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Sorption isotherms and isosteric energy of *Manihot esculenta* leaves

*Edoun, M. and Kuitche, A.

Department of Energetic, Electrical and Automatic Engineering, ENSAI, P.O. Box 455
University of Ngaoundere-Cameroon

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Abstract

This work presents the study sorption isotherms of *Manihot esculenta* leaves. Desorption-adsorption isotherms were determined by using static gravimetric method at 30, 40 and 50°C for water activity ranging from 0.10 to 0.90. Results obtained show that at high water activities, adsorption of water increased with increasing temperature and isotherm curves of adsorption and desorption did not follow the same lend: the hysteresis cycle. The sorption isotherms of *M. esculenta* leaves are of type II. Five different models were used to study these experimental curves and GAB'S model best describes these sorption isotherms (MRE < 10% and $R^2 > 0.996$). The sorption heat of the monolayer HM was 3.073 kJ.mole^{-1} while that for the multilayer was 0.970 kJ.mole^{-1}. For *M. esculenta* isosteric leaves heat of sorption varies from 32.15 to 3.54 kJ.mole^{-1} and the values of q_0 and X_0 for desorption were respectively 71.43 kJ.mole^{-1} and 7.643 g water/g (d.b).

Introduction

Manihot esculenta leaves is consumed as a vegetable by a large majority of the people living in Africa. These leaves can be consumed raw or used to cook traditional dishes. Collect and sales of *M. esculenta* leaves in the market has become a daily activity that generates income through international trade. But after harvest, fresh Casava leaves undergo continuous physical, chemical and microbiological changes. The quality of the leaves can be thereby seriously degraded within a few days. In fact, these changes are particularly influenced by moisture content of the material, relative humidity of ambient air. Actually, about 40% of the production was lost postharvest. It therefore becomes necessary to investigate appropriate preservation methods which are acceptable to all. The drying process looks like the only means to these exigencies. To design of an adapted dryer, the essential parameters that should be known are: the drying time, the energy required for drying and the isosteric heat of sorption of product. The isosteric heat of sorption can be determined using the sorption isotherm data.

The moisture sorption isotherm describes the relationship between the water activity (A_w) and the equilibrium moisture content (X_{eq}) of a product at a constant pressure and temperature. Sorption isotherms of some fruits and leaves were reported in literature. Nogueira-Terrones *et al.* (2004); Adebowale *et al.* (2008); Iguedjtal *et al.* (2008); Zhengyong *et al.* (2008) have determined sorption isotherms and the isosteric heat of some fruits while Timoumi and Zagrouba (2005); Jamali *et al*. (2006); Lemus *et al*. (2008); Edoun *et al*. (2010) did it for some leaves. However, sorption isotherms data of *M. esculenta* leaves are not available in the literature. The present work consists to establishing the sorption isotherms at three different temperatures and to determine the best model which will enable the best experimental curves and thirdly to estimate the isosteric heat of sorption of *M. esculenta* leaves.

Materials and methods

Biological material

Samples used for this work are leaves of *M. esculenta* collected in the south of Cameroun and available in the local markets. After the harvest, leaves were preserved at 4°C and five days are between the harvest and the use of samples.

Determination of sorption isotherms

To determine the equilibrium moisture content of *M. esculenta* leaves, the static gravimetric method was used at 30, 40 and 50°C. These temperatures are often used in storage and drying of food products. Hence, eight saturated salt solutions (LiCl, KCH_3CO_2, $MgCl_2$, K_2CO_3, NaBr, $SrCl_2$, NaCl and of $BaCl_2$) were used to control the relative humidity in the experimental jars. These salt solutions were obtained by dissolving an appropriate quantity of salt in distilled water (Table 1) recommended by the COST 90 project (Lowell and Shields, 1991). The jars were put in a controlled temperature in the oven for 24 h to be stabilised at (30, 40 or 50^{o}C). A small quantity

