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Adsorption Isotherms and Isosteric Heat of Dry Fermented Cocoa Beans (*Theobroma Cacao* L.)

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The San Martín region, located in the northeast of Peru, is the area with the highest cocoa production in the country, highlighting the cocoa clones CCN-51, ICS-39 and ICS-95, of which their physicochemical behavior under storage conditions in the region is unknown. The objectives of present work were: Determine the equilibrium humidity of cocoa bean clones at different temperatures and relative humidity (water activity), model the experimental values of equilibrium humidity and water activity using different sorption equations, estimate the water content monolayer for the different temperature and relative humidity conditions used, and to evaluate the sensory attributes of cocoa beans during storage. The isotherms were obtained by the static gravimetric method, using saturated saline solutions of known water activity, in the range of 0.1 to 0.9. The mathematical models used were GAB, BET, Chung–Pfost, Harkins–Jura, Henderson, Smith, Halsey, Caurie and Kuhn, evaluating the quality of fit to the experimental data by means of the coefficient of determination (R^2). The isotherms obtained were sigmoid, characteristic of type II. The GAB model presented the best degree of adjustment to the experimental data, with R^2 values greater than 0.92. The parameter values of the proposed models showed dependence on temperature.

1. Introduction

Cocoa (*Theobroma cacao* L.) is a crop in great demand worldwide due to its nutritional composition and sensory characteristics. Since 2007, the San Martín region has been the main cocoa-producing region in Peru, reaching a production of 65,011.7 tons in 2022, which represented 38.42 % of the national cocoa production (Ministerio de Desarrollo Agrario y Riego [MIDAGRI], 2023).

During storage and transport, cocoa beans can deteriorate due to variations in temperature and relative humidity that, by generating adsorption or desorption of water vapor in the beans, can promote rapid deterioration of their sensory and physical-chemical characteristics (Saza and Jimenez, 2020). The adsorption process is a phenomenon that occurs on the surface of almost all solids and depends on several factors: the physicochemical properties of the adsorbent, the chemical characteristics of the adsorbates, the prevailing experimental conditions, and the affinity between the adsorbent and the adsorbate. The adsorption capacity of the adsorbent may be related to functional groups and affect the adsorption efficiency of solids (Collazos-Escobar et al., 2020). Knowledge of the effect of temperature on sorption isotherms is of great importance since foods can be exposed to a variety of temperatures during storage and processing (Aviara, 2020). A measure of the hygroscopicity of a product turns out to be the magnitude of the increase or decrease in its water content depending on the relative humidity at a given temperature (Labuza and Altunakar, 2020). Weakly hygroscopic products show no or only a slight change in their water content as a consequence of variations in relative humidity. In strongly hygroscopic products, the water content can vary widely (Medeiros et al., 2006).

The sorption phenomenon has been extensively studied in various resources and agro-industrial products. Among the works related to water vapor sorption in cocoa beans, whole or ground, it is possible to indicate those carried out by Sandoval et al. (2002), who determined the sorption isotherms of fermented cocoa (*Theobroma cacao*) in the temperature range of 5 °C to 30 °C; Sandoval and Barreiro (2002), for second quality Venezuelan cocoa; Medeiros et al. (2006), who studied the sorption isotherms for cocoa and cupuassu products;

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Koua et al. (2016), who estimated thermodynamic properties during the desorption of water from cocoa beans; Hermann et al. (2018), who determined the adsorption and desorption isotherms of Amazonian cocoa at a temperature of 25 °C; Sandoval et al. (2020), who obtained water sorption data on dehulled fermented beans and Trinitario cocoa husk; Barreiro and Sandoval (2020), who developed a model to predict water adsorption on dry cocoa beans during storage; Saza and Jiménez (2020), who evaluated temperature and relative humidity conditions to prolong the shelf life of cocoa beans during storage; Collazos-Escobar et al. (2020), who fixed the water adsorption isotherms at temperatures of 25, 30 and 40 °C and water activities between 0.10 and 0.85, and Bidias et al. (2022), who established desorption isotherms of cocoa beans from Cameroon in the temperature range of 20 ºC to 45 ºC and relative humidities between 10 % and 90 %.

