

SORPTION BEHAVIOR OF FOUR TROPICAL WOODS USING A DYNAMIC VAPOR SORPTION STANDARD ANALYSIS SYSTEM

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ABSTRACT

Sorption behavior of four tropical woods coming from Cameroon is studied using a Dynamic Vapor Sorption-Intrinsic apparatus. Sorption isotherms at 20°C and 40°C of *Triplochiton scleroxylon* (obeche), *Entandrophragma cylindricum* (sapele), *Sterculia rhinopetala* (lotofa) and *Terminalia superba* (frake also called limba) are compared to those in the literature with methods using saturated salts for lotofa and frake with satisfactory results. Experimental values are fitted using two sorption isotherm models. Sorption hysteresis is greatest in sapele and least in frake. When temperature increases, differences in the hysteresis of sorption among the woods decreases. Hystereses of all studied woods ranged from 0,5 to 3%. Our studied woods have lower equilibrium moisture content probably caused by the higher values of extractives content. The water sorbed molecules on multilayer during both adsorption and desorption at 20°C and 40°C is greater in the case of frake and lower in the case of lotofa. The water sorbed molecules on multilayer of sapele is greater than those of obeche. The predominance of multilayer over monolayer of our woods is from 0,2 to 0,4 relative humidity in adsorption phase, and from 0,3 to 0,4 relative humidity in desorption phase.

Keywords: Adsorption, desorption, equilibrium moisture content, sorption isotherm, sorption hysteresis, tropical woods.

INTRODUCTION

It is well known that tropical countries have a lot of woods species. For example, Cameroonian forests have more than 300 woods species. These woods are used in many domains such as plywood, decorative veneers, interior joinery, furniture and packaging (Fernández *et al.* 2014).

The sorption isotherms are essential properties in the analysis and design of various biological material processes such as preservation, drying (Salin 2011), storing, packaging and mixing. It can be used to determine the net isosteric heat of sorption (Ouafia *et al.* 2015), for selecting appropriate storage conditions and packing systems that optimize or maximize the mechanical stability of biological material.

Deliiski (2011) presents wood sorption isotherms, but no specific data is given on each species. Jannot *et al.* (2006) use GAB and BET models to plot desorption isotherms and estimate specific areas of doussié, moabi, ebony, obeche and iroko at 20, 30, 40, 50, 60°C. In the same temperatures previously cited, Bonoma and Simo-Tagne (2005) use Henderson and Chung-Pfost models to explain

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the evolutions of equilibrium water content in the desorption phases of ebony and obeche with relative humidity. Simo-Tagne *et al.* (2011) present a correlation between equilibrium water content in function of temperature and relative humidity and estimate the isosteric heat of sorption of obeche and ebony using GAB model. Also, they studied the Soret effect on obeche for various temperatures. Fernández *et al.* (2014) determine the sorption isotherms and thermodynamic properties of fraque and obeche at 15, 35 and 50°C.

In the literature, several authors use the standard static gravimetric method to determine the sorption isotherms (Ouafia *et al.* 2015). Description of this method is available on Jannot (2008). This method is so long to obtain an equilibrium state of the sample on a given conditions. Applied on wood material, the samples with 1mm of thickness are often used and 30 to 40 days are necessary to obtain one equilibrium state (Jannot *et al.* 2006, Fernández *et al.* 2014, Esteban *et al.* 2004, De Palacios *et al.* 2012).

Simo-Tagne *et al.* (2016) have presented the experimental EMC, mass diffusivity coefficients and air permeability of these woods. Concerning the EMC, discussion between values obtained with DVS apparatus and others methods, the monolayer saturation moisture content, the modeling of the monomolecular water sorbed and the polymolecular water sorbed are not studied in Simo-Tagne *et al.* (2016).

The objectives of present work are: (1) To use DVS apparatus to determine and analyze the sorption isotherms (adsorption and desorption) of *Triplochiiton scleroxylon* (obeche), *Entandrophragma cylindricum* (sapele), *Sterculia rhinopetala* (lotofa) and *Terminalia superba* (fraque), four tropical woods most commercialized. (2) To deduce the hysteresis of sorption of each studied wood. (3) To study the behavior of water molecules in the scale of pore and (4) to compare the values obtained in this work by those obtained in the literature.

MATERIALS AND METHODS

Materials Preparation

All tests presented here have been done in the Studies and Research on Wood Material Laboratory (LERMAB) of University of Lorraine, located in Epinal, France.