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*Corresponding author.
Email: *edounmacel@yahoo.fr*
Tel: (00237) 77 52 60 89

Table 1. The composition and water activities (A_w) of saturated salt solutions at 30, 40 and 50°C

Salt Solutions	**Quantity used**		**Aw**		
	Salt (g)	Distilled water (ml)	**30 °C**	**40 °C**	**50 °C**
LiCl	150	85	0.1116	0.1116	0.1105
KCH_3CO_2	200	65	0.2204	0.2163	0.2162
$MgCl_2$	200	25	0.3238	0.3159	0.3054
$K2CO_3$	195	90	0.4317	0.4221	0.4091
NaBr	200	80	0.5632	0 .5323	0.5091
$SrCl_2$	200	50	0.6911	0.6810	0.5746
NaCl	200	60	0.7520	0.7501	0.7484
$BaCl_2$	240	70	0.8980	0.8910	0.8823

Table 2. Models applied to the experimental sorption data of *Manihot esculenta* leaves

Models	**Equations**		**Parameters**	**References**
GAB	$X_e = \frac{ABCa_w}{(1 - Ba_w)(1 - Ba_w + BCa_w)}$	(2)	A, B, C	(Van der Berg and Bruin,1981)
Haley	$X_e = \left(\frac{A}{Ln\left(1/a_w \right)} \right)^{\frac{1}{B}}$	(3)	A, B	(Lemus *et al.*, 2008)
Oswin	$X_e = A\left(\frac{a_w}{1 - a_w} \right)^B$	(4)	A, B	(Oswin, 1946)
Handerson	$X_e = \left(\frac{A}{Ln\left(1/a_w \right)} \right)^{\frac{1}{B}}$	(5)	A, B	(Henderson, 1952)
Smith	$X_e = A + BLog(1 - a_w)$	(6)	A, B	(Smith, 1947)

of solid salt was added to make sure that solutions remain saturated. Thus, each jar provides a fixed relative humidity (RH) corresponding to a fixed water activity (A_w) for each temperature. The saturated salt solutions allow obtaining a water activity ranging from 0.1105 to 0.8980 (Edoun *et al.*, 2010).

The quantities of leaves used for desorption were 0.4000 ± 0.0001 g and 0.2000 ± 0.0001 g for adsorption. Triplicate samples were introduced into each jar. These samples were suspended into the jar using a perch. Fresh leaves were used for desorption experiments. Samples used for adsorption isotherms were previously dried for 24 h in an oven at 70°C. The eight jars containing saturated salt solutions and samples were tightly closed and put in an enclosure at a fixed temperature (30, 40 or 50 ± 1◦C) for equilibration. The samples were weighed using a Satorius BELCO balance (±0.0001 g). The weighing began after the 10th day and equilibrium moisture content (X_{eq}) was acknowledged when three consecutive weight measurements showed a difference of less than 0.001 g (after 10 - 14 days). Thus for desorption, the equilibrium moisture was obtained after fourteen (14) days and for adsorption after twelve (12) days. The dry matter content of equilibrated samples was determined through an oven at 105◦C (±0.1°C) for 24 h (A.O.A.C, 1990). The difference of mass before (M_h) and after (M_s) drying in the oven gives the equilibrium moisture content of the product.

$$X_{eq} = \frac{M_h - M_s}{M_s} \qquad (1)$$

Modeling of sorption isotherms

In the literature, they exists many models which describe sorption isotherms but in this work, five models are used and presented in Table 2. These five models used in this study present the advantage of correctly describing sorption isotherms of agro-food products for water activity values between 0.05 and 0.95 (Iguedjtal *et al.*, 2008; Mariana *et al.*, 2008). The three constants in GAB'S Model (A, B, C) are determined from experimental data. C is the GAB'S constant and B is the correction factor of the properties of the multilayer of the product.

