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Research Paper

MOISTURE DESORPTION ISOTHERM AND THERMODYNAMICS PROPERTIES OF SAFOU PULP (*DACRYODES EDULIS*)

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This work aimed at determining the isotherm of desorption of safou pulp (*Dacryodes edulis*). The isotherm of desorption of the pulp was studied at 25, 35, 45 and 55 °C using static gravimetric method. The results revealed that curves obtained have a sigmoid form (type II isotherm). The BET, GAB, Hasley and Oswin models were used to correlate the experimental kinetics. The equations of GAB and BET fitted very well the experimental data. The molecular monolayer moisture content, Xm deriving from the BET model were 2,48%, 3.21%, 4.07, and 7.20% at 55°C, 45°C, 35°C, respectively, while the corresponding GAB values were respectively 2.24%, 3.07%, 3.20 % and 3.42%. The heats desorption was calculated from the Clausius-Clapeyron equation and the equivalent value at moisture content 4% was 2134, 44 kJ/kg. A good adjustment was observed between the experimental values and the calculated GAB values with a coefficient correlation of 0.998 and a standard estimate error of 0.24 at 45 °C.

Keywords: Desorption isotherms, Isosteric heat, Model, Safou

INTRODUCTION

Safou tree (*Dacryodes edulis*) is a non conventional oilseeds plant belonging to the family burseraceae (Kengué, 2002). The fruit called "safou" is very rich in oil with a value range 30 -70 g/100g dry matter (Ali *et al*., 1997). The oil extracted from the fruits is used in cosmetic for the manufacture of soaps and perfumes, the decoction of its sheets is used to fight against digestive disorder and tooth aches. Safou has a great economic importance and industrial potential. In 1997 the market trade of safou fruit was evaluated to about 5 billion francs CFA (Noumi *et al*., 2006). According to Kengue (2002), up to 105 tons of safou fruits are exported from central Africa in direction of Europe where the

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prices vary from a country to another: 8.85 €/kg in Belgium and 0.77 €/kg in France. Unfortunately, the safou fruit is very perishable under tropical conditions leading to enormous post-harvest losses. In addition no post harvest technology and control is actually applied to the fruit. As a consequence, losses accounted to about 50% in Cameroun and 65% in Nigeria (Noumi *et al.*, 2006). It then becomes necessary to control the conditions of conservation. One of the characteristics necessary to improve the conservation of a foodstuff is the isotherm of sorption (Nkouam, 2006). In fact the moisture sorption isotherm describes the relationship between the water activity (aw) and the equilibrium moisture content (Xe) of a product at a constant temperature (Bahloul *et al.*, 2008). Thus, with knowledge of the moisture desorption isotherm, it is possible to predict the maximum moisture that safou fruit can be allowed to gain or lose during storage. Sorption isotherms are essential tools in dehydration processes for predicting shelf-life stability, packaging and drying of a desired product. In particular the isosteric heat of sorption which is a measure of the energy requirement to remove water from a product provide a measure of the physical, chemical and microbiological stability of food material under given storage conditions and supplies valuable data for energy consumption calculations and subsequent design of drying (Bahloul *et al.*, 2008).

Al-Muhtaseb *et al.* (2002) made a literature review on the isotherms of sorption of foodstuffs and examined some models of kinetics of sorption and their conditions of validity; and they listed some of the products on which these models were tested. From this review it becomes clear that there is no mathematical model that well described the sorption isotherms of all foot products. In this respect there is a need to determine the appropriate moisture sorption isotherm equation for a given product. In addition Staudt *et al.* (2013), Zhengyong *et al.* (2008), Iguedjtal *et al.* (2008) and Tillered *et al.* (2005) used the sorption isotherm to evaluate the isosteric heat of sorption of some fruits. Similarly Edoun *et al.* (2010), Bahloul *et al.* (2008), Lemus *et al.* (2008), Abdelkader *et al.* (2007), Jamali *et al.* (2006), Timoumi and Zagrouba (2005) determined the isosteric heat of sorption of some leaf legumes. In spite of that, in the literature, work relating to the isotherms of sorption of safou is almost non-existent.