As Engelund *et al.* (2010) and Jalaludin *et al.* (2009), isotherms were performed using a dynamic gravimetric water sorption analyzer from Surface Measurement Systems (DVS-Intrinsic) (Figure 1). Specimens were collected from natural forests of Douala Cameroon and from mature log. The specimens used were very fine wood-chips cut with a microtome blade to obtain samples on each board with dimensions approximately 1x0,5x2mm.

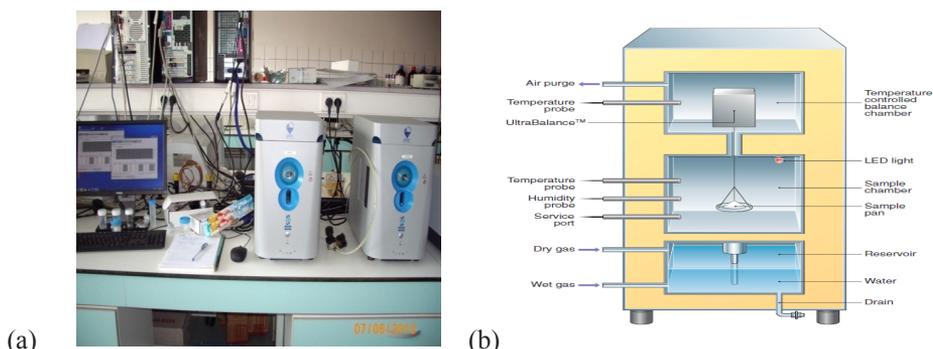


Figure 1. DVS apparatus used to measure different sorption isotherms. (a) Picture; (b) Schematic of the main components of the DVS intrinsic (Dynamic Vapor Sorption, surface measurement systems 2015).

Test Conditions

The sample is weighted using a digital microbalance (total capacity of 1 g; noise < 1 µg). Approximately 26 mg of each sample were used for this measurement and two tests are made on each species. The sorption cycles applied in this work started from 0% relative humidity (RH) to 90% RH. The dry mass is determined at the end of this plateau. Isotherms were made at 20°C (± 0,02 °C) and 40°C (± 0,02 °C). Samples were maintained at a constant RH level until the weight change per minute (dm/dt) value reached 0,0005% per minute. Figure 2 is an example of evolutions of RH and weight change with the time in the case of sapele at 20°C. We see that this experiment (adsorption and desorption) was made around 4500min. The raw DVS data was first analyzed using the DVS Standard Analysis System. This software permits construction of isotherm and hysteresis curves. For all samples, Equilibrium Moisture Content values were determined using the last fifteen data of each plateau.

Sorption Analysis

The Hailwood-Horrobin sorption model (HH-model) (Hailwood and Horrobin 1946) and the Guggenheim, Anderson and De Boer-Dent model (GAB-model) (Ouafia *et al.* 2015) are used for describing the envelope curves (desorption and adsorption isotherms from the extreme values 0% and 90% of RH) because the agreement of the measured values with the computed isotherms are usually very good (Fernández *et al.* 2014, Popper *et al.* 2009). Mathematical equations of these models are briefly recalled on Table 1.

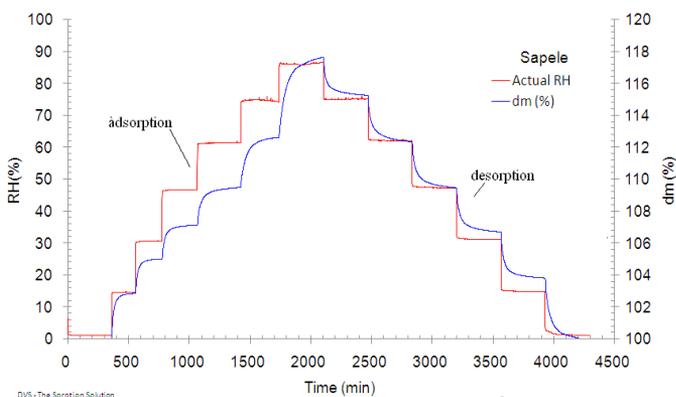


Figure 2. Air relative humidity and weight change versus time, Sapele, 20°C.

Table 1. Equations used to model equilibrium desorption and adsorption isotherms.

