith, presented in Table I, using Microsoft Office Excel 2003 software for resolution.

The models were evaluated in terms of statistical error by the percentage of the root mean square (% RMS), which predicts the differences between the experimental and predicted moisture content, the residual must be less than 10% to consider the model as acceptable and states:

$$\% \ RMS = \left(\sqrt{\frac{1}{n} \sum_{i=1} \left(\frac{M_{\exp} - M_{pre}}{M_{\exp}} \right)^2} \right) 100 \qquad (7)$$

Where n is the number of observations, M_{exp} is the experimental value of equilibrium moisture content, M_{pre} represents the predicted value of equilibrium moisture content [29].

D. Isosteric Heat of Sorption

Isosteric heat of sorption (Q_S) for a given temperature range is calculated from the Clausius-Clayperon:

$$Q_{st} = R\left[\frac{T_1 T_2}{T_2 - T_1} \ln(a_w) \right] \qquad (8)$$

Where T is the temperature of study (K), a_w refers to water activity, R is the universal gas constant (8.314 J mol / K) [30].

III. RESULTS & DISCUSSION

A. Experimental Results

The hygroscopic equilibrium for mango pulp freeze-dried is attained within 30 - 35 days for the three storage temperatures. Sorption isotherms obtained at 288.15, 298.5 and 308.15 K are shown in Figure 2. It is noted that the increased moisture in equilibrium is analogous to increasing water activity; however the moisture content decreases with temperature. The results showed the typical shape of the sorption isotherms for foods with high sugar content corresponding to a sigmoidal type III isotherm. Similar results were found for the isotherms of mango and apple African [31]. The characteristic shape of the isotherms obtained will depend on the variety and total hygroscopic material present in the heterogeneous mixture of hydrophilic substances. Characterized in that, at low water activities, there is an increase in moisture content indicating a tightly bound physical sorption on active sites of the biopolymer. As a_w increased, the sugar molecules become more mobile in the internal phase, resulting in a crystallization of the same. Then the crystallization process results in an increase of adsorption of water because the water-sugar interaction is sufficient to cause a dissociation-sugar, allowing active sorption [32].

TABLE II
ESTIMATED PARAMETERS OF DIFFERENT MODELS FOR THE SORPTION ISOTHERMS OF MANGO PULP FREEZE-DRIED.

Parameters	**Temperature (K)**		
	288.15	**298.15**	**308.15**
B.E.T			
A	0.1597	0.1358	0.1284
B	-12.3739	-19.5386	-18.1585
R^2	0.9615	0.9706	0.9852
%RMS	2.3724	3.3736	2.1734
G. A. B.			
A	0.1686	0.1242	0.1175
B	0.9376	0.9399	0.9363
C	15.0253	15.9943	15.1964
R^2	0.9622	0.9000	0.9074
% RMS	1.1311	1.5851	1.6458

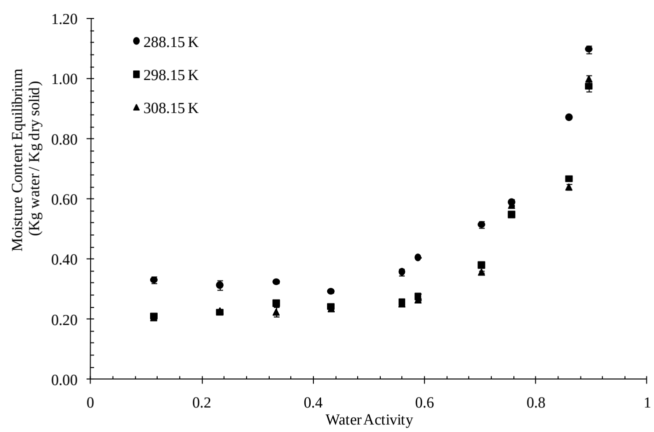

Fig.2 Sorption isotherms of mango pulp freeze-dried at 288.15, 298.15, and 308.15 K.

B. Modeling of the Sorption Isotherms

A large number of models have been proposed to determine sorption isotherms. In this paper we proved the following models: BET (1938), GAB (1966), Smith (1947), Henderson (1952), Oswin (1946), Halsey (1948). These relate the water activity (a_w) and moisture content equilibrium. From the experimental data it is possible to determine the theoretical moisture stability to the lyophilized product. Models that predict the moisture content of mango pulp freeze-dried for the three storage temperatures are: BET and G. A. B. See Table II, which boast correlation coefficients (R2) and values less than 0.90% RMS, 10% respectively according to the criteria established [29, 33, 34]. The G. A. B model is the most widely used for foods with high sugar content such as the mango [35].

