

MOISTURE SORPTION ISOTHERMS OF ORGANIC SOY PROTEIN (NGMO)

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Abstract. In this paper, the sorption isotherms for the organic soy protein were determined at 10, 20, 30, 40°C using the standard static gravimetric method developed by the European Cooperation Project COST 90. These curves have an important role in the design and the optimization of the unit operations such as preservation, drying, storing, packaging and mixing. Ten different salts are used in order to maintain the relative humidity created of the environment constant. Crystalline thymol is placed in a recipient in the hygrostats to prevent microbial spoilage at high water activities ($a_w > 0,7$). A number of models has been suggested in the literature for the dependence between the equilibrium moisture content and the relative humidity of the air. The description of the relationship between equilibrium moisture contents, water activities and temperature is verified according to three different groups of models: the first one is not dependent on the temperature it is function of two parameters; the second is not dependent on the temperature it is function of three parameters and the third group that is function of more than three parameters. The results have showed that the models from Oswin, Anderson, Anderson-Hall, BET, Chirife *et al.*, Ferro Fontain, GAB presented the best fit, so that mean relative deviation modulus $E < 10\%$.

Keywords: isotherms, sorption, organic soy.

1. Introduction

The soybean has a high protein content (~40%) and relatively high oil content (~20%) on a dry weight basis in comparison with other legumes. Its high protein level and well-balanced amino-acid composition makes the soybean an important source of proteins, with potential to replace meat and dairy products, if necessary. At the moment less than 1% of the world soybean production is being used for human food; the rest of it is used for animal feed. In the future, the necessity for vegetable proteins is expected, especially in areas where population growth is high. In addition, the recently accepted health claim that soy protein helps to reduce the blood cholesterol level will increase market growth of soy foods in western countries. In order to improve the use of soy proteins in food products, basic knowledge on their functional properties under food conditions is required.

Soybeans are used to make traditional soy foods like tofu, tempeh, soymilk, oil and defatted meal, among others. The meal is mainly used as animal feed; a small portion, in growth, is further processed into food ingredients including, soy flour, concentrates, isolates and textured protein. These ingredients can be used as emulsifiers, foaming agents, and texture-enhancers in foods or have nutritional value in new soy foods like soy burgers and soy cheese (Renkema, 2001).

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Analysis of processes affecting physical, biochemical, and microbiological stability of foods, which in turn determine their quality, is largely based on moisture sorption isotherm data of the materials involved. They are important in design and optimization of unit operations such as preservation, drying, storing, packaging, and mixing (Tsami *et al.*, 1990). Knowledge of the sorption characteristics of food is important in both the design of drying processes and equipment and in studies of shelf-life and packaging requirements of dehydrated products. (Johnson and Brennan, 2000)

The relationship between water activity and the moisture content in a product is often expressed as a sorption isotherm. The typical shape of an isotherm reflects the manner in which the water is bound to the system. Up to water activity of about 0.30, water is considered to be held on polar sites of relatively high energies. This is called the monolayer. The water activity in the range of 0.30–0.70 is referred to multilayer water; it consists of layers of water, which are adsorbed onto the first layer by hydrogen bonds. Above a water activity of 0.7, the water approaches the condition of “condensed water” and it is relatively free water and the isotherm reflects solution and surface capillary effects (Wolf, Spiess, & Jung, 1985 apud Lahsasni *et al.*, 2002).

In the literature, just a few works deal with isotherms for soy to Riganakos *et. al* (1994) accomplished the comparison of three methods (gravimetric (static), inverse gas chromatographic (IGC) and computerized inverse gas chromatographic (CIGC)) for obtaining the isotherms of wheat flour and soy only for the temperature of 30°C. Good agreement was observed between sorption isotherms for wheat and soy flour obtained with the IGC and static methods. Differences between the two methods were in the order of 7,5% for wheat flour and 12 % for soy flour.

Several empirical and semi-empirical equations have been proposed for the correlation of the equilibrium moisture content of food materials (Iglesias and Chirife, 1995). Numerous models for predicting the relationship between equilibrium moisture, water activity and temperature have been developed. The main objective of the present work is obtain experimental equilibrium sorption isotherms and find out the most suitable model describing the isotherms.

