

MODELLING MOISTURE SORPTION ISOTHERMS OF *RHECKTOPHYLLUM CAMERUNENSE* VEGETABLE FIBER

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ABSTRACT

The aim of this work was to modelling the moisture sorption isotherm of the *Rhecktophyllum Camerunense* (RC) fiber at 23°C by using the BET, GAB and DLP models. The fibers samples of 0.1g were submitted to hygro-thermal ageing in an environmental enclosure of 23%, 54% and 75% relative humidities at 23°C in accordance with NF EN ISO 483: 2006-01 standard. After 7 hours of exposure the equilibrium moisture was reached and gravimetric measurements were achieved on the specimens. The maximum moisture content was calculated. Their isotherms were modelled by interpolating the experimental datas of the maximum water content as a function of the relative humidity with the BET, GAB and DLP equations. The isotherms presented the sigmoid shape of type II. The parameters of those mathematical models were also deduced and the goodness of fit have been evaluated. The DLP model gave an excellent adjustment.

Keywords: *Rhecktophyllum Camerunense*, sorption isotherms; equilibrium state, water activity, GAB, BET and DLP models.

1. INTRODUCTION

The interest in natural fibre reinforced polymer composites is growing rapidly due to its high mechanical performances, low cost and ease of processing. Natural fibers are relatively cheap, pose no health hazards and offer solution to environmental pollution by finding new uses for waste materials [1]. However, their applications are still limited due to several factors like moisture absorption, poor wettability and large scattering in mechanical properties [2], [3]. Natural fibers and its composites are hydrophilic materials. Moisture content in fiber composites significantly affects their physical and mechanical properties. The absorbed moisture results in to the deterioration of mechanical properties since the water not only affects the unfilled polymer matrices physically and/or chemically but also attacks the hydrophilic natural fiber as well as the fiber-matrix interface [3]. To completely understand water relations in a product requires an understanding of the amount of water (moisture content) that can be held at a given energy state (water activity). Moisture sorption isotherms describe the relationship between water activity and moisture content at a constant temperature. The nature of this relationship depends on the interaction between water and other ingredients. The amount of water vapor that can be absorbed by a product depends on its chemical composition, physical-chemical state, and physical structure. Consequently, the isotherm shape is unique to each product type due to differences in capillary, surface, and colligative effects. Products that lie in the low water activity portion of the isotherm are often referred to as dry, those in the range of 0.60 a_w to 0.90 a_w are intermediate moisture products, and those having water activities higher than 0.90 are high water activity products. For ease of interpretation, isotherms are often identified by Brunauer classifications. Most food and pharmaceutical products fall under type I, II, or III. Type I isotherms are typical of very hygroscopic

materials, Type II (sigmoidal) isotherms are typical for intermediate moisture products, and type III (J-shaped) isotherms are typical for crystalline and coated materials. These general classifications proved useful when conducting isotherms on every product was not feasible due to time and labor constraints. However, with automation and improved speed, isotherms can easily be conducted on any product and the uniqueness of each isotherm often proves more valuable than placing them in a common classification. Constructing an isotherm consists of collecting water activity and moisture content values over a range of water activities. The range of water activities used depends on the situation, but normally is 0.10 a_w up to 0.90 a_w . For experiment, we can use two methods: Traditional Desiccator Method and Dynamic Dewpoint Isotherm Method.

Several isotherm models have been proposed and compared in the literature. These models are necessary to predict the moisture content at a given water activity and are used to evaluate thermodynamic functions of water in materials. They are also necessary when using isotherm methods with low data resolution to interpolate between isotherm data points. While there are 270 proposed isotherm models, the most commonly used models are the GAB and BET. Since the BET model is only applicable up to 0.50 a_w , the GAB model is widely accepted as the most useful for characterizing isotherms across the entire water activity range. Its coefficients also have theoretical physical meaning such as providing monolayer moisture content. A new empirical model called the Double Log Polynomial (DLP), which is an adaptation of the Chi plot [4], has proven to be even better than the GAB at characterizing complex isotherms. It is the topic of the present work. By doing gravimetric measurement and using predictive models we want to understand the sorption isotherm of RC fiber. RC plant as presented on figure 1 is the camerunense type of the *Rhectophyllum* species, a member of Araceae family. It was identified for the first time in 1981 by the botanist Colette Ntépe-Nyamè in the forests of Southern Cameroon, Nigeria and Gabon [5]. It is a fiber recently studied and the first works carried out show that it has good mechanical properties [6].



