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Study of the diffusion behavior of water vapor sorption in natural fiber: *Rhecktophyllum camerunense*

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ABSTRACT

The present work focuses on the study of the water adsorption phenomenon in *Rhecktophyllum camerunense* fiber. Indeed the diffusive behavior of this natural fiber was investigated. 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±1°C. Periodic gravimetric measurements were achieved on the specimens in order to follow their kinetics adsorption. The results show that the kinetic adsorption is rapid at the first moments no matter the relative humidity and begin saturation at the seventh hour. The maximum moisture content increases with the relative humidity. Predictive Fick Law was used to modelling experimental data. The curve of all humidities obeys to the Fick model and the diffusion coefficients have been deduced. We observed that the diffusion coefficient was an increasing linear function of the relative humidity.

Keywords: *Rhecktophyllum camerunense*, diffusion, moisture, Fick law

1. INTRODUCTION

Plant fibers already used in textile could act as reinforcement in composite materials. Those types of natural fibers are suitable alternative to glass fibers for reinforcing polymeric matrix. They are light, abundant, biodegradable and renewable. Moreover, they exhibit appreciable specific mechanical properties compared to glass and other synthetic fibers [1]. For their use as reinforcement, the hydrophilic nature of plant fibers has to be considered with carefulness for several reasons. First, during the lifecycle of the material, water absorption could induce a volume change of the fibers inside the composite, leading to the development of internal stresses. On the other hand, during the polymerizationprocessofthematrixabove100◦C, a vaporization of water trapped inside fibers could occur, leading to their shrinkage. These swelling and shrinkage of the fibers surrounded by the matrix generate internal stresses at the fiber/matrix interface and can eventually lead to the damage of the latter and to a significant degradation of the initial properties of the composite. The works of Rangaraj and Smith [2]; Dhakal et al. [3]; LeDuigou et al. [4]; Huetal. [5]; Assarar et al. [6] deal with water sorption of composites reinforced by bio-based fibers. For example, in their work on the water uptake of a flax fiber composite material, Assarar et al. [6] showed an increase of the water content absorbed, compared to material consisting of the same matrix reinforced with glass fibers. LeDuigou et al. [4] studied the behavior of a composite PLLA/flaxin immersion in seawater. The weight gain curves showed the influence of the cellulose fibers. The saturated moisture contents of the specimens were around 5.6%. Secondly, Le Duigou et al. [4] showed loss of the mechanical properties of the composite and an irreversible damage of the fiber/matrix interface during wet-ageing. To prevent this phenomenon, fiber surface properties could be modified by physical treatments (cold plasma treatment and corona treatment) and chemical treatments (maleic anhydride, organosilanes, isocyanates, sodium hydroxide, permanganate, alkaly and peroxide) in order to promote adhesion [1, 7]. A lot of investigations have been done in this sense but few people have explored the diffusion phenomenon in order to understand the mechanism of moisture adsorption in fibers [8]. It is the topic of the present work. By doing gravimetric measurement on natural wet fibers and using predictive model we want to understand the diffusion moisture behavior of *Rhecktophyllum camerunense* (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 Ntépé-Nyamè [9] in the forests of Southern Cameroon, Nigeria and Gabon. Thefibers are extracted from the long air roots and are traditionally used to make ropes, baskets and fishing nets. RC is a fiber recently studied and the first works carried out show that it has good mechanical properties [10]. The hydrophilic character of RC brings to the plaster more moisture than when it is not reinforced [11]. Recently the moisture sorption isotherm was modelling using BET, GAB and DLP equations. The isotherms presented the sigmoid shape of type II as for more vegetable fibers like cotton, hemp, coconut, wood and the parameters of those models were deduced [12].

Figure 1 RC plant [12]

The moisture diffusion in some fibers has been studied like hemp and coco [12], hemp, flax, jute and sisal, ramie [14] and flax [15, 16]. Celino et al [14] used Fick and Langmuir models to describe the diffusion behaviour of sisal, jute, hemp and flax fibers immersed in water at room temperature and exposed to vapor humidity at 80% RH and 23°C. Despite their different origins, the four natural fibers showed a similar diffusion behaviour. The hydrophilic behaviour of natural fibers and their relation with the presence of free hydroxyl group in its structure was related by many practical [16, 17] and modelling studies [8]. These authors employed physical models applied to polymers to describe the diffusion behaviour of water in natural fibers, such as Fick law [17], the Langmuir theory and dual-stage Fick law [18]. The Langmuir model was found to describe very well the diffusion inside fibers immersed in water while the Fick law better described the diffusion behavior of fibers exposed to relative humidity ageing. Gouanvé et al [8] found that the water diffusion in flax fibers exposed to hygrothermal ageing at 23°C may be explained on the basis of a combination of Langmuir and Henry sorption models at low levels of relative humidity (RH).