Figures 3 and 4 shows the experimental data fit to the BET and GAB model respectively, yielding sigmoidal type III isotherm. In both models, the curves show a kinship own food with high sugar content, with equilibrium moisture content increased abruptly when exposed to high water activities, due to the dissolution of fruit sugar.

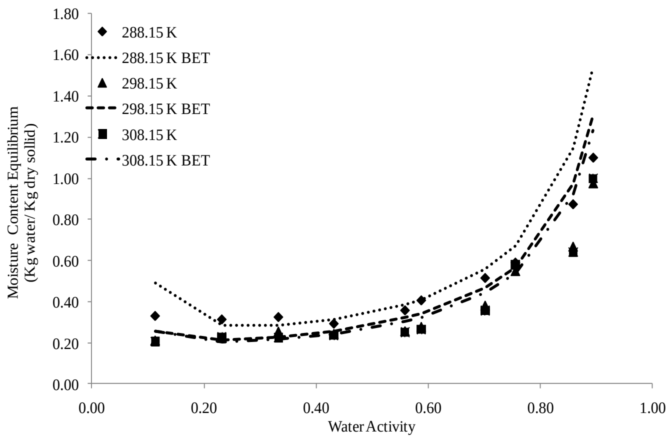

Fig. 3 Influence of Temperature of the Sorption Isotherms of Mango Pulp Freeze-dried, using BET Model.

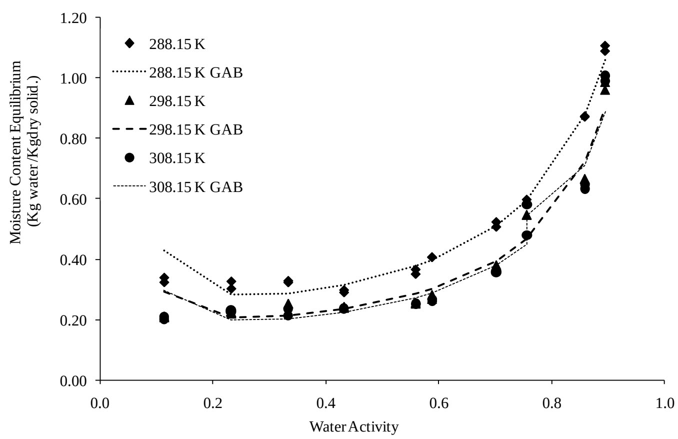

Fig. 4 Influence of Temperature of the Sorption Isotherms of Mango Pulp Freeze-dried, using GAB model

The values obtained for the monolayer mango pulp freeze-dried according to the BET model were 0.1597, 0.1358 and 0.1284 for the GAB model are: 0.1686, 0.1242, and 0.1175 (kg water / Kg dry solid) at 288.15, 298.5 and 308.15 K respectively. It was observed that monolayer values decrease with temperature. This decrease in the monolayer may be due to a reduction in the total number of active sites to be covered, as a result of a physical and / or chemical change induced by temperature. Similar results have been reported for mango yogurt powder [29].

It was found that the value of the second constant for the GAB model related to the effects of temperature (C) had values greater than 1, indicating that physical disturbance and size reduction when the product is dried.

C. Isosteric Heat of Sorption (Qs)

The determination of isosteric heat of sorption is a valuable tool for understanding the water sorption mechanism. It can be defined as the amount of energy required to remove water from the substrate in excess of the amount of energy required for normal or free water vaporization [36]. Figures 5, 6 and 7 shows the behavior of isosteric heat for mango pulp freeze-dried using the Clausius-Clayperon intervals depending on temperature and moisture content in dry basis using the GAB model.

According to the results shown in Figures 5 and 6 and comparing with the results provided for a Tunisian fruit [37], very high Qs values indicate the presence of active polar sites on the surface of food material , which is covered by water molecules on the monolayer. The results for the ranges of 298.15-288.15 K and 298.15-308.15 K reach 28307.2912 kJ / mol and 29265.3467 kJ / mol respectively, these values suggest Qs physical sorption of water on polar groups of biopolymers through bridges hydrogen and Van der Waals forces.