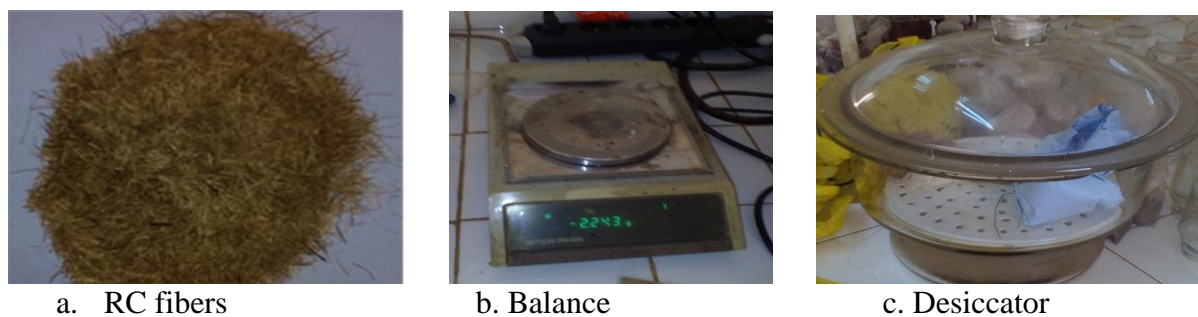
Figure1. RC plant.

Several authors have modelled the sorption isotherms of vegetable fibres as hemp and coco using BET and GAB models [7], cotton [8] and cellulose materials [9]. Some agro-food products as corn and barley have been modelled using GAB and the polynomial equation of the second degree models [10]. The red powder of curry for ten types of models and the GAB model followed by Lewiski-3 gave better adjustments [11]. The sorption isotherms of seeds of six forest tree species in Ghana were analyzed [12]. Isotherms sorption of RC and sisal were also traced [13]. All of those materials presented sigmoidal form of type II.

2. MATERIALS AND METHODS

2.1. Materials

The untreated RC fibers were used in this study. They were extracted from the roots of plants harvested in the wet equatorial forests of the center Cameroon. The roots used were fresh. We also use the thermostatic oven of Memmert mark to dry fibers. A balance PMI 400 mark with a precision of 0.001g was used to carry out the weighing. A desiccator containing silica crystals to maintain fibers at dry state. Figure 2 below presents materials used. Three chemical salts were used for preparing saturated aqueous solutions to create relative humidities in a small enclosure as mentioned in table 1. The conditioning small enclosure was made up with the plastic bottles and capsules on the grid were used to contain specimens as indicated on figures 3 and 4 below.



a. RC fibers

b. Balance

c. Desiccator

Figure2. Materials used.

Table1. Salt solutions used and their relative humidity (RH) at saturation

Saturated Salt solution	Solubility (g/l)	Quantity for 20 ml of water (g)	RH (%) at 23°C
Potassium acetate	2000	42	23
Magnesium nitrate hexahydrated	1250	27.5	54
Sodium chloride	357	8	75

2.2 Methods

The extraction of fibers was made by our care according to an unstandardized method used at the reference [10]. The adsorbed test procedure is described by NF EN ISO 483: 2006-01 standard. It was carried out in science du sol laboratory of the Faculty of Agronomic and Agricultural Sciences (FASA) of Dschang University. The saturated salt solutions were prepared with distilled water 24 hours before the beginning of the test at $23 \pm 1^\circ\text{C}$. The fibers were dried to 80°C for 48 hours and introduced into a desiccator to avoid reabsorption of ambient moisture. For each environmental condition three specimens of 0.1g were introduced in small enclosure as presented on figures 3 and 4. Gravimetric measurements were made at the saturation time and the maximum moisture content M_n were calculated by using equation (1).

$$M_n (\%) = \frac{M_t - M_o}{M_o} \times 100 \quad (1)$$

Where M_t is the mass of sample in wet state,

M_o the mass of anhydrous sample.

Table 2 contains different isotherm sorption models used in this work and their parameters.