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 central Cameroon. The roots used were freshes. We also used the thermostatic oven of Memmert mark to dry fibers. A balance PM 400 mark with aprecision of 0.001g were used to carry out the weighing. A desiccator containing silica crystals to maintain fibers at dry state. Three chemical salts were used for preparing saturated aqueous solutions to create RH 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 figure 2 below.

(a). RC fibers (b). Balance (c). Desiccator

Figure 2 Materials used

Table 1 Salt solutions used and their RH at saturation [12]

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 test procedure is described by NF EN ISO 483: 2006-01 standard. It was carried out in science du sollaboratory 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±1°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 a small enclosure as presented on figures 3 and 4. Periodic gravimetric measurements were achieved until the saturation period was reached in order to observe the kinetic adsorption of moisture on the specimens. After the saturation, fibers were maintaining in their environment many weeks ago to control any variation of moisture content.

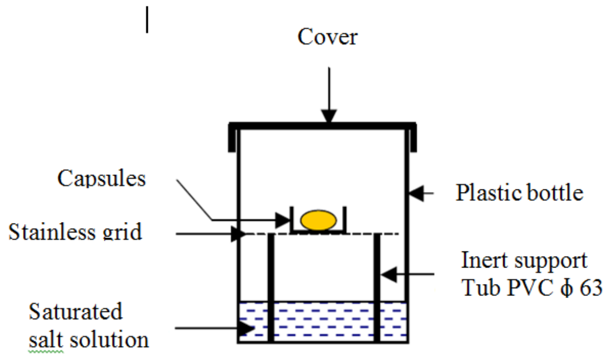

Figure 3 Experimental setup

Figure 4 Small enclosure chamber with specimens

The mass of water gain X is calculated for each relative humidity according to the following expression:

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

Where M_t is the mass of sample in wet state,
M_0 the mass of anhydrous sample.

This equation is used to study the kinetic absorption of moisture in vegetable fibers and other materials by observing the curve of water content as a function of the time.

There are three major mechanisms of moisture absorption in natural fiber composite. First diffusion of water molecules inside the microgaps between polymer chains; second the capillary transport of water molecules into the gaps and flaws at the interface between fibers and the polymer due to the incomplete wettability and finally the third mechanism is the transport of water molecules by micro cracks in the matrix, formed during the compounding process.These three cases of diffusion can be distinguished theoretically by the shape of the sorption curve represented by:

$$\frac{M_t}{M_n} = kt^n \qquad \text{And}$$

$$\log\left(\frac{M_t}{M_n}\right) = \log(k) + n\log(t) \qquad (2)$$

Where, M_t, M_n, k, and n are the water absorption at time t, the water absorption at the saturation point, and constants, respectively. The coefficients (n and k) are calculated from slope andthe value of n is different for each caseintercept of log plot of M_t/M_n versus time which can draw from experimental data. When n = 0.5 following Fickian behavior. For non-Fickian diffusion, the value of n is between 0.5 and 1. When the value of n is less than 0.5, anomalous diffusion takes place.

The experimental data are fitted with the traditional Fickian diffusion model [17]. This is the most common model used for the diffusion of humidity in the materials. The model is consistent on the so-called free volume theory [18].Considering a long circular cylinder in which diffusion is everywhere radial (one dimensional case), the moisture concentration C is then a function of radius r and time t, and the diffusion equation is written as follow [19]:

$$\frac{\partial C}{\partial t} = D\left(\frac{1}{r}\frac{\partial C}{\partial t} + \frac{\partial^2 C}{\partial r^2}\right) \quad (3)$$

Where D is the diffusion coefficient.