2. Materials and methods

2.1 Materials

The extract of protein of organic soy was obtained from the aqueous emulsion of the integral grains of soy, submitted to appropriate thermal treatments for denaturation of the anti-nutritional factors. The soluble fraction is concentrated and afterwards dried in a of spray-drying operation. This fraction is defined as Provesol SM-N obtained from OLVEBRA INDUSTRIAL S/A, Eldorado do Sul – Brazil.

2.2 Experimental procedure

The standard sorption apparatus recommended by COST 90 Project (Wolf *et al.*, 1985) was used for equilibrium studies isotherms of protein of organic soy. It consisted of sorption containers and support for weighing dishes in which the samples were exposed to humid atmosphere in the containers. Ten analytical reagent grade salts were used for preparation of salt slurries. The salts were dissolved in distilled water at 100°C and cooled to test temperatures (10°C, 20°C, 30°C and 40°C) for crystallization to form saturated solutions. Water activities of saturated salt solutions thus prepared were presented in the table 1. The sorption apparatus

were allowed to equilibrate at respective temperatures for 24 hours before the samples were placed into them. Weighed quantity of the test samples (4 ± 0.05 g) was taken into tared sample dishes, which were then transferred to the apparatus. Samples were weighed at regular intervals (after every 72 h) and the equilibrium was judged to have been attained when the difference between the three consecutive weighings did not exceed 1 mg. The total time for removal, weighing and replacing the samples did not exceed 30 s. Average of duplicate measurements was used to develop the moisture sorption isotherms (data from triplicate measurements were not significantly different). At high water activities ($a_w > 0,7$) crystalline thymol was placed in the hygrometers to prevent microbial spoilage. After reaching equilibrium, the samples were taken to a temperature of 105°C for 24h in order to obtain their dry mass (AOAC, 1990). The difference of mass before and after drying in the oven determines the moisture content of the product at hygroscopic equilibrium (X_{eq}).

Table 1. Relatively humidity of saturated salt solutions in the four temperatures:

salt	Relatively humidity			
	10°C	20 °C	30°C	40°C
NaOH	0.102	0.089	0.0760	0.063
LiCl	0.113	0.110	0.108	0.106
CH ₃ COOK	0.246	0.280	0.220	0.385
MgCl ₂	0.335	0.331	0.325	0.316
K ₂ CO ₃	0.432	0.432	0.432	0.432
Mg(NO ₃) ₂	0.675	0.655	0.635	0.615
NaCl	0.757	0.753	0.743	0.729
KCl	0.868	0.851	0.836	0.823
BaCl	0.914	0.907	0.900	0.893
Cu SO ₄	0.978	0.973	0.968	0.962

Source: Greenspan, 1977.

2.3 Data analysis

A Nonlinear Estimation (MatLab 5.3 Math Works Inc.) was used to estimate the constants of the equilibrium moisture models (presented in Tables 1, 2 and 3). To confirm that the regression parameters were in deed unique, the regression was repeated with various initial guessed values above and below those calculetd (Peleg, 1993, apud Figueira et al 2004). The criterion used to evaluated the quality of the fit was the mean realitive deviation modulus (E):

$$E = \frac{100}{n} \sum \frac{|Y_{exp} - Y_{calc}|}{Y_{exp}} \quad \text{Eq. 1}$$

Where n is number of the experimental data, Y_{exp} is the experimental value, Y_{calc} is predicted value for the model. It is generally considered that E values below 10% indicate an adequate fit for pratical purpose (Aguerre et al. apud Figueira *et al.* 2004, Bassal *et al.*, 1993, Prado *et al.*, 1999, MacLaughlin & Magee, 1988). Besides this method of evaluating the estimate others that we can mention exist such as: the value of root mean square error

(RMSE) (Hossain *et al.*, 2001); the root mean square percent error (RMS%) and residuals (R^*) (Kumar *et al.*, 2005).

2.3. Modelling equations

A number of models have been suggested in the literature for the dependence between the equilibrium moisture content and the relative humidity of the air. The equations without dependence of the temperature, models bi-parametrics fifteen models were tested: Bradley, Caurie, Day & Nelson, Freundlich, Iglesias and Chirife, Halsey, Halsey simplified, Harkins & Jura, Henderson, Kuhn, Mizrahi, modified Mizrahi, Oswin, Smith, White and Eiring; models tri-parametrics fourteen models were tested: Anderson, Anderson-Hall, BET, Chen, Chirife et al. Ferro-Fontan, GAB, Gascoyne-Pethig, Hailwood & Horrobin, Modified Halsey, Haynes, Lewicki, Polynomial, Young & Nelson; and models tetra e penta-parametrics: Peleg and D'Arcy-Watt were used to predict experimental data for water activity range 0-0.95. Among the models mentioned previously are presented in the tables 1, 2 and 3, the ones that obtained the best behavior in the estimate nonlinear of the parameters.