The isosteric heat of sorption is a thermodynamic property related to the amount of energy necessary for the unit mass of a product to change from the liquid phase to the vapor phase, at a certain temperature and water activity (a_w), which can be calculated from the sorption isotherms obtained at least two different temperatures (Akmel et al., 2015).

In the San Martín region, the most cultivated cocoa clones are CCN-51, ICS-39 and ICS-95, of whose fermented and dried beans their behavior under storage conditions applied at the regional level is unknown. The determination of the equilibrium humidity, associated with mathematical models of adsorption isotherms, will allow the generation of information to predict the optimal storage conditions and avoid post-harvest losses of this product of great economic interest in the San Martín region, for which the objectives of the present work were: to determine the equilibrium moisture and security moisture of cocoa clone beans at different temperatures and relative humidity (water activity), to model the experimental values of equilibrium moisture and water activity, through different sorption equations, evaluate the monolayer water content and estimate the isosteric heat of sorption for the different conditions of temperature and relative humidity used.

2. Materials and methods

The present work was carried out in the Research and Engineering and Design Laboratories of the School of Agroindustrial Engineering of the National University of San Martín.

2.1 Materials

The cocoa beans used, from clones ICS-95, ICS-39 and CCN-51, came from crops installed in the provinces of Huallaga and Mariscal Cáceres in the San Martín Region.

Table 1: Mathematical models of sorption isotherms

Eq.	Model	Model equation	References
1.	GAB	$X_e = \frac{X_m C_{GAB} K_{GAB} a_w}{(1 - K_{GAB} a_w)(1 - (1 - C_{GAB}) K_{GAB} a_w)}$	Medeiros et al. (2006)
2.	BET	$X_e = \frac{X_m C_{BET} a_w}{(1 - a_w)(1 + a_w(C_{BET} - 1))}$	Medeiros et al. (2006)
3.	Harkins–Jura	$X_e = \left[\frac{B}{ln a_w - A}\right]^{0.5}$	Akmel et al. (2015)
4.	Smith	$X_e = B + A\, log(1 - a_w)$	Vega et al. (2006)
5,	Halsey	$X_e = \left[\frac{A}{ln(1/a_w)}\right]^{1/B}$	Aviara (2020)
6.	Caurie	$X_e = exp\left[a_w ln(v) - \frac{1}{4.5X_s}\right]$	Vega et al. (2006)
7.	Chung–Pfost	$X_e = A - B ln[-(T + c) ln(a_w)]$	Aviara (2020)
8.	Henderson	$X_e = 0.01\left[\frac{-log(1 - a_w)}{10^f}\right]^{1/n}$	Vega et al. (2006)
9.	Kuhn	$X_e = \frac{A}{ln a_w} + B$	Akmel et al. (2015)

In Table 1: X_e = equilibrium moisture (g water/g d.s.); a_w = water activity; X_m = monolayer moisture (g water/g d.s.); A, B, C_{BET}, C_{GAB}, K_{GAB}, X_s, v, k, n, f, are constants of the equations; T= temperature (K).

2.2 Proximal analysis

The proximal characterization of the cocoa beans, fermented and dried, was carried out using validated methods (A.O.A.C., 1996).

2.3 Reagents

Saturated solutions of lithium chloride (LiCl), potassium acetate (CH_3CO_2K), magnesium chloride ($MgCl_2$), magnesium nitrate ($Mg(NO_3)_2$), sodium nitrite ($NaNO_2$), sodium chloride (NaCl), ammonium sulphate ($(NH_4)_2SO_4$), potassium chloride (KCl) and barium chloride ($BaCl_2$), were used.