Indeed, the integration of the analytical solution of Eq. (3), over the cylinder radius r yields the following expression of moisture uptake:

$$\frac{M_t}{M_\infty} = 1 - \sum_{n=1}^{\infty} \frac{4}{a^2\alpha_n^2} \exp(-D\alpha_n^2 t) \quad (4)$$

By simplify the equation (4) the diffusion coefficient D can be deduced at the initial linear stage of Fick´s curve by the following equation (5): $D=\pi\left(\frac{b}{4M_m}\right)^2\left(\frac{M_2-M_1}{\sqrt{t_2}-\sqrt{t_1}}\right)^2 (mm^2/s)$ (5)

Where b is the fiber diameter (mm)

M_m maximum moisture content(g/g)

M_2, M_1 are the moisture contents corresponding to the times t_2, t_1 of the initial linear phase of the Fick curve.

The Fick curve expresses the moisture content versus the square root of time. The first phase of that curve is linear and the second phase is curvilinear.The diffusion coefficient D expresses the speed to which the process of the diffusion takes place. It is the diffusion speed of substance per unit of section divided by the gradient of concentration in that section.

The statistical analysis of experimental data have been done in the Matlab R2014a software environment which enabled drawing experimental curves.The Fick model 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(6) [20]:

$$\text{RMSE} = \sqrt{\frac{\sum_{i=!}^{n}(m_{ci}-m_{ei})^2}{n}}$$

$$\text{SSE} = \sum_{i=!}^{n}(m_{ci}-m_{ei})^2 \quad (6)$$

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

3. RESULT AND DISCUSSIONS

Figure 5 presents the kinetic absorption of RC at the RH of 23, 54 and 75% at 23°C. Those curves have the same look for all the RH. They rather quickly absorb moisture in the first moments and start saturation at the end of seventh hour independently of ambient RH. Beyond seventh hour there is no variation even after many weeks of observations.

The moisture content increases with the RH. A similar example observed in the literature is from Betene [11] where the RC adsorbs12 % of moisture at 98% RHin spite of the fact that he observed saturation at 24h. This great difference at saturation time should be the fact of environmental conditions. The kinetic adsorption of the RC are in the same range as those observed in the literature like hemp, coco, jute, ramie, flax, and sisal [11, 13, 14, 15, 16]. The mechanism of fixing water molecules on the RC fibers surface is similar to the majority of other fibers. It is characteristic of microporous materials and the adsorption is multimolecular [11]. The low adsorption proportion moisture of RC compared to other fibers is thus highlighted. Table 2presents the coefficients parameter of diffusion. Modeling by the Fick law consisted of adjusting the experimental data of water content with respect to the square root of time by using Fick equations as presented at equations (4).

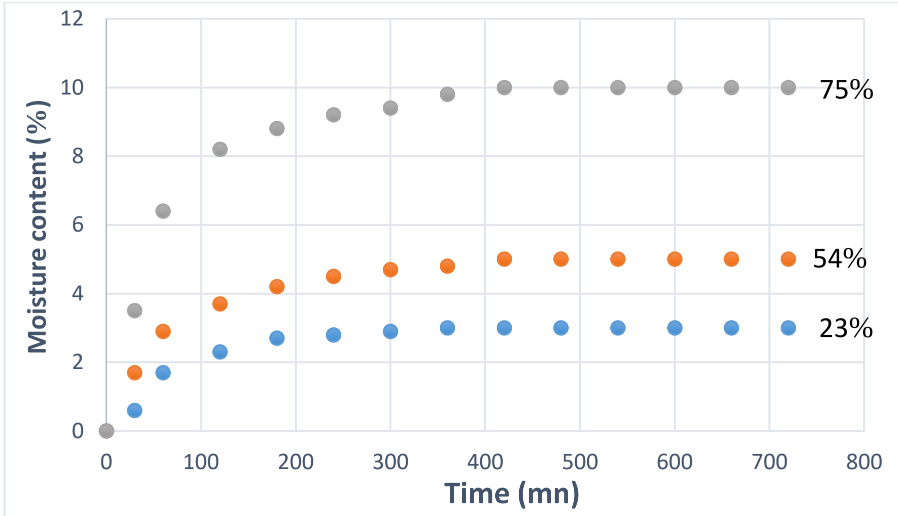

Figure 5 Kinetics adsorption of fibers at different relative humidities

Table 2 Moisture content, n and k coefficients versus RH

Relatives Humidities (%)	Moisture content Mn (g/g)	n	k
23	3	0.4892	0.111
54	5	0.4946	0.117
75	10	0.5018	0.157