Table 2. Non-temperature dependency models bi-parametrics for fitting the sorption isotherms of soy protein NGMO:

Name of the model	Model	
Simplified Halsey (Iglesias & Chirife, 1976, apud: Figueira et al 2004))	$X_{eq} = \left(-\frac{A}{\ln a_w} \right)^{1/n}$	Eq. 2
Halsey (Castillo <i>et al.</i> , 2003)	$X_{eq} = \exp[a + b \ln(-\ln a_w)]$	Eq. 3
Harkins & Jura (Castillo <i>et al.</i> , 2003)	$X_{eq} = \sqrt[3]{a + b \ln a_w}$	Eq. 4
Henderson (Castillo <i>et al.</i> , 2003)	$X_{eq} = \exp[a + b \ln(-\ln(1 - a_w))]$	Eq. 5
Modified BET (Hossain <i>et al.</i> , 2001)	$X_{eq} = a/(1 - ba_w)$	Eq. 6
Mizrahi (Castillo <i>et al.</i> , 2003)	$X_{eq} = A[a_w/1 - a_w] + B(1 - a_w)^{-1}$	Eq. 7
Oswin (Castillo <i>et al.</i> , 2003)	$X_{eq} = \exp(a + b \ln[a_w/(1 - a_w)])$	Eq. 8
Oswin (Lomauro, Bakshi, & Labuza, 1985, apud Figueira et al 2004))	$X_{eq} = A \left(\frac{a_w}{1 - a_w} \right)^B$	Eq. 9
Smith (Sandoval and Barreiro, 2002, Al-Muhtaseb <i>et al.</i> 2004)	$X_{eq} = A + B \ln(1 - a_w)$	Eq. 10
White e Eiring (Castillo <i>et al.</i> , 2003)	$X_{eq} = 1/(a + ba_w)$	Eq. 11

Where A, n, B, a, b are constants and a_w is water activity.

Table 2. Non-temperature dependency models tri-parametrics for fitting the sorption isotherms of soy protein NGMO:

Name of the model	Model	
Anderson (Boquet et al., 1980)	$X_{eq} = \frac{ABCa_w}{[1 + (B - 2)Ca_w + (1 - B)C^2a_w^2]}$	Eq. 12
Anderson-Hall (Boquet et al., 1980)	$X_{eq} = \frac{ABa_w}{[1 + (B - 2C)a_w + (C^2 - BC)a_w^2]}$	Eq. 13
BET (Brunauer, Emmett, & Teller, 1938, apud: Figueira et al 2004)	$X_{eq} = \frac{(X_m Ca_w)[1 - (n + 1)a_w^n + na_w^{n+1}]}{(1 - a_w)[1 + (C - 1)a_w]}$	Eq. 14
BET linear (Brunauer et al., 1938, apud: Figueira et al 2004))	$X_{eq} = \frac{X_m Ca_w}{(1 - a_w)(1 + (C - 1)ka_w)}$	Eq. 15
Chen (Castillo et al., 2003)	$X_{eq} = A + B \ln(-\ln a_w + C)$	Eq. 16
Chirife et. al. (Castillo et al., 2003)	$X_{eq} = \exp[a + b \ln(c - \ln a_w)]$	Eq. 17
Ferro-Fontan (Iglesias & Chirife, 1995 and Al-Muhtaseb et al. 2004):	$X_{eq} = \left[\frac{\gamma}{\ln(\alpha/a_w)} \right]^{1/r}$	Eq. 18
GAB (van der BERG, 1984, apud: Figueira et al 2004, Tsami et al. 1990)	$X_{eq} = \frac{CKX_m a_w}{(1 - Ka_w)(1 - Ka_w + CKa_w)}$	Eq. 19
Modified Halsey (Iglesias e& Chirife, 1976 apud: Figueira et al 2004)	$X_{eq} = X_m \left(\frac{-A}{\ln a_w} \right)^{1/n}$	Eq. 20
Modified Mizrahi (Kumar et al., 2005)	$X_{eq} = \frac{a + a_w(ca_w + b)}{a_w - 1}$	Eq. 21

Where A, B, C, n, γ , α , r, K, a, b, c were constants of models and X_m is monolayer moisture.