The aim of this study was to determine the isotherms of water desorption of safou pulp at 25, 35, 45 and 55 °C, to test the sorption isotherm model that best describe the experimental data, model in order to determine the water content of the molecular mono layer, the determine the monolayer moisture content and the heats of desorption (Qd) of safou fruit.

MATERIELS AND METHODS

Safou fruits *(Dacryodes edulis*) were collected from Ngaoundere (located at 7°20' N of latitude and 13°33' E longitude, at an altitude of 6380 m), in the region of Adamaoua (Cameroun). After washing, the fruits were longitudinally cut into four sections using a stainless steel knife. The core was then discarded and the pulp kept for subsequent analysis. Selected fruits have the following characteristics: mean fruit weight 68.7±0.2 g; mean pulp weight 45.4±0.1 g; relative pulps percentage 66.1%; mean fruit length 7.1±0.1 cm; mean pulp thickness 4.5±0.4 mm.

Experimental Design and Methodology

The equilibrium moisture content of safou pulps at 25, 35, 45 and 55 °C were determined using the static gravimetric method. These temperatures range are generally those used for the drying of foodstuffs. In the procedure, six saturated salt solutions were used to create relative humidity in 2 L volumetric glass jar. These solutions were LiCl, $MgCl_2$, K_2CO_3, KI, NaCl and KCl. The glass jars were one half filled, tightly closed and stabilized in a controlled temperature for 24 h. Table 1 gives the water activity for each temperature and saturated salt, hence the relative humidity of the surrounding environment.

Duplicate fresh safou pulp weighting 0.70±0.02 g was used for the experiment. For each desorption test, safou sample was suspended on the top of the solution using a perch. The various jars were then placed in a temperature controlled enclosure. The weights of the pulp were determined every day after the 12th day using a Satorius balance (precision 0.01 g). The moisture content was considered as equilibrium when variation from two consecutive weight measurements was less than 0.01 g. In all cases, 17 days was adequate to achieve equilibrium. The dry matter content of samples at equilibrium was determined following the AOAC (1990) drying method.

Modeling of Desorption Isotherms and Statistical Analysis

The models used in this study are presented in Table 2. They were selected based on their regular use on the moisture isotherm studies of oil seeds (Noumi *et al*., 2006 on aiele; Nkouam, 2006 on Shea tree and aiele; Bup, 2008 on shea tree; Reza, 2010 on sesame). The values of the parameters were determined from the nonlinear regression using the software Table Curve 5.1. The coefficient of correlation (r) and the standard error SEE of the moisture were used to evaluate the ability of each model to fit the experimental data (Noumi *et al*., 2004; Edoun *et al*., 2010; Ilkay, 2011).

$$r = \sqrt{\frac{\sum_{i=1}^{N}\left(X_{cal} - \bar{X}_{exp}\right)^2}{\sum_{i=1}^{N}\left(X_{exp} - \bar{X}_{exp}\right)^2}} \quad ...(5)$$

$$SEE = \sqrt{\sum_{i=1}^{N}\frac{\left(X_{exp} - X_{cal}\right)^2}{df}} \quad ...(6)$$

Table 1: Water Activity of Saturated Salt Solutions at 25, 35, 45 and 55 °C (Tillered *et al*.. 2005)

Solutions Saltworks	With $_w$			
	25°C	35°C	45°C	55°C
LiCl	0,11	0,11	0,11	0,11
$MgCl_2$	0,33	0,32	0,31	0,2 3
K_2CO_3	0,46	0,43	0,43	0,40
KI	0,69	0,65	0,65	0,64
NaCl	0,75	0,75	0,75	0,74
KCl	0,88	0,82	0,82	0,81

The mean moisture content of the product ($\bar{X}_{exp}$) was calculated using the equation:

$$\bar{X}_{exp} = \frac{1}{N}\sum_{i=1}^{N} X_{exp} \quad ...(7)$$

with $\bar{X}_{exp}$ the mean moisture content,

X_{exp} the experimental equilibrium moisture content,

X_{cal} the calculated equilibrium moisture content,

N the number of experimental points,

df the degree of freedom of the regression model, {df = N – n}, n number of variables of each model.