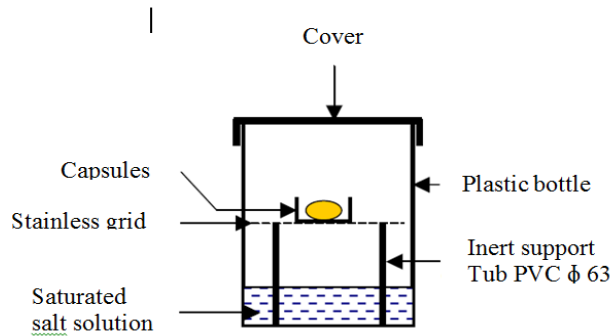


Figure 3. Experimental setup



Figure 4. Small enclosure chamber with specimens

Table 2. Different models used.

Autors	Models	Parameters
BET	$M_n = \frac{m_0 C x}{(1-x)(1+Cx-x)}$	m_0, C
GAB	$M_n = \frac{m_0 K C x}{(1-Kx)(1-Kx+CKx)}$	m_0, C, K
DLP	$M_n = b_3 \chi^3 + b_2 \chi^2 + b_1 \chi + b_0$	b_3, b_2, b_1, b_0

Where M_n is the moisture in g/100 solids or g/g solids at water activity a_w and m_0 is the monolayer value in the same units. K is a constant in the range of 0.70 to 1. The constant C is calculated by:

$$C = \exp\left(\frac{Q}{RT}\right) \tag{2}$$

Where Q is the surface interaction energy in J/mole,

R is the gas constant (8.314 J/mol K)

T (K) is the temperature

$$\chi = \ln[-\ln(a_w)]$$

The statistical analysis of experimental data have been done in the MATLAB 2014a software environment which enabled drawing experimental curves. Parameters models are deduced from the modeling and for that, we have used the nonlinear regression of Marquardt-Levenberg algorithm with the support of Curve Fitting Toolbox of MATLAB software. Models will fit well the experimental data if it has an average correlation coefficient near to the unit, the square root of mean error average near zero, and the sum of square error average near zero. These two last statistical parameters are defined by the following equations (3) and (4):

$$RMSE = \sqrt{\frac{\sum_{i=1}^n (m_{ci} - m_{ei})^2}{n}} \tag{3}$$

$$SSE = \sum_{i=1}^n (m_{ci} - m_{ei})^2 \tag{4}$$

where m_{ci} , m_{ei} , and n are respectively the theoretical mass, the predicted mass, and the number of observation respectively.

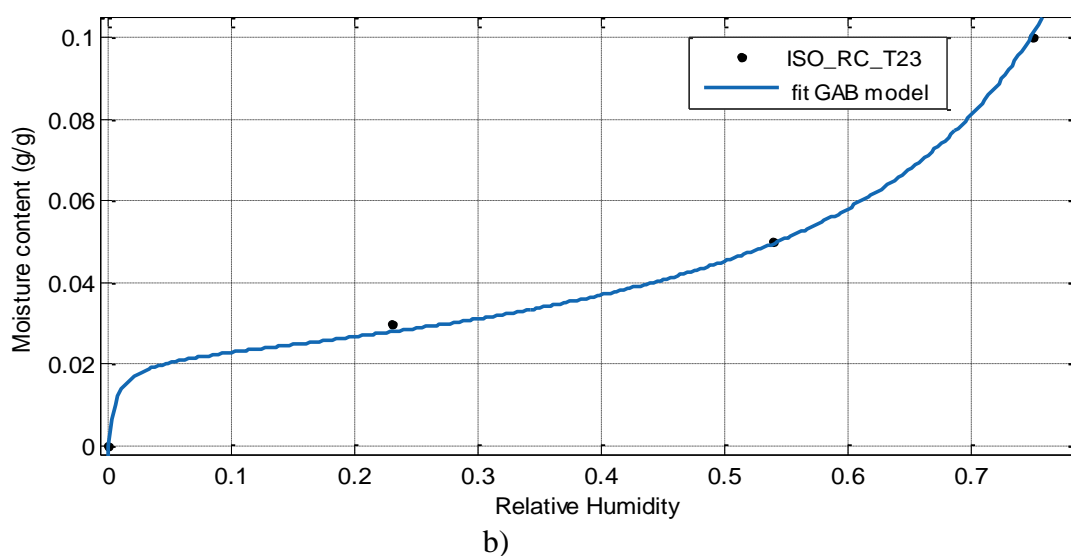
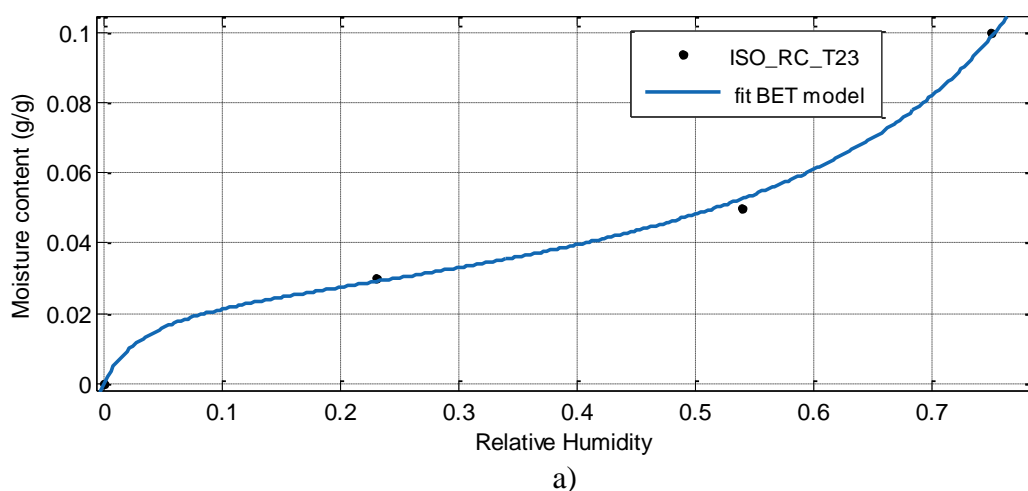
3. RESULTS AND DISCUSSION