Expression of C is given by: $C = C_0 \exp\left(\frac{H_M - H_N}{RT} \right)$ (7)

And the constant B is given by: $B = B_0 \exp\left(\frac{H_L - H_N}{RT} \right)$ (8)

Where: C_0; B_0: entropic accommodation factors
H_L: Latent heat of vaporisation of liquid water (J/mole), suppose 43980 J/mole (10500 cal/mole) on the domain between 20 and 80°C
H_M: Total sorption heat of the monolayer (J/mole).
H_N: Total sorption heat of the multilayer (J/mole).
R: Universal gas constant (kJ/mol K)
T: The absolute temperature (K)

Calculation of the isosteric heat of sorption

The concept of net isosteric heat of sorption (q_n) is defined as the total heat of sorption in the food (q_t) minus the vaporization heat of pure water (ΔH_v) at a given temperature:

$$q_t = q_n + \Delta H_v \qquad (9)$$

The net isosteric heat of sorption is thermodynamically given by:

$$q_{st} = -R \frac{\partial (Lna_w)}{\partial \left(1/T \right)} \qquad (10)$$

Where q_{st} is the net isosteric heat of sorption at constant moisture content (kJ mol^{-1} water). This relationship was derived from the Clausius-Clayperon equation, applied to the system and pure water after some assumptions. In an attempt to describe the relationship between the net isosteric heat of sorption and the moisture content, Tsami *et al.* (1990) proposed an empirical exponential correlation, which can be written as:

$$q_{st} = q_0 \exp\left(\frac{X_{eq}}{X_0}\right) \qquad (11)$$

where q_0 is the net isosteric heat of sorption of the first molecules of water in the food (kJ mol^{-1}), X_{eq} is the equilibrium moisture content, (kg/kg$_{\text{dry solid}}$), and X_0 is the characteristic moisture content of the food material, (kg/kg$_{\text{dry solid}}$), q_0 provides important information on both the physico-chemical interactions of water with the major food constituents and the state of water within the food system. It is an invaluable parameter, for estimation of the energy requirements during drying. Practically, the net isotherm heat of sorption can be deduced from the slope of the sorption isotherms representing $Ln(a_w)$ as a function of $1/T$ for each product water content X, and then the curve $Q_{sorp} = f(X)$ can be plotted.

Evaluation of models

Three parameters were used to evaluate the correlated models to the experimental kinetics. But the correlation coefficient (R^2) is the first criteria to appreciate the best model which describes isotherms sorption. To refine the selection, the Mean Relative Error (*MRE*) and the Standard Error of Estimate (*SEE*) were also used to improve the precision of reading the curves. These statistical parameters were calculated using the following equations:

$$MRE = \frac{100}{N}\sum_{i=1}^{N}\left(\frac{M_{i,\exp} - M_{i,pre}}{M_{i,\exp}}\right) \qquad (12)$$

$$SEE = \sqrt{\frac{\sum_{i=1}^{N}\left(M_{i,\exp} - M_{i,pre}\right)^2}{d_f}} \qquad (13)$$

$M_{i,pre}$: The predicted equilibrium moisture content (kg/kg dry solids),
$M_{i,\exp}$: The experimental equilibrium moisture content (kg/kg dry solids),
N : The number of experimental data

Results and Discussion

Experimental sorption kinetics

Figures 1 and 2 compare the desorption and adsorption isotherm respectively at 30°C, 40°C and 50°C. The sorption isotherm curves of *Manihot esculenta* are of type II sigmoidal characteristic of agro-food products rich in carbohydrates (Emilio *et al.*, 1999). For $a_w < 0.7$, the higher the temperature, the lower the equilibrium composition. Tsami *et al.* (1990) obtained the similar results for Raisins, Currants, Figs, Prunes and Apricots.