Determination of the Heat of Desorption

The heat of desorption was calculated from the following relation derived from the Clausius-Clapeyron equation (Do Amaral *et al.*, 1999; Edoun *et al.*, 2010):

$$\frac{\partial \ln(a_w)}{\partial(1/T)} = -\frac{Q_d}{R} \quad ...(8)$$

where a_w is the water activity, T is the absolute temperature (K), R is the universal constant gas (0.4689 KJ/kg K).

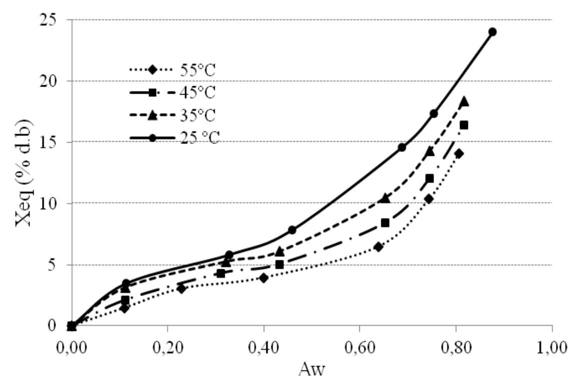
By plotting $\ln(a_w)$ against 1/T, the slope ($-Q_d/R$) is determined and Q_d, the net isosteric heat of desorption (KJ/kg), was calculated at each moisture content.

RESULTS AND DISCUSSION

Isotherm of Desorption of Safou Pulp

The isotherms of desorption of safou pulp obtained at 25, 35, 45 and 55 °C are presented in Figure 1. As expected the equilibrium moisture content generally decreases with the increase in temperature. This behavior is usually explained by the increase in the thermal agitation which activates the water molecules, increasing their distance apart and decreasing the attractive forces between them, leading to the weakening of the adsorption to the food material and consequently their reduction in food (Nkouam, 2006; Ferradji *et al.*, 2008). This result is in agreement with observation made on all type of food stuff including oilseeds such as aiele, shea tree (Noumi *et al.*, 2004; Nkouam, 2006; Bup, 2008) and sesame seeds (Reza, 2010).

Figure 1: Isotherms of Desorption of Safou Pulp at 25, 35, 45 and 50°C



The desorption isotherm of safou pulp is typical to must food stuffs with the first region of the isotherm curve, $0.00 < a_w < 0.40$, corresponding in the product to the monolayer where the water molecules are strongly fixed on the primary adsorption sites by several hydrogen bonds (Ferradji *et al.*, 2008; Moraes and Pinto, 2012). Water molecules in the monolayer are characterized by a high heat of desorption value, cannot move easily and consequently cannot participate to reactions neither as solvent nor substrate (Matallah, 2004; Ferradji *et al.*, 2008). The second region of the isotherm is for aw range 0.40-0.65 where the curve is of linear form, corresponding to the multilayer film. In this region, additional water adsorb over the monolayer water by hydrogen bonds is available for reaction and can easily escape from the product. In the third part of the curve (aw>0.65) an increase in moisture content led to an exponential increase in the water contents. In fact at high water activity, water is in the liquid form and is not adsorb on the surface of the product (Matallah, 2004). Due to the delimitation of it's desorption isotherm into 3 zones, safou pulp was classify as type II according to the BET classification. This is in agreement with several agricultural products such as sesame seeds (Reza, 2010), soya beans (Reza *et al.*, 2010), *Gnetum africanum* (Edoun *et al.*, 2010), fruits of aiele (Noumi *et al.*, 2004), potato (Amaral *et al.*, 1999) and dry apricots (Ferradji *et al.*, 2005).

Modeling of the Isotherm of Desorption of Safou Pulp

In order to find the model which describes as well as possible the isotherms of desorption of safou pulp, four mathematical models were tested (Table 2). The values of the parameters of these various models tested are deferred in Table 3. It can be seen that the GAB model presented the highest coefficient of correlation (r=0.998 at 45°C) and the lowest standard error (SEE=0.24 at 45°C). The coefficient of correlation and the standard error ranges were respectively 0974-0.998 and 0.24-2.43. It can then be recommended that GAB model described best the desorption isotherms of safou pulp. This result is in conformity with those reported for many agricultural products such as potato (Amaral *et al.*, 1999), aiele (Noumi *et al.*, 2004), dry apricots (Ferradji *et al.* 2005) and apples (Ferradji *et al.* 2008).