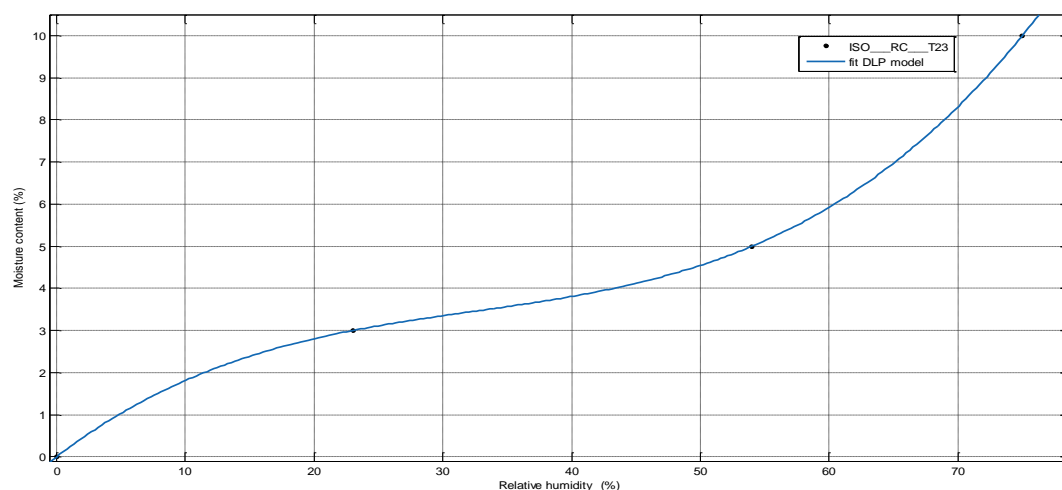
The experimental data of the maximum water content according to the relative humidities are presented in table 3 below. The experimental data of the RC fiber sorption isotherm indicated that the equilibrium moisture content increase with the relative humidity what is characteristic of microporous materials and granular medium. The moisture absorption of fibres resembles the multi-layer absorption in the water - lignocellulosic fibre systems like

cotton, sisal, hemp, coconut and wood [7]. The m_0 , C and K parameters are also closed to those fibers. This means that they have the same mechanism of fixing water molecule. The evolution of the moisture content of this fiber is the sigmoid shape of type II as presented at figures 5a, 5b and 5c below. This result agrees with those obtained in the literature [7], [8], [9], [10], [11], [12] and [13]. Good correlation between experimental datas and model was found, as indicated by the values on tables 4 and 5. The values of calculated correlation coefficients for the water absorption curves are higher than the critical value of the correlation coefficient for a set of measured values at elected significance level $p = 0.05$. The correlation coefficient for BET and GAB model are strongly significant. The DLP model is excellent. It is thus concluded that DLP model gave us the best adjustment.