Table 3. Estimated parameters and fitting criteria of the models applied to experimental desorption and adsorption data of *Manihot esculenta* leaves

Models	Estimated Parameters	Desorption			Adsorption		
		30ºC	40ºC	50ºC	30ºC	40ºC	50ºC
GAB	A	0.085	0.079	0.066	0.058	0.054	0.042
	B	0.884	0.886	0.872	0.910	0.868	0.903
	C	43.153	70.094	58.596	99.021	97.000	92.001
	R²	0.996	0.993	0.982	0.994	0.992	0.970
	MRE	0.046	0.033	0.012	0.026	0.006	0.010
	SEE	0.007	0.008	0.025	0.007	0.017	0.012
Oswin	A	0.157	0.135	0.122	0.113	0.101	0.086
	B	0.434	0.425	0.407	0.458	0.403	0.413
	R²	0.990	0.986	0.977	0.982	0.982	0.939
	MRE	0.092	0.065	0.049	0.052	0.033	0.015
	SEE	0.011	0.011	0.012	0.011	0.009	0.025
Haley	A	0.556	0.539	0.507	0.508	0.466	0.361
	B	9.53	10.30	10.93	11.090	12.22	16.910
	R²	0.722	0.70	0.659	0.769	0.633	0.687
	MRE	0.067	0.046	0.033	0.041	0.021	0.017
	SEE	0.060	0.053	0.049	0.042	0.042	0.034
Henderson	A	0.492	0.533	0.567	0.571	0.624	0.674
	B	10.74	11.64	12.36	12.46	13.60	14.690
	R²	0.892	0.872	0.837	0.904	0.849	0.801
	MRE	0.083	0.057	0.042	0.048	0.029	0.020
	SEE	0.037	0.034	0.034	0.027	0.027	0.027
Smith	A	0.041	0.037	0.036	0.027	0.029	0.0270
	B	-0.36	-0.303	-0.263	-0.273	-0.217	-0.184
	R²	0.962	0.954	0.937	0.959	0.951	0.90
	MRE	0.089	0.063	0.047	0.051	0.032	0.023
	SEE	0.022	0.020	0.021	0.017	0.015	0.019

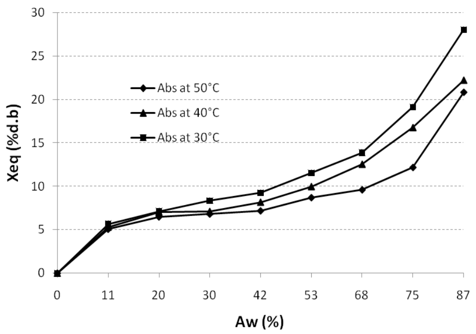

Figure 1. Adsorption isotherm of *Manihot esculenta* leaves at 30, 40 and 50°C

At constant temperature, the equilibrium moisture content increased with increasing water activity. In general, both temperature and water activity have significant effects on experimental equilibrium moisture content values. Similar trends have been observed in several other studies for agricultural and food products (Zanoelo, 2005; Garcia-Pèrez *et al.*, 2008).

At high water activities, adsorption of water increased with increasing temperature. The isotherm curves of adsorption and desorption did not follow the same lend. Adsorption-desorption curves showed that at a same relative constant humidity, the water composition of desorption are greater than that of adsorption (Figure 3). These results corroborate with