Names and References	Equilibrium moisture content model
HH-model (Hailwood and Horrobin 1946)	$X_{eq} = \frac{K C X_m RH}{(1 - K RH)(1 - K RH + C K RH)}$
GAB-model (Ouafia <i>et al.</i> 2015)	$\frac{RH}{X_{eq}} = A + B RH - C_0 RH^2$

With:

X_{eq} : Equilibrium moisture content (%); X_m : Monolayer saturation moisture content (%). The

insignificant values of X_m parameter may result from the weak presence of the active sorption sites available within the wood cell walls; C: Guggenheim constant (-) which corresponds to the total sorption heat of the first sorbed layer (Alix *et al.* 2009); K: Constant associated to water sorbed molecules on multi-layer (-) (Alix *et al.* 2009); RH: Water activity or Relative humidity on a scale of zero to one (-).

A, B and C_0 are the shape factors. We can use these values to estimate the equilibrium between the activities of absorbed water and water vapor (K_d), the equilibrium between the activity of hydrated wood over the activity of dry wood and absorbed water (K_h) and the apparent molecular weight of wood per sorption site W_i (Dieste 2010). We have (Dieste 2010):

$$K_d = \frac{50 \left(-B + \sqrt{B^2 + 4AC_0} \right)}{A} \quad (2a)$$

$$K_h = 1 + \frac{B \left(B + \sqrt{B^2 + 4AC_0} \right)}{2AC_0} \quad (2b)$$

$$W_i = 1800 \sqrt{B^2 + 4AC_0} \quad (2c)$$

A low K_h implies a decrease in the activity of hydrated wood with respect to both the activities of dry wood and dissolved water, the increase of W_i is taken as evidence for a decrease in the number of active sorption sites. The HH model is composed into the adsorbed water or monomolecular water sorbed (X_h) and the absorbed water or polymolecular water sorbed (X_d). We have (Dieste 2010):

$$X_{eq} = X_h + X_d \quad (3)$$

$$X_h = \frac{1800K_dK_hRH}{W_i(1 + K_dK_hRH)} \quad (4a)$$

$$X_d = \frac{1800K_dRH}{W_i(1 - K_dRH)} \quad (4b)$$

X_d in %, X_h in % and X_{eq} in %.

Monolayer moisture is the water within the cell wall that is associated closely with the OH groups, while polylayer water is when water occurs within the transient microcapillaries but is not intimately associated with the OH groups (Hill 2009).

The sorption isotherm fits were considered valid if the correlation coefficient (R) was superior to 0,990 (Fernandez *et al.* 2014). The water taken up by the sample via monolayer sorption from RH_m to 1RH is given by (Esteban *et al.* 2006):

$$X_{up} = X_m - X_{ref} \quad (5)$$

Where X_{ref} is the moisture content corresponding to RH_m that gives the minimum of the derivative of equilibrium moisture content in relation to the RH.

RESULTS AND DISCUSSION

Figure 3 presents the measured data obtained in this study at 20°C and 40°C respectively. It is clear that the sorption behavior of tropical species is different and apparatus used does not permit the obtaining an idea on the sorption behavior when water activity is above 90%. But, during the conservation, storage and packaging of finished products, air relative humidity used are below 90%. Both adsorption and desorption phase, obeche has higher equilibrium moisture content (EMC) values compared with others, fraike has lower EMC. EMC of sapele is higher than the one of lotofa.

The temperature dependence of the EMC seems to be weak (Figure 3). This influence of temperature is conform to the results obtained by Rodriguez-Ramirez *et al.* (2011) and Björk and Rasmuson (1995). Also, we noticed that differences on our EMC are probably caused by the differences of extractive content, history of the sample, the wood structure and the part of the sample in the log.