2.4 Cocoa bean adsorption isotherm

For the determination of the equilibrium moisture of the fermented and dry cocoa beans, the gravimetric static method was used (Sandoval et al., 2002). The mathematical models of GAB, BET, Harkins-Jura, Smith, Halsey, Caurie, Chung-Pfost, Henderson and Kuhn, were applied to adjust the equilibrium moisture values using the MicroCal Origin program 9.0, evaluating the quality of fit to the experimental data through the coefficient of determination (R^2). Monolayer moisture was determined using the BET and GAB models; the security moisture (X_s) was calculated using the Caurie model. The mathematical models of isotherms used are shown in Table 1.

2.5 Isosteric heat of sorption

The net isosteric heat of sorption (q_{st}) defined as the difference between the total isosteric heat of sorption (Q_{st}) and the heat of condensation (ΔH_{vap}) (Eq 10), was calculated using the Clausius-Clapeyron equation (Eq 11):

$$q_{st} = Q_{st} - \Delta H_{vap} \quad (10)$$

$$ln(a_w) = -\left(\frac{q_{st}}{R}\right)\left(\frac{1}{T}\right) + C \quad (11)$$

In the above equations:
ΔH_{vap} = 43 kJ/mol
R = 8.314 kJ/mol K
C = constant

From the experimental sorption data, plotting the natural logarithm of water activity ($ln\ a_w$) versus $1/T$, the q_{st} can be obtained considering that the slopes of the straight lines obtained are constant in the temperature range studied (Akmel et al., 2015).

2.6 Statistic analysis

The quality of the fit of the proposed models to the experimental values was evaluated by means of the coefficient of determination (R^2) and the chi-square statistic X^2.

3. Results and discussion

3.1 Adsorption isotherms

Figure 1 shows the adsorption isotherms of the three cocoa clones considered, for temperatures of 25 °C, 30 °C and 35 °C, fitted with to the GAB model. It can be seen that the isotherms corresponded to type II according to the Brunauer classification, which is the most common in food and implies a multilayer physical adsorption (Saza and Jiménez, 2020). This result is similar to that obtained by Herman et al. (2018) for Amazonian cocoa and is representative of food products rich in soluble components. Thus, it can also be noted that the equilibrium moisture content is dependent on temperature for the entire range of a_w considered, being evident that the samples adsorbed more water at 25 °C than at 35 °C, probably due to the fact that the water at low temperatures have a lower kinetic energy, which is not enough to overcome the corresponding adsorption energy (Koua et al., 2016).

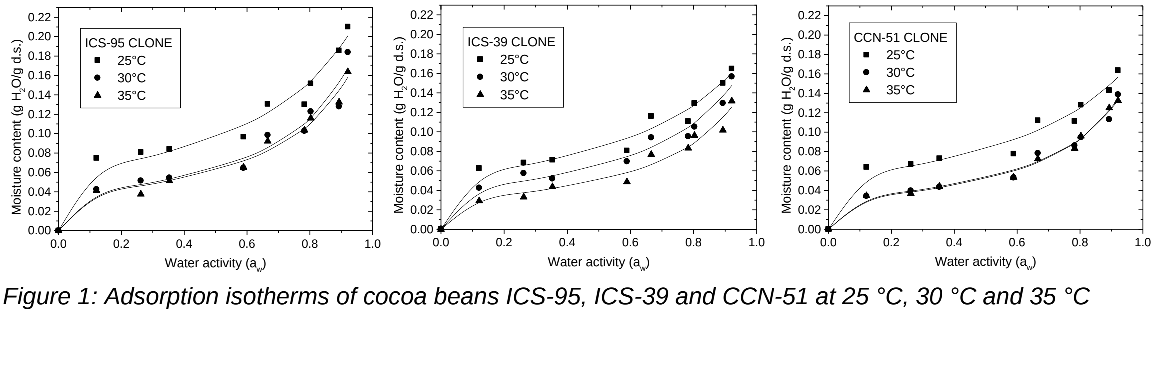

Figure 1: Adsorption isotherms of cocoa beans ICS-95, ICS-39 and CCN-51 at 25 °C, 30 °C and 35 °C