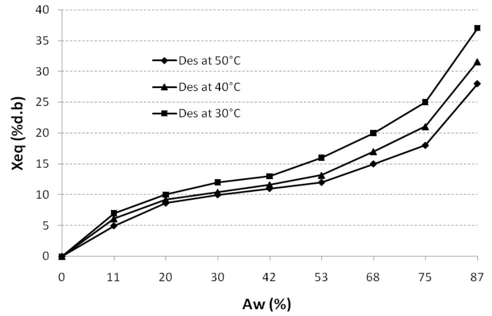

Figure 2. Desorption isotherm of *Manihot esculenta* leaves at 30, 40 and 50°C

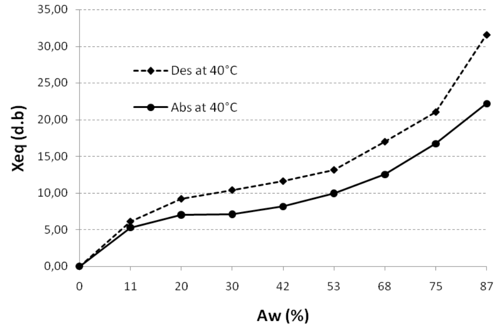

Figure 3. Sorption isotherm of *Manihot esculenta* leaves at 40°C

those of (Lemus *et al*., 2008; Mariana *et al*., 2008). The different equilibrium moisture contents observed at the same water activity, between adsorption and desorption processes constitute the hysteresis cycle. The hysteresis phenomenon was observed in each case. This results is similar to those presented by (Ait Mohamed *et al*., 2005; Kouhila *et al*., 2007; Moreira *et al*., 2008; Edoun *et al*., 2010).

Modeling data

To best describe the sorption isotherms five models were tested. The values of the parameters of these different models tested are presented in Table 3. It can be seen from this table that the GAB'S model, in accordance with literature, is the one that best describes the sorption kinetics of Manihot esculenta leaves for a wide range of water activity (0.1105 - 0.8823). The values of R^2, *MRE* and *SEE* are ranging respectively from 0.993 to 0.996, from 0.046 to 0.010 and from 0.0007 to 0.025 for desorption and adsorption isotherms. In Figure 4, the adjustment of experimental data with the GAB'S model is also shown. A good agreement between experimental and predicted data was found. GAB'S model has also been successfully applied to several agricultural products like potato (Al-Muhtaseb *et al*., 2004) and turnip top leaves and stems (Moreira *et al*., 2005) and *Gnetum africanium* leaves (Edoun *et al*., 2010).

The values of B_0, C_0, $(H_L - H_N)$ and $(H_M - H_N)$ of the GAB'S model determined from the experimental data by using the non linear regression are represented in the table 4. $(H_M - H_N)$ represents the difference between the enthalpy of the monolayer and the multilayer and estimated at 2.103 kJ.mole^{-1} for desorption and 13.200 kJ.mole^{-1} for adsorption. These values indicate that there exist a strong link between the primary hydrophilic sites and the first layer of molecular water. But the difference in the enthalpy variation between desorption value and adsorption value translates the irreversibility of the phenomenon taking into consideration the properties of water in the product.

Table 4. Parameters and constants of GAB'S model

	B_0	$(H_L - H_N)$	C_0	$(H_M - H_N)$
Desorption	0.599	43.01	0.418	2.103
Adsorption	0.371	0,418	42	13.200

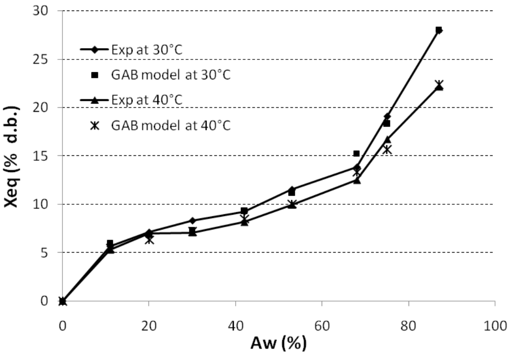

Figure 4. Experimental and theorical adsorption isotherms of *Manihot esculenta* leaves at 30 and 40°C

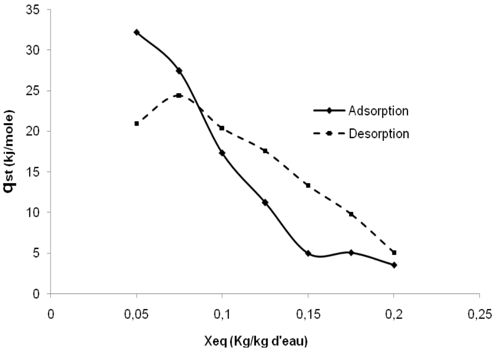

Figure 5. Sorption heat as a function of the equilibrium of *Manihot esculenta* leaves