Table 3. Non-temperature dependency models tetra e penta-parametrics for fitting the sorption isotherms of soy protein NGMO:

Name of the model	Model	
D'Arcy-Watt (Saravacos, 1986)	$X_{eq} = \frac{k_1 k_2 a_w}{1 + k_1 a_w} + k_5 a_w + \frac{k_3 k_4 a_w}{1 - k_3 a_w}$	Eq. 22
Peleg (Peleg, 1993, apud Figueira et al 2004)	$X_{eq} = k_1 a_w^{n_1} + k_2 a_w^{n_2}, n_1 < 1, \text{ and } n_2 > 1$	Eq. 23

Where $K_1, K_2, K_3, K_4, K_5, n_1$ and n_2 were constants of models.

3. Results and discussion

The Tables 4, 5 and 6 present the values for the constants for each model and the mean relative deviation modulus (E). The experimental and predicted values (best fit) are plotted in Figure 1 (non-temperature dependence bi-parameters) and 2 (non-temperature dependence tri-parameters) for the temperature of 10, 20, 30 and 40°C. According to the criteria suggested by Aguerre et al. (1985), the models that presented more best fit are Oswin (Eq. 13) with the mean relative deviation modulus (E) to models bi-parametrics for approximately 7% to 10°C and 20°C, 5% to 30°C and 4% to 40°C; to models tri-parametrics highlight: Anderson, Anderson-Hall, BET, Chirife et al., Ferro Fontain, GAB, among the ones which the with smaller E was it of Anderson-Hall (Eq. 17) with approximately 2% to 10°C, 20°C and 40°C and 4% to 30°C. In the tables 4, 5 and 6 were in prominence in red the values the mean relative deviation modulus (E) smaller than ten percent.

Table 4. Non Parameters values for the non-temperature dependency models bi-parametrics

Model	Parameters	Temperature(°C)			
		10	20	30	40
Simplif Halsey	A	0,0092	0,0080	0,0062	0,0048
	n	2,0058	2,0130	2,0004	2,0641
	E(%)	18,98	17,52	16,53	13,22
Halsey	a	-2,3368	-2,3988	-2,5374	-2,5855
	b	-0,4986	-0,4968	-0,4998	-0,4845
	E(%)	18,96	17,51	16,54	13,22
Harkins e Jura	a	0,3495	0,5064	0,7203	1,4549
	b	-97,6502	-111,571	-146,244	-166,939
	E(%)	21,99	19,29	18,64	13,51
BET modified	a	0,0523	0,0513	0,0459	0,0452
	b	0,9368	0,9274	0,9212	0,9063
	E(%)	8,00	8,52	10,39	10,12
Oswin (2003)	a	0,1155	0,1094	0,0963	0,0916
	b	0,4496	0,4427	0,4394	0,4211
	E(%)	7,08	7,39	4,97	4,06
Oswin (2004)	A	-2,15809	-2,2126	-2,3402	-2,3898
	B	0,4496	0,4427	0,4394	0,4210
	E(%)	7,08	7,39	4,97	4,06
Smith	A	0,0060	0,0128	0,0156	0,0211
	B	-0,1511	-0,1338	-0,1125	-0,0973
	E(%)	19,29	14,18	11,75	6,46
White & Eiring	a	19,0919	19,4794	21,7427	22,1437
	b	-17,887	-18,0658	-20,0306	-20,0686
	E(%)	8,00	8,52	10,39	10,12

Table 5: Parameters values for the non-temperature dependency models tri-parametrics

Model	Parameters	Temperature(C)			
		10	20	30	40
Anderson	A	0,0551	0,0551	0,0496	0,0485
	B	20,5789	16,9778	21,0750	26,5039
	C	0,9324	0,9200	0,9122	0,8960
	E(%)	2,63	4,05	1,53	1,75
Anderson-Hall	A	0,0547	0,0540	0,0505	0,0485
	B	23,9944	24,2174	14,5272	24,0289
	C	0,9331	0,9221	0,9101	0,8961
	E(%)	1,78	1,90	3,64	1,80
BET aw<0.5	Xm	0,0554	0,0502	0,0474	0,0465
	C	21,0551	31,7635	22,5658	24,6133
	n	20,5070	26,0000	23,9105	50,0000
	E(%)	2,43	2,36	1,62	0,92