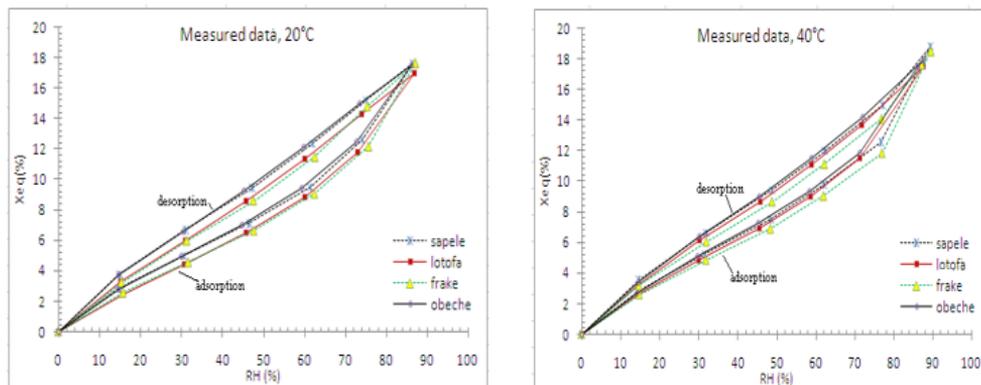
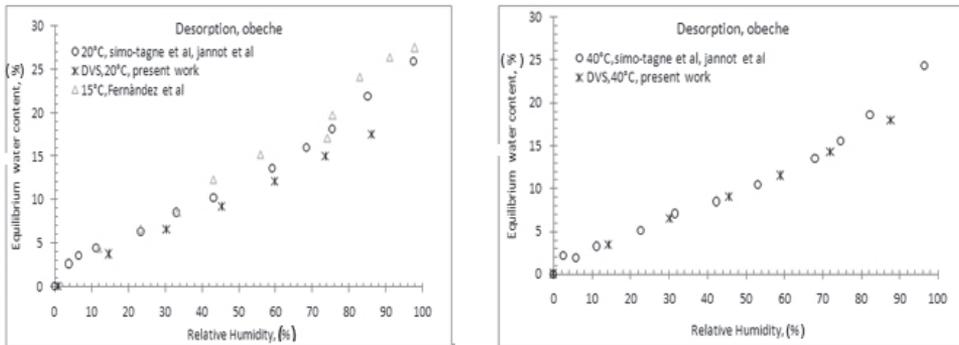


Figure 3. Measured data of sorption isotherms given by our DVS system.

Figures 4 and 5 present a comparison between results obtained in this work and those of the literature using standard static gravimetric method which requires a lot of time for reaching the hygroscopic equilibrium. On the Figure 4a, we see that present results at 20°C are lower than those obtained by Fernández *et al.* (2014) at 15°C, because EMC decreases when temperature increases (Esteban *et al.* 2004, Fernández *et al.* 2014, Jannot *et al.* 2006, Simo Tagne *et al.* 2011). But, we see a weak difference between present values and those obtained in the literature (Jannot *et al.* 2006, Simo-Tagne *et al.* 2011) on obeche at 20°C in desorption phases. It is probable that the origin, history of each sample used and natural variability from the species are the reason of this difference. Figure 4b gives a good agreement between present results and those obtained in the literature (Jannot *et al.* 2006, Simo-Tagne *et al.* 2011) on obeche at 40°C in desorption phases. Figure 5 shows that our results at 20°C are lower than those obtained on the same wood species (fraike) at 15°C.

Figure 6 presents hysteresis of sorption given by present results. We see that at 20°C, hysteresis of sorption are greater than those at 40°C. Hysteresis increases when relative humidity increases. Tekleyohannes (1995) had obtained the same results. Sapele has higher hysteresis values compared with obeche. Fraike has lower levels of sorption. When temperature increases, difference of the sorption hysteresis between our woods decreases. Also, we see that sorption hysteresis of studied woods are ranged from 0,5 to 3%.



(a) (b)
Figure 4. Desorption isotherms of obeche. (a) at 15 and 20°C; (b) at 40°C.

At a given air characteristic (temperature and relative humidity), studied woods have a near EMC.

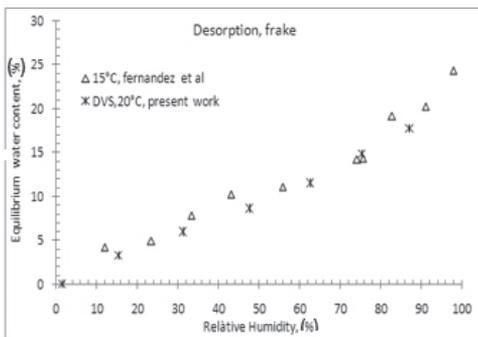


Figure 5. Desorption isotherms of frake at 15 and 20°C.

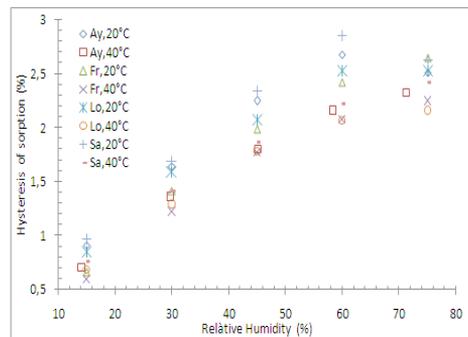


Figure 6. Hysteresis of sorption of tropical woods Sa: Sapele; Lo: Lotofa; Fr: Frake; Ay: Obeche