In Figure 2 are adjusted the experimental points and calculated points from GAB model at various 25, 35, 45 and 55°C. It can be seen from the figure

Table 2: Models Applied to the Experimental Desorption Data of Safou Pulp

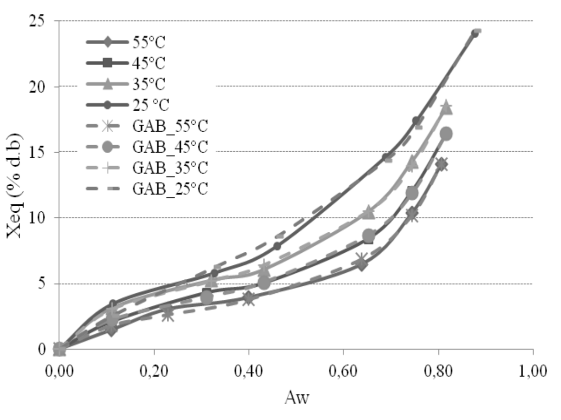
Models	Equations	Estimed paramters	References
BET	$Xeq = \frac{(X_m C a_w)}{(1 - a_w)(1 - a_w + C a_w)}$ ...(1)	X_m, C	Ferradji *et al.*, 2008
Oswin	$Xeq = C_1 \left(\frac{a_w}{1 - a_w} \right)^n$ ...(2)	C_1, n	Figen *et al.*, 2000
Halsey	$a_w = \exp\left(-\frac{A}{Xeq^b} \right)$ ...(3)	A, b	Figen *et al.*, 2000
GAB	$Xeq = \frac{X_m C K a_w}{(1 - K a_w)(1 - K a_w + C K a_w)}$ ...(4)	X_m, C, K	Ferradji *et al.*, 2008

Note: Xeq equilibrium moisture content expressed in g/100g dry matter; X_m monolayer moisture content expressed in g/100g dry matter; a_w water activity; C, C_1 K, A, b, n are model constant parameters.

Table 3: Estimate of the Parameters Of The Models Tested

Models	Estimated parameters	Desorption			
		55°C	45°C	35°C	25°C
GAB	X_m	2,48	3,21	4,07	7,20
	K	1,03	0,99	0,96	0,83
	C	14,48	13,37	16,00	4,59
	R	0,996	0,998	0,998	0,995
	SEE	0,35	0,24	0,27	0,68
Oswin	N	12,80	11,05	10,19	9,38
	C_1	0,51	0,55	0,54	0,49
	R	0,987	0,986	0,993	0,992
	SEE	1,43	1,26	0,62	0,82
Halsey	A	40,79	24,08	20,80	25,11
	B	1,61	1,49	1,49	1,62
	R	0,984	0,991	0,994	0,980
	SEE	1,55	1,03	0,69	1,63
BET	Xm	2,24	3,07	3,20	3,42
	C	9,30	16,24	19,20	42,64
	R	0,974	0,983	0, 975	0,976
	SEE	1,01	1,15	1,50	2,43

Figure 2: Experimental and GAB Predicted Isothermes of Desorption at Various Temperatures