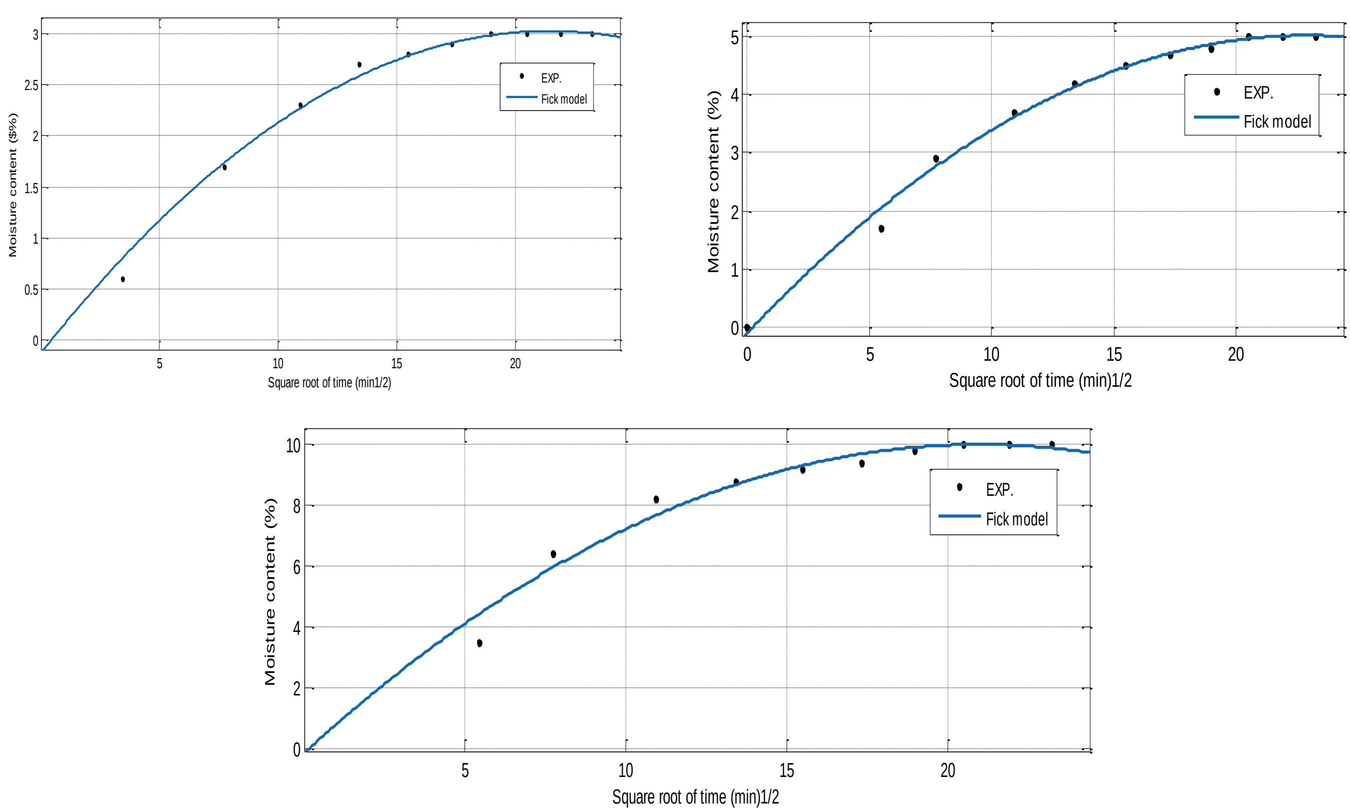

Figure 6 Modeling the experimental diffusion by Fick law **a)** 23%, **b)** 54% and **c)** 75% RH at 23°C.

Observing figure 6 we have an initial phase which is linear on a very short time and for a moisture content going around 60% of the maximum moisture content. The second phase which is nonlinear and extends over a long period is concave to horizontal axis. The shape of those curves and the values of n which are close to 0.5 according to table 2 permit to conclude that the diffusion is Fickian at all RH. The diffusion behavior in RC is characteristic of ligno-cellulosic materials. The diffusion coefficients are deduced by using equation (5) and the result are presented in table 3. They also increase with relative humidity. These values are of the same order as those of the majority of vegetable fibers encountered in the literature [13, 14, 15, 16].