Table 2 shows that intensities of correlation are greater than 0,9. Table 3 presents K_d , K_h and W_i . We observe that both adsorption and desorption phases at 20°C and 40°C, obeche has a low equilibrium between the activities of adsorbed water and water vapor (K_d) and the one of frake is the great. The one of sapele is greater than the one of lotofa. Both adsorption and desorption at 20°C and 40°C, lotofa has less accessibility for monolayer water (K_h is weaker). Sapele has most accessibility for monolayer water (K_h is higher). Both adsorption and desorption, at 20°C, frake has less accessibility for monolayer water compared of obeche, but at 40°C, inverse is presented. Both adsorption and desorption phases at 20°C and 40°C, frake has a great apparent molecular weight of wood per sorption site (W_i) and obeche has the lower. At 20°C, obeche has the great W_i than lotofa. K_d , K_h and W_i increase with the temperature in desorption, but decrease with temperature in adsorption. Table 4 confirms that isotherms plotted are the type II sigmoid (because $C > 2$ according to Esteban *et al.* (2004) and Fernandez *et al.* (2014)) and intensities of correlation are greater than 0,9. We saw that monolayer saturation water content is greater in the case of desorption than the case of adsorption. As well as adsorption than desorption, the monolayer saturation moisture content X_m is lower in the case of frake and higher in the case of obeche. X_m of lotofa is greater than the one of sapele.

Table 2. Identified parameters for HH sorption curves and intensity of correlation (R^2).

Wood species	HH model							
	Adsorption				Desorption			
	A	B	C_o	R^2	A	B	C_o	R^2
20°C								
Lotofa	5,302	8,405	9,878	0,998	4,094	4,259	3,621	0,933
Frake	4,601	11,650	12,830	0,990	4,068	5,354	5,100	0,946
Sapele	3,613	12,000	12,080	0,995	3,140	6,068	4,750	0,972
Obeche	3,929	10,420	10,740	0,999	3,352	5,115	3,912	0,957
40°C								
Lotofa	4,447	9,000	9,484	0,981	3,764	5,158	4,307	0,997
Frake	4,028	12,420	12,600	0,935	3,679	7,152	6,489	0,980
Sapele	3,646	11,190	10,940	0,939	3,188	6,805	5,641	0,992
Obeche	4,034	9,280	9,319	0,981	3,554	5,028	4,006	0,995

Table 3. Estimation K_d , K_h and W_i from identified parameters for HH sorption curves.

Wood species	Adsorption						Desorption					
	20°C			40°C			20°C			40°C		
	K_d	K_h	W_i (kg/kmol)	K_d	K_h	W_i (kg/kmol)	K_d	K_h	W_i (kg/kmol)	K_d	K_h	W_i (kg/kmol)
Lotofa	0,786	3,018	30127,11	0,765	3,646	28443,50	0,555	2,876	15839,65	0,585	3,342	17213,42
Frake	0,830	4,053	34709,95	0,805	4,832	34022,74	0,641	3,054	19019,84	0,674	3,885	21797,34
Sapele	0,810	5,104	32127,87	0,780	4,937	30374,97	0,598	4,232	17680,42	0,638	4,345	19573,05
Obeche	0,793	4,343	29977,77	0,756	4,044	27680,81	0,560	3,727	15959,75	0,568	3,489	16322,55

K_d and K_h don't have unity

Table 4. Identified parameters for GAB sorption curves and intensity of correlation (R^2)

Wood species	GAB model							
	Adsorption				Desorption			
	X_m (%)	C	K	R^2	X_m (%)	C	K	R^2
20°C								
Lotofa	6,02123048	3,97560688	0,780342	0,99534	9,85158182	4,13581965	0,636667	0,9881
Frake	5,18791904	5,05352862	0,827875	0,98963	8,41998247	4,33013916	0,700698	0,98785
Sapele	5,57780368	6,13328062	0,813057	0,99524	9,06574514	5,55060591	0,663477	0,99727
Obeche	6,01201598	5,3317543	0,79179	0,99958	9,45324903	5,1000101	0,658499	0,99137
40°C								
Lotofa	6,3223862	4,6569258	0,7649	0,98313	10,15549	4,529503	0,5865	0,9991
Frake	5,2935741	5,8046786	0,8058	0,93656	8,903485	4,669546	0,6345	0,99578
Sapele	5,959128	5,8808621	0,7755	0,94012	9,562488	5,272886	0,6136	0,99544
Obeche	6,5164785	5,0407089	0,7560	0,98041	10,6308	4,5790	0,59	0,99703