$(H_L - H_N)$ represents the difference between the latent heat of condensation of pure water and the sorption heat of the multilayer (Van den berg, 1984). This difference was estimated at 43.01 kJ.mole^{-1} for desorption and 0.418 kJ.mole^{-1} for adsorption. These results corroborate with literature (Jamali *et al*., 2006; Moreira *et al*., 2008). Considering H_L (the latent heat of vaporisation of pure water) equals 43.980 kJ.mole^{-1} (10500 cal.mole^{-1}), H_N = 0.970 kJ.mole^{-1}. These results enable the calculation of the desorption heat of the monomolecular layer H_M = 3.073 kJ.mole^{-1}.

We notice that the desorption heat of the monolayer (H_M = 3.073 kJ.mole^{-1}) was greater than the multilayer (H_N = 0.970 kJ.mole^{-1}). This difference is probably due to a strong fixation of water. In the monolayer, the water is strongly fixed to the polar sites by hydrogen bonds of strong energy, while the molecular water in the multilayer are retained by hydrogen bonds which energies are less strong. This explains why water in the monolayer is relatively difficult to remove. Moreover the fibrous texture of *M. esculenta* leaves resists extraction of water. The desorption heat of the multilayer H_N will enable the design and the calculation of the quantity of heat necessary to evaporate water contained in the *M. esculenta* leaves after dimensioning and chose the energetic system adapted for the drying process.

Isosteric heat of sorption

Figure 5 represents the evolution of the sorption heat as a function of water content. The analysis of this figure shows that q_{st} increases with decrease in water content, and this is explained by the strong interaction that exist between water and the adsorbant components of *M. esculenta* leaves. The isosteric heat of sorption (q_{st}) varies from 32.15 to 3.54 kJ.mole^{-1} with a variation in the equilibrium water content ranging from 0.05 to 0.20 g water/g dry basis (d.b.). The high value of the isosteric heat at lower water content indicates that the interaction between the components of the product and water is important. And at high water content, it tilts towards the heat of condensation of pure water (Matallah, 2004; Neila *et al*., 2008).

With respect to Tsami's equation, the values of q_0 and X_0 for desorption were respectively 71.43 kJ.mole^{-1} and 7.643 g water/g (d.b). Lemus *et al.* (2008) obtained for red seaweed q_0 values of 123.73 kJ.mole^{-1} and X_0 values of 0.04 g water/g (d.b). Kiranoudis *et al.* (1993) obtained for fruits and some plants a q_0 value varying between 40 and 115 kJ.mole^{-1} and X_0 varying from 0.08 to 21 g water/g (d.b).

Conclusion

We have presented in this work the sorption isotherms at 30, 40 and 50°C of *Manihot esculenta* leaves and determined. The isotherms sorption follows a sigmoidal curve and was of type II according to the BET classification, typical of most biological products. The GAB'S models permitted us to fit these isotherms with an R^2 above 0.99% and an *MRE* less than 10%. The desorption heat of the monolayer (H_M = 3.073 kJ.mole^{-1}) was greater than that of the multilayer (H_N = 0.970 kJ.mole^{-1}) and the sorption heat determined by the Clausius-Clapeyron equation reduce with increase in equilibrium water content. The isosteric heat of sorption (q_{st}) varies from 32.15 to 3.54 kJ.mole^{-1}. The values of q_0 and X_0 for desorption were respectively 71.43 kJ.mole^{-1}and 7.643 g water/g (d.b). This knowledge will help us to better understand the phenomenon related to the drying and preservation of these leaves, to envision modelling of drying kinetics and to optimize also the design parameters of an adapted dryer to this product.

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