Continuation Table 5: Parameters values for the non-temperature dependency models tri-parametrics

Model	Parameters	Temperature(C)			
		10	20	30	40
Chen	A	0,0919	0,0875	0,0782	0,0743
	B	-0,0885	-0,0788	-0,0661	-0,0575
	C	-0,0198	-0,0235	-0,0271	-0,0308
	E(%)	19,65	18,12	15,97	13,18
Chirife et al.	a	-2,5203	-2,5439	-2,6517	-2,6428
	b	-0,7413	-0,7341	-0,7382	-0,6490
	c	0,0407	0,0479	0,0560	0,0461
	E(%)	2,42	2,26	2,59	3,01
Ferro-Fontan	γ	0,0357	0,0328	0,0353	0,0334
	α	1,0450	1,0520	1,0744	1,0932
	r	1,3140	1,3413	1,2509	1,2711
	E(%)	3,41	2,53	4,35	3,83
GAB	X _m	0,0357	0,0328	0,0353	0,0334
	C	1,0450	1,0520	1,0744	1,0932
	K	1,3140	1,3413	1,2509	1,2711
	E(%)	3,41	2,53	4,35	3,83
Modified Halsey	X _m	0,1912	0,2458	0,2221	0,1849
	A	0,2545	0,1348	0,1267	0,1568
	n	2,0055	2,0126	2,0007	2,0643
	E(%)	18,96	17,50	16,54	13,23
Modified Mizrahi	a	-0,0022	-0,0064	-0,0105	-0,0138
	b	-0,2429	-0,2122	-0,1697	-0,1453
	c	0,2360	0,2096	0,1718	0,1513
	E(%)	17,53	15,69	12,91	9,71

Table 6: Parameters values for the non-temperature dependency models tetra e penta-parametrics

Model	Parameters	Temperature(C)			
		10	20	30	40
peleg	k ₁	0,1785	0,1650	0,1323	0,1256
	n ₁	0,6984	0,6728	0,5799	0,5444
	k ₂	0,5702	0,4668	0,3787	0,3067
	n ₂	11,1174	9,7089	8,3668	7,8796
	E(%)	6,32	8,59	3,94	3,96
Darcy Watt	k ₁	-1,7296	-0,1782	-0,4268	-0,2027
	k ₂	-0,0039	-0,3398	-0,3473	-0,4637
	k ₃	0,9560	0,9669	1,0331	0,9794
	k ₄	0,0334	0,0226	0,0000	0,0117
	k ₅	0,1572	0,0981	0,0198	0,0509
E(%)	14,50	16,26	21,04	17,10	

It was certain the monolayer moisture for the models of BET, GAB and modified Halsey presents in table 5. Monolayer moisture contents determined for the model BET were found to decrease with increasing temperatures. For the model of GAB the monolayer moisture didn't present a significant difference, being on average 0,034 with standard deviation of 0,001. The parameters adjusted by the model of Modified Halsey don't represent a physical value for X_m, due to the fact of the mean relative deviation modulus (E) above 10%.