In Figure 1, for clones ICS-95 and CCN-51 it can be seen that the 30 °C and 35 °C isotherms are very close or overlap. This behavior is similar to that reported by Sandoval and Barreiro (2002), for dry, fermented and non-fermented cocoa, and it can probably be attributed to the fact that the heat of dissolution of the solutes and the heat of adsorption turned out to be approximately the same. The energy involved in the sorption process is constituted by the algebraic sum of the heat of dissolution of the solute (endothermic process) and the heat of sorption of the remaining solids (exothermic process); the net effect of temperature on the isotherms would depend on the magnitude of both. In addition, it can be seen that the equilibrium moisture values are higher, for clone ICS-95, for the three temperatures used (25 °C, 30 °C and 35 °C), throughout the entire range of a_w considered. This behavior can be attributed to the different chemical composition of the grains of the clones used and the initial moisture content, which was higher in the case of clone ICS-95 (8.76 %). In this regard, Labuza and Altunakar (2020), mention that the sorption properties of food are affected by the composition, processing conditions, temperature, pressure and relative humidity. The values obtained for the parameters of the models used, in addition to the values of R^2 and X^2, are presented in Table 2. With the exception of the BET equation, the entire range of experimental values of a_w was considered, the models used presented a good degree of adjustment to the experimental data, with R^2 values close to or greater than 0.9 and low X^2 values.

Table 2: Isotherm parameter values of dry fermented cocoa beans at 25°C, 30°C and 35°C

Eq.	Para-meters	25 °C			30 °C			35 °C		
		ICS–95	ICS–39	CCN–51	ICS–95	ICS–39	CCN–51	ICS–95	ICS–39	CCN–51
1.	C	3,426.28	674.541	834.748	1,532.50	100.738	218.358	134.865	37.542	116.860
	K	0.7654	0.7255	0.7185	0.8428	0.7912	0.8343	0.8351	0.8335	0.8422
	X_m	0.05924	0.05323	0.05302	0.03743	0.04007	0.03086	0.03652	0.03131	0.03024
	χ^2	1.10E-04	7.64E-05	7.63E-05	1.73E-04	6.45E-05	3.39E-05	5.77E-05	6.56E-05	1.67E-05
	R^2	0.9586	0.9523	0.9486	0.9291	0.9605	0.9772	0.9737	0.9535	0.9892
2.	C	10.4914	10.2553	9.5152	12.4365	14.0873	13.8876	22.9645	15.7250	15.2787
	X_m	0.03884	0.03242	0.03128	0.02623	0.02805	0.02159	0.02662	0.02010	0.021766