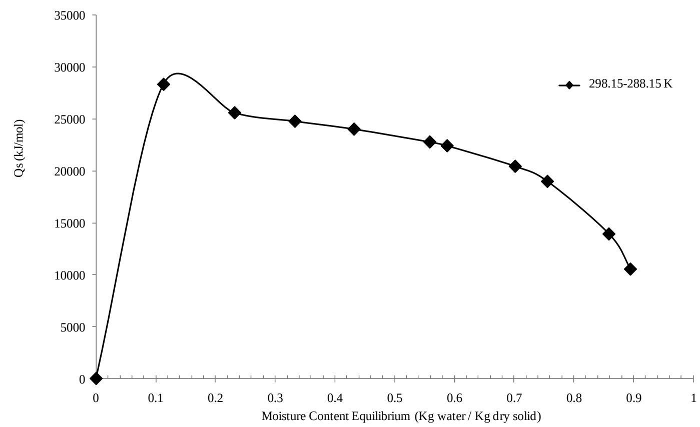

Fig. 5 Isosteric Heat of Sorption of Mango Pulp Freeze-dried for temperature range of 298.15-288.15°C

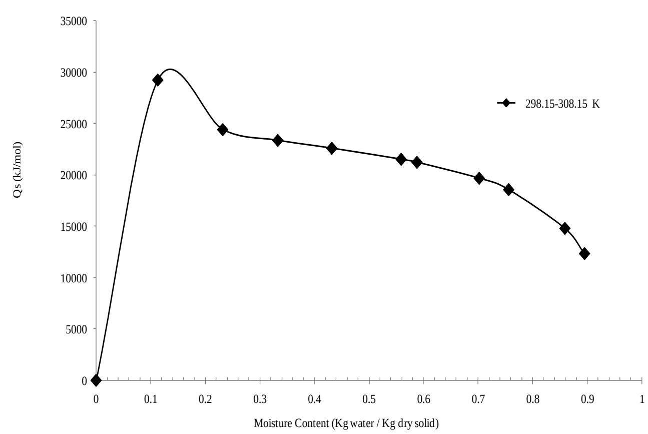

Fig. 6 Isosteric Heat of Sorption of Mango Pulp Freeze-dried for temperature range of 298.15-308.15°C

Figure 7 shows the behavior of isosteric heat for the range of 288.15-308.15 K, producing values of 1494.05 kJ / mol, compared to the study for a variety of different mango [38], it is observed that when moisture content increases, the available sites to suck the water are reduced, resulting in very small values of Qs, giving rise to constant rearrangements in the structure by the water and sugar molecules within fruit.

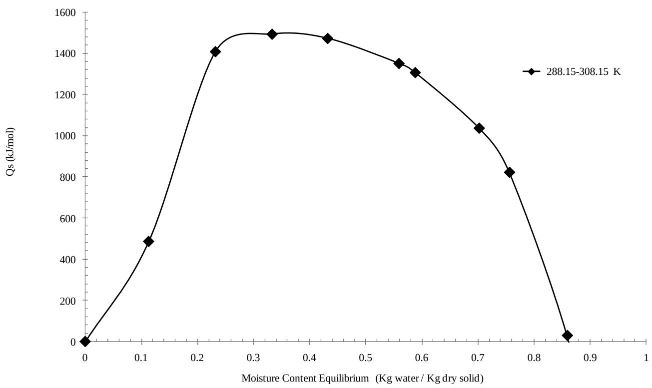

Fig. 7 Isosteric Heat of Sorption of Mango Pulp Freeze-dried for temperature range of 288.15-308.15 K

IV. CONCLUSION

The sorption curves provide valuable information about the hygroscopic equilibrium mango pulp freeze-dried. They give a clear idea of the stability domain of this product after freeze - drying. The sorption isotherms mango pulp freeze-dried were analyzed for six models to predicting the equilibrium moisture content. The comparison between the experimental points and predicted sorption isotherms showed that the GAB and BET models seems to be the most suitables to describe the sorption isotherms mango pulp freeze-dried in a temperature range 288.15–308.15 K and a_w values from 0.11– 0.89. The equilibrium moisture content and values of monolayers obtained by BET and GAB models decreased as temperature increased, which is related to the isosteric heat of sorption (Qs), confirming the existence of physical sorption of water on the polar groups of the biopolymers (sugars) through hydrogen bonds and Van der Waals forces. The constant C of G. A. B. is greater than unity indicating that there was impairment of water sublimated by the migration of the sugar molecules to occupy the available sites. For Mango pulp freeze-dried , the optimal storage temperature is 308.15 K and a relative humidity of 60% due to the equilibrium moisture content is maintained around the values of monolayer.

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