Table 3 Coefficient diffusion versus relative humidity

Relatives Humidities (%)	Diffusion Coefficients D (m^2/s)	R^2	RMSE	SSE
23	0.68E-10	0.9579	0.2223	0.6423
54	0.89E-10	0.9939	0.1219	0.1933
75	1.14E-10	0.9995	0.04307	0.02411

Good correlation between experimental and model was found, as indicated by the values of correlation coefficients in table 3. The values of calculated correlation coefficients for the water absorption curves of all RH are higher than the critical value of the correlation coefficient for a set of measured values at elected significance level p = 0.05. The existence of a relationship between variables such as water content, time and RH of RC at given significance level can be regarded as proven. The diffusion coefficients can be compared with some found in literature and presented in the following table 4.

Table 4 Diffusion coefficient of some fibers in the literature

Fibers	Aigeing conditions	Diffusion Coefficients D (m^2/s)	Mn(%)	References
Flax	80%RH/ 23°C	2.0E-10	12	[13]
Hemp	80%RH/ 23°C	2.3E-10	10.5	
Jute	80%RH/ 23°C	4.0E-10	12.3	
Sisal	80%RH/ 23°C	1.2E-10	11.2	
Flax	33%RH/ 23°C	2.06E-10	-	[14]
	50%RH/ 23°C	2.79E-10	-	
	75%RH/ 23°C	3.69E-10	-	
Flax	66%RH/ 23°C	4.04E-10	-	[15]

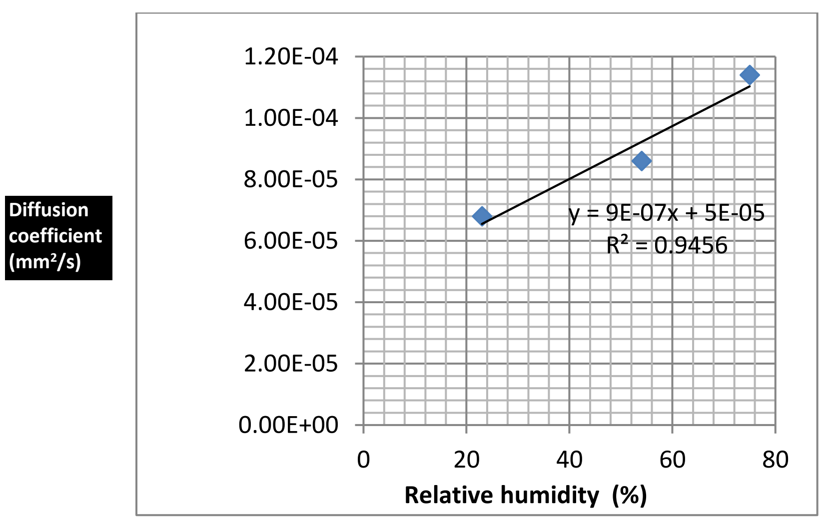

Figure 7 Diffusion coefficients versus relative humidity.

After getting the diffusion coefficients, we evaluate his behavior with respect to the RH as presented in figure 7. It appears that the RH increases with the diffusion coefficient. It is important to note that the evolution of the diffusion coefficient of water with respect to the relative humidity is linear. From the linear regression carried out on the experimental points, it is thus possible to deduce the diffusion coefficient in the RC fiber for all RH. The similar results have been observed on flax, hemp and coco fibers [13, 15, 16].

4. CONCLUSION

At the end of this study which was to evaluate the moisture diffusion of RC fibers versus the relative humidities of 23, 54 and 75% at 23°C, it comes out from this analysis that it is characteristic of ligno-cellulosic materials and vegetable fibers. Its kinetic adsorption is very fast at the first moments before going to saturation. The water content increases with the RH and the modeling of experimental data is Fickian at 23°C and finally the diffusion coefficient is an increasing linear function of the RH. From that adjustment the diffusion coefficients can be deduced at all RH. We have notice that the equilibrium adsorption of water by RC is among the lowest in spite of the fact that it is collected in the wetland. This study on the diffusion of water in fibres will be the preliminary for the type of treatment in order to return them less hydrophyl before their use as reinforcement in the composite materials.

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Conflicts of Interest: The authors declare no conflict of interest.

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