	χ^2	1.26E-04	2.01E-04	2.76E-04	2.76E-04	1.81E-04	3.12E-04	1.71E-04	6.66E-04	2.92E-04
	R^2	0.9735	0.9707	0.9633	0.9727	0.9790	0.9786	0.9812	0.9606	0.9794
3.	A	0.1008	0.1560	0.1654	0.0260	0.0658	0.0352	0.0350	0.0413	0.0301
	B	-0.00751	-0.00611	-0.00606	-0.00315	-0.00331	-0.00214	-0.00299	-0.00198	-0.00208
	χ^2	1.35E-04	9.30E-05	9.73E-05	1.60E-04	5.72E-05	3.24E-05	6.02E-05	6.77E-05	2.17E-05
	R^2	0.9491	0.9419	0.9346	0.9344	0.9649	0.9782	0.9725	0.9520	0.9860
4.	A	-0.12834	-0.09911	-0.0954	-0.12032	-0.10057	-0.09642	-0.11696	-0.09293	-0.09877
	B	0.06037	0.0544	0.05478	0.03176	0.03686	0.02557	0.02841	0.0232	0.02392
	χ^2	8.93E-05	6.86E-05	6.59E-05	1.75E-04	5.39E-05	3.53E-05	5.40E-05	6.26E-05	2.50E-05
	R^2	0.9664	0.9572	0.9557	0.9281	0.9670	0.9762	0.9753	0.9556	0.9839
5.	A	8.74E-04	2.73E-04	2.25E-04	0.00148	6.20E-04	8.23E-04	0.00132	7.24E-04	9.50E-04
	B	2.8879	3.1808	3.2468	2.2795	2.5990	2.3165	2.2799	2.3175	2.2536
	χ^2	7.17E-05	7.27E-05	6.88E-05	1.50E-04	4.21E-05	3.30E-05	6.90E-05	7.57E-05	2.66E-05
	R^2	0.9731	0.9546	0.9537	0.9383	0.9742	0.9778	0.9685	0.9464	0.9828
6.	*V*	4.3409	3.8276	3.6894	7.1882	5.3431	7.1678	7.6519	7.3707	7.8993
	X_s	0.0735	0.0714	0.0716	0.0604	0.0631	0.0570	0.0588	0.0559	0.0558
	χ^2	2.61E-04	1.29E-04	1.31E-04	2.73E-04	1.30E-04	8.16E-05	9.44E-05	8.17E-05	6.82E-05
	R^2	0.9018	0.9194	0.9115	0.8880	0.9204	0.9451	0.9569	0.9421	0.9559
7.	A	0.2212	0.1965	0.1938	0.1343	0.1281	0.1203	0.1422	0.1260	0.1291
	B	0.0418	0.0326	0.0313	0.0393	0.0330	0.0316	0.0384	0.0306	0.0323
	C	-274.729	-258.515	-255.254	-296.238	-295.031	-292.957	-298.280	-293.502	-295.036
	χ^2	2.18E-04	1.18E-04	1.18E-04	2.88E-04	1.09E-04	8.76E-05	1.20E-04	9.48E-05	8.55E-05
	R^2	0.9298	0.9370	0.9317	0.8985	0.9428	0.9495	0.9530	0.9424	0.9526
8.	n	2.2261	2.4571	2.5316	1.6232	1.9315	1.6354	1.5819	1.6177	1.5619
	f	-2.8054	-2.8748	-2.9367	-1.9039	-2.1729	-1.7612	-1.8250	-1.7071	-1.6879
	χ^2	2.95E-04	1.55E-04	1.60E-04	2.71E-04	1.30E-04	8.77E-05	1.09E-04	8.63E-05	8.07E-05
	R^2	0.8891	0.9030	0.8925	0.8886	0.9201	0.9410	0.9501	0.9389	0.9479
9.	A	-0.0116	-0.0087	-0.00842	-0.0109	-0.00904	-0.0086	-0.0103	-0.00813	-0.00877
	B	0.0805	0.07098	0.07058	0.05055	0.05287	0.04119	0.04787	0.03888	0.04007
	χ^2	1.74E-04	2.03E-04	1.78E-04	2.40E-04	1.25E-04	1.22E-04	2.31E-04	1.91E-04	1.28E-04
	R^2	0.9346	0.8730	0.8803	0.9012	0.9234	0.9178	0.8944	0.8645	0.9172