Table 3. Average value of the moisture content versus the relative humidity at 23°C

Humidité relative (%)	Teneur moyenne en humidité (g/g)
0	0
23	0,032
54	0,049
75	0,104





c)

Figure 5. Sorption isotherms according to models of a) BET, b) GAB and c) DLP.

Table 4. Parameters of BET and GAB models for the sorption isotherm

model	Coefficients					
	m_0	C	K	R^2	SSE	RMSE
BET	0.02499	28.76	/	0.9981	9.904e-06	0.002225
GAB	0.02188	14.95	0.9999	0.9997	1.519e-06	0.001233

Table 5. Parameters of DLP model for the sorption isotherm

b_3	b_2	b_1	b_0	R^2	SSE	RMSE
0.4387	0.3816	0.7655	0.7952	1	5.039e-32	/

CONCLUSION

Modelling the moisture sorption isotherm of the RC fiber at 23°C by using the BET, GAB and DLP models have been done in this work. Traditional Desiccator Method have been used for the treatment. The samples fibers were submitted to hygro-thermal ageing in an environmental enclosure of 23%, 54% and 75% relative humidities at 23°C and gravimetric measurements were done. As the majority of vegetable fibers the moisture content increases with relative humidity. Tree model have been used to model the sorption isotherm. All those model present the type II which is characteristics of vegetable fibers and granular media. The correlation coefficient for BET and GAB model was strongly significant but the DLP model give the best adjustment with experimental data. The acceptable validity of the DLP model also states that the moisture absorption of fibres resembles a multi-layer absorption in the systems water - lignocellulosic fibre like cotton, hemp, coconut and wood.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

REFERENCES

- [1] Bongarde U.S. and Shinde V.D. (2014). Review on natural fiber reinforcement polymer composites, International Journal of Engineering Science and Innovative Technology, Vol. 3, issue 2, pp. 431-436.
- [2] Amar, K.M., Manjusri, M. and Lawrence, T.D. (2005). Natural Fibers, Biopolymers, and Bio composites. CRC. Press, Taylor & Francis, pp. 1-3.
- [3] Bledzki A.K., Gassan J (1999) Composites reinforced with cellulose based fibres. Progress in Polymer Science, 24, pp. 221–274.
- [4] Condon, J.B. (2006). Theories behind the chi plot. p. 91-125. In J.B. Condon (ed.) Surface area and porosity determinations by physisorption. Elsevier, Oxford.
- [5] C. Ntéppé-Nyame. (1981). Une nouvelle espèce pour le genre rhextophyllum. Adansonia, 20(4) :451–457.
- [6] A. Béakou, R. Ntenga, J. Lepetit, J.A. Atéba, L.O. Ayina, (2008). Physico- chemical and microstructural characterization of Rhextophyllum camerunense plant fiber. Composites part A: applied science and manufacturing journal: 67-74.
- [7] Ho Thi, Thu Nga (2008), Etude de l'influence de la température et de l'humidité sur les propriétés mécaniques en traction des fibres de chanvre et de coco, Ph.D thesis, université du Québec.
- [8] S. L. Gupta and R. K. S. Bhafia, (1971). "Studies in sorption – desorption hysteresis of water vapour with raw and modified cotton cellulose," *Textile Research Journal*, vol. 41, pp. 97-100.
- [9] H. W. Haslach, J. Pecht, and M. G. Pecht, (1989). "Thermodynamic modeling of load-sorption relations in cellulosic materials," *Mechanics of Cellulosic and Polymeric Materials*, vol. 99, pp. 15-22.
- [10] Neagu Corina, Tofan Clemansa, Borda Daniela. Mathematical models for moisture sorption isotherms of barley and wheat. Paper presented at the International Symposium Euro - aliment 2009, 9th – 10th of October 2009, Galati – ROMANIA
- [11] Sudathip Inchuen, Woatthichai Narkruga and Pimpen Pornchaloempong. Moisture sorption of Thai red curry powder. Maejo International Journal of Science and Technology ISSN 1905-7873
- [12] J. M. Asomaning, Moctar Sacande and Nana S. Olympio, (2011) West African Water Sorption Isotherm Characteristics of Seeds of Six Indigenous Forest Tree Species in Ghana Journal of Applied Ecology, vol. 18,.
- [13] Betene E. F. (2012). Etude des propriétés mécaniques et thermiques du plâtre renforcé de fibres végétales tropicales. Ph.D thesis. Université Blaise Pascal - Clermont-Ferrand II.