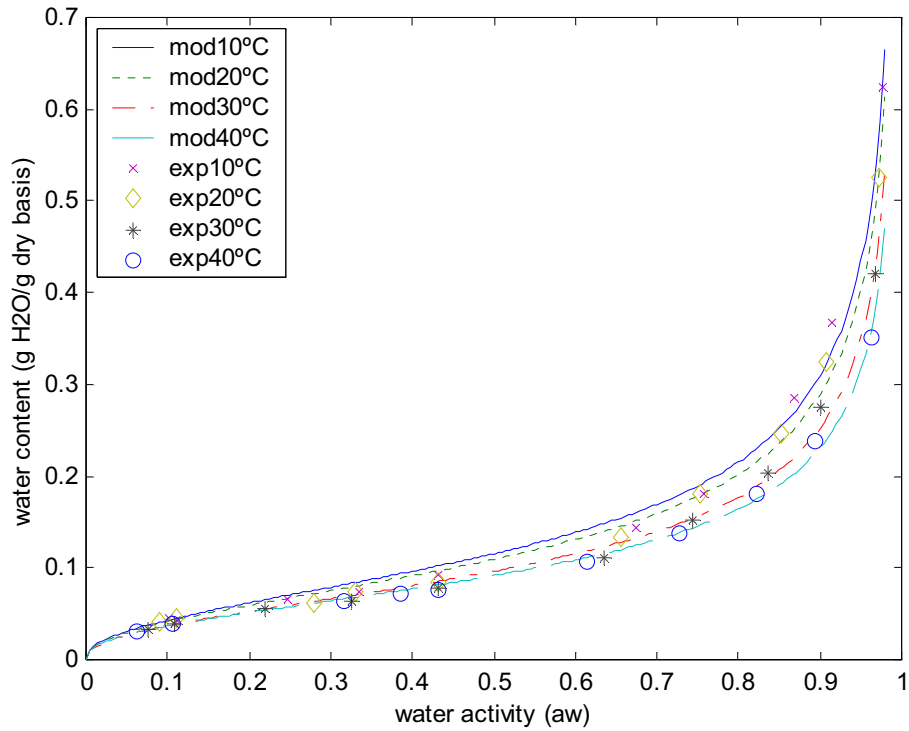


Fig. 1. Sorption isotherm of organic soy protein for the Model of Oswin (Lomauro, Bakshi, & Labuza, 1985, apud: Figueira et al 2004).

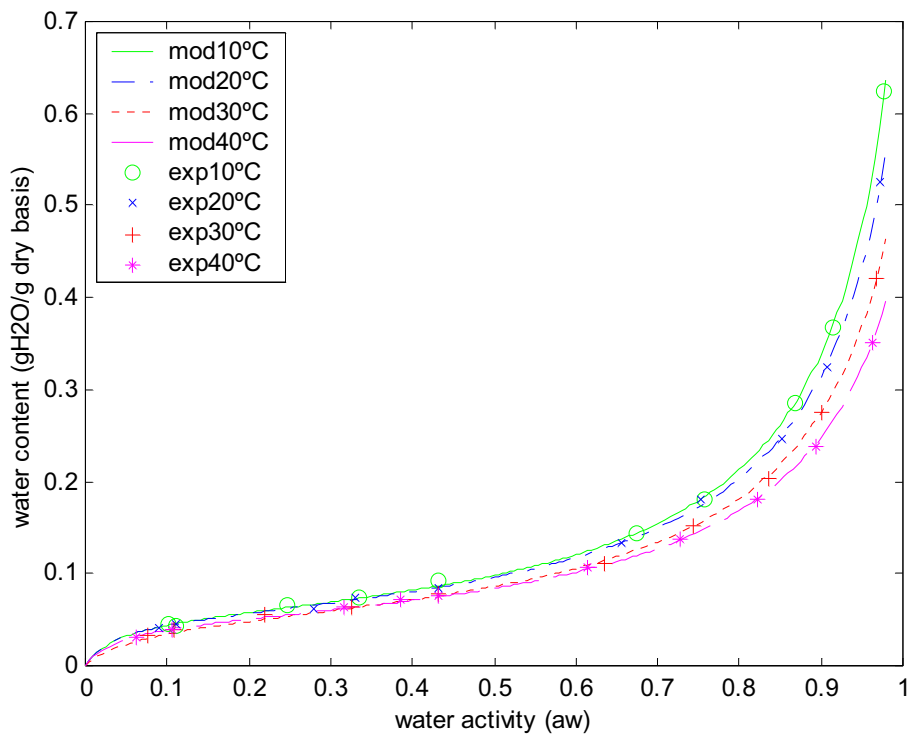


Fig. 2. Sorption isotherm of organic soy protein for the Model of Anderson-Hall (Boquet et al., 1980)

4 Conclusions

The sorption isotherms for the organic soy protein were determined at 10, 20, 30, 40°C using the standard static gravimetric method developed by the European Cooperation Project COST 90. These curves have an important role in the design and the optimization of the unit operations such as preservation, drying, storing, packaging and mixing.

The experimental data were fitted to thirty models (bi-tri-tetra and penta parametrics). The sorption curves for temperatures of 10, 20, 30 and 40°C were better fitted to the model of Anderson-Hall with $E < 5\%$, Anderson, Anderson-Hall, BET (in the range of water activity 0-0.50), Chirife et al., Ferro Fontan and GAB.

Up to water activity of about 0.30, water is considered to be held on polar sites of relatively high energies. This is called the monolayer. Monolayer moisture contents determined for the model BET were found to decrease with increasing temperatures. For the model of GAB the monolayer moisture didn't present a significant difference.

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