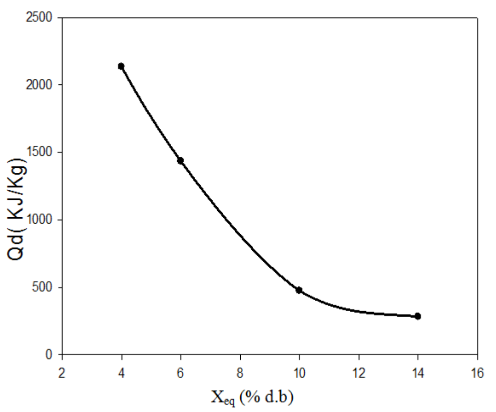


the good agreement between the experimental and theoretical points. These results are in agreement with several reports on *Gnetum africanum* (Edoun *et al*., 2010), on the shea tree and aiele (Nkouam, 2006; Noumi *et al*. 2004) on grape, apricot, potatoes and sweet potatoes (Figen *et al.* 2004). Based on the BET and GAB models, the values of the monomolecular moisture content (X_m) reported in Table 3 were determined. The monolayer moisture content represents the quantity of water for which all the absorbent sites are related to the first layer of water (Ferradji *et al*., 2008). It can be seen from the Table 3 that Xm, irrespective of the model, depends significantly on the temperature. In fact Xm decreased with the increase in temperature. According to Kechaou *et al*. (1999) changes in temperature induces physical and chemical modifications leading to a reduction of the total number of active sites of the water connections. The values of X_m from the GAB model (7.19% at 25°C) were systematically lower than the BET equivalent values (3.42 % at 25°C). This difference has been attributed to the inadequacy of the BET model to adjust the experimental data (Bup, 2008). The average moisture content of safou pulp determined from the GAB model (0.042) between 40 and 70°C was lower compared to 0.058 g/g dry matter reported for potato (Do Amaral *et al*., 1999) and 0.051 g/g dry matter reported for aiele pulp (Noumi *et al*., 2004).

The Net Isosteric Heat of Desorption

The net isosteric heat of desorption gives an estimate on the energy needed for drying and informs on the state of water in the product. The values of the net isosteric heat of desorption in the temperature range 25-55°C with reference to moisture content of the product are presented on Figure 3.

Figure 3: Experimental and Predicted Net Isosteric Heat f Desorption of Safou Pulp as Affected by Moisture Content From 4 To 14 G/100g Dry Matter



According to the Figure 3, the heat of adsorption increases when the moisture content decreases up to 4 g/100 g dry matter. The high heat observed at lower moisture content indicated the highest energy for removal of water and this might result from the higher resistance to movement of water in the pulp (Bahloul *et al*., 2008). In addition high interaction of water with active polar sites on the surface of the monomolecular layer might contribute to the high heat of desorption at lower moisture content (Nkouam, 2006). This behavior simply show that during drying, the energy needed to remove water increase as the moisture content decreases. From moisture content 4 to 10 g/100 g, the change in moisture induced a rapid change in the heat of desorption, why above 10 g/100 g little change was observed. Generally water at the monolayer is difficult to remove due to the strongly active polar sites on the surface of the product (Touati *et al*., 2007, Akkad *et al*., 2008).

The maximum of heat of desorption observed at moisture content 4 g/100 g was 2134.44 water KJ/kg, indicating that the interaction between the components of the safou pulp and water is significant (Edoun *et al*., 2010). Our value was lower compared to 4280 kJ/kg reported on shea tree Bup (2008) for moisture content between 0 and 5%, indicating that the energy needed in the desorption process is higher for shea fruit than safou pulp. A polynomial modeling was check for fitting to the experimental heat of desorption curve (Nkouam, 2006; Touati *et al*., 2007) and it was observed that the heat of desorption (Q_D) of safou pulp can be expressed as a function of moisture content (X_{eq}) by the following third order polynomial equation:

$$Q_D = 0{,}572X_{eq}^3 + 6{,}820X_{eq}^2 - 461{,}3X_{eq} + 3834$$

The experimental data of the heat of desorption of the water of the safou highly correlated with satisfaction (r=1) to the theoretical values.

CONCLUSION

The objective of this was the determining of the desorption isotherms of safou fruits at four temperatures. It can be concluded from the results obtained that the isotherms of desorption of safou fruits are of type II. The GAB model well describes the isotherm behavior in the range of temperature and moisture content investigated. The heat of desorption of safou fruits estimated by the equation of Clausius-Clapeyron is less than 2134.44 water kJ/kg, indicating significant interaction between water and the product sites at low moisture content. These results represent a basic contribution to the mastering of the processes and techniques of conservation of Safou fruits.

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APPENDIX

Nomenclature	
a_w	Activity of water
M_s	Dry matter
Q_d	Heat of desorption
X_{exp}	Experimental moisture content
X_{cal}	Predicted moisture content
r	Coefficient of correlation
SEE	Standard error
T	Absolute temperature (K)
R	Constant of perfect gases (0.4689 KJ/kg K)
X_{eq}	Equilibrium moisture content (dry basis)
X_m	Monomolecular moisture content (dry basis)
d.b.	Dry basis

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