3.2 Monolayer moisture and security moisture values

Table 2 shows the monolayer moisture values (X_m) calculated through the BET and GAB equations. Sandoval and Barreiro (2002), Sandoval et al. (2002) and Sandoval et al. (2020), reported monolayer moisture values, calculated using the BET equation, of 2.94 g of water/100 g d.s., 3.56 g of water/100 g d.s., and 3.53 g of water/100 g d.s., for dry fermented cocoa, dry non-fermented cocoa and dehulled fermented cocoa, respectively; which are close to the values determined in this work. The monolayer moisture content determined by the GAB equation was between 0.06 and 0.03 g of water/g d.s., for the temperature interval between 25 °C and 35 °C, respectively, which are close to the value reported by Koua et al. (2016) of 0.061 g water/g d.s. for 30 °C, and that reported by Herman et al. (2018) of 0.029 g water/g d.s. at 25 °C; differences in monolayer moisture content can be attributed to differences in the chemical composition of the different cocoa clones. In addition, in Table 2 it can also be seen that the monolayer moisture content values decrease with increasing temperature for the three cocoa clones studied, a similar behavior was reported by Koua et al. (2016) in experiences carried out with cocoa beans in the Ivory Coast.

The behavior described can be attributed to the lower availability of the active sites (or hydrogen bonds) in the molecules of the substances that make up the cocoa beans; thus, it must also be taken into account that at a lower temperature the kinetic energy of the water molecules decreases, which limits the hygroscopic capacity of the product (Iglesias and Chirife, 1976). Furthermore, according to Palipane and Driscoll (1992), it could be that with increasing temperature, water molecules are activated due to an increase in their energy level, making them less stable and moving away from sites binding with food, thus decreasing the monolayer moisture content. These observations imply that knowing the storage temperature, and equilibrium moisture, of cocoa beans can determine the optimum moisture content for maximum stability.

Table 2 shows the security moisture values (X_s), a parameter related to the stability of dehydrated foods, calculated using the Caurie equation for the cocoa clones used. These X_s values are around 0.07 g of water/g d.s., 0.06 g of water/g d.s. and 0.05 g of water/g d.s., for temperatures of 25 °C, 30 °C and 35 °C, respectively, and are close to the equilibrium moisture values determined for the same cocoa clones considering a value of a_w of 0.6. Care should be taken not to exceed the value of X_s during the storage of dry cocoa beans, since spoilage reactions may occur at high equilibrium moisture values, as a result of microbial development and enzymatic activity (Rockland and Nishi, 1980).

3.3 Net isosteric heat of sorption

The net isosteric heat of sorption (q_{st}) of the three cocoa clones as a function of the equilibrium moisture content obtained using the Clausius-Clapeyron equation, is presented in Figure 2. It can be seen that the q_{st} decreases with increasing content equilibrium moisture. Thus, for an equilibrium moisture content of 6 %, the q_{st} value is close to 200 kJ/mol for clone ICS-95 and 100 kJ/mol for clones ICS-39 and CCN-51, respectively, while for an equilibrium moisture content of 16 % the q_{st} decreases to values between 10 and 20 kJ/mol. This behavior can be explained considering that sorption occurs initially in the sites of highest activity, with a higher interaction energy, while when the equilibrium moisture content increases, the sites available for water sorption decrease, resulting in lower net isosteric heat of sorption values (Akmel et al., 2015). Similar results have been reported by Medeiros et al. (2006) for cocoa and cupuassu and by Koua et al. (2016) for cocoa beans.

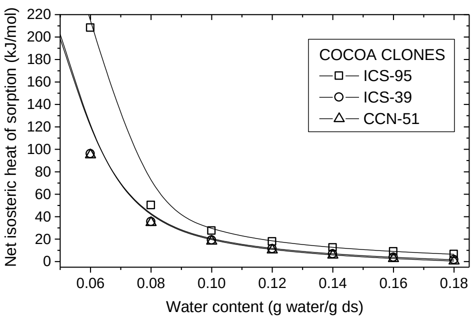

Figure 2: Isosteric heat of sorption of cocoa clones ICS-95, ICS-39 and CCN-51

4. Conclusions

The adsorption isotherms of the fermented and dry cocoa beans, clones ICS-95, ICS-39 and CCN-51, at temperatures of 25 °C, 30 °C and 35 °C, presented the shape corresponding to type II. This behavior is characteristic of products that contain soluble components, such as sugars. In addition, the adsorption isotherms showed dependence on temperature for the entire range of a_w values considered (0.1 to 0.9).

The equations used, except BET, adequately modeled the experimental data for the three cocoa clones for the working conditions considered, with R^2 values greater than 0.9 and low values of the X^2. The BET equation presented a good degree of adjustment for values of a_w less than 0.5.
The monolayer moisture showed dependence on temperature, because at a lower temperature the kinetic energy of the water molecules decreases, which limits the hygroscopic capacity of the material until it reaches the maximum value that corresponds to the monolayer. The storage of fermented and dry cocoa beans must be carried out at relative humidity values not greater than 60 % (a_w≈0.6), so as not to exceed the safety moisture content (X_s) and maintain the quality of the cocoa beans. of cocoa.
In the considered temperature range, the net isosteric heat of sorption decreases with increasing equilibrium moisture content, for the three cocoa clones studied.

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