Figure 7 presents evolutions of measured and modeled EMC (during desorption and adsorption), also the monolayer sorption (X_n) and polylayer water sorption (X_d) applied on sapele (at 40°C) and on lotofa (at 20°C). The HH and GAB models seem to explain plausibly the sorption of water in wood. The monolayers are always greater in desorption than in adsorption and it is most affected by the sorption hysteresis. For this reason, X_m obtained between adsorption and desorption are more different. The polylayers in desorption and in adsorption are near when RH is inferior of 0,7. The monolayers sorption are reached at equilibrium moisture fractions in the range 0,04-0,06 such as announced in the literature (Björk and Rasmuson 1995).

Figure 8 shows that the point of inflexion of the isotherm corresponds to a moisture content of 0,04 kg/kg and occurs at 0,3 RH in adsorption at 20°C (Figure 8a). This point of inflexion corresponds to a

moisture content of 0,08 kg/kg and occurs at 0,4 RH in desorption at 20°C (Figure 8b). Beyond each of these points, multilayer sorption predominates over monolayer sorption. These findings indicate that, from 0,3 to 1 RH (in adsorption) and from 0,4 to 1 RH (in desorption), the water taken up by the samples via monolayer sorption is minimal, at only is ranged from 0,6 to 3,85% of the total (given in Table 5). Effectively, example given on Figura 8, shows that when EMC is higher than X_m , X_h versus RH is not constant and vary slowly. At the given temperature, X_{up} is higher in desorption than in adsorption. Applied on the old wood of *Pinus sylvestris*, Esteban *et al.* (2006) indicate that in desorption, from 0,316 to 1 RH the water taken up by the sample via monolayer sorption is minimal, at only 0,7% of the total.

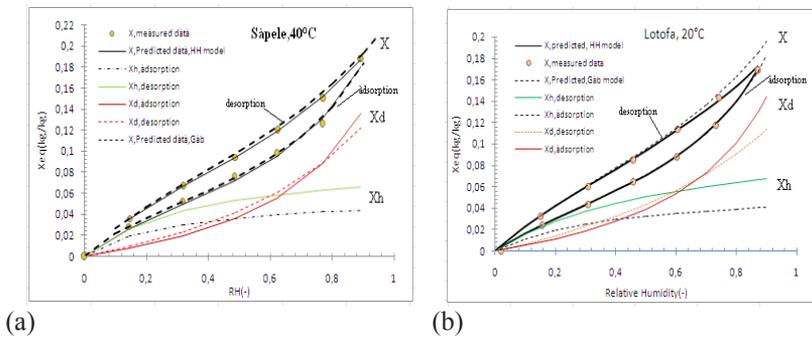


Figure 7. Sorption isotherms and deconvoluted adsorption-desorption curves fitted by the HH model, GAB model and measured data. (a) sapele at 40°C; (b) lotofa at 20°C

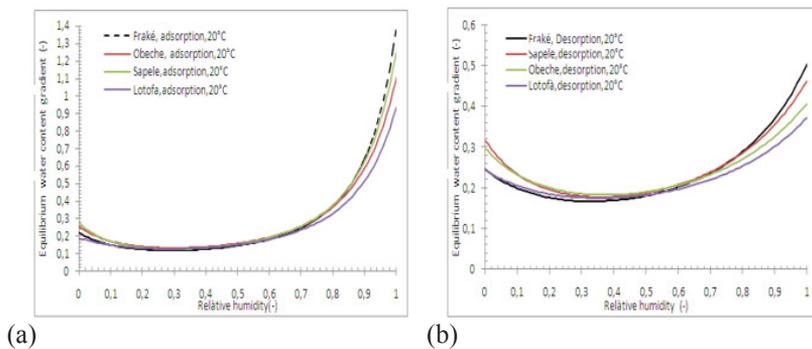


Figure 8. Equilibrium moisture content gradient versus relative humidity of our woods (a) adsorption, 20°C; (b) desorption, 20°C.

Table 5. Deduced X_{up} .

Wood species	$X_{up}, \%$			
	Adsorption		Desorption	
	T=20°C	T=40°C	T=20°C	T=40°C
Lotofa	1,22	1,52	3,85	3,36
Frake	0,69	0,79	1,12	1,71
Sapele	0,78	0,76	0,97	0,96
Obeche	0,61	1,12	1,06	2,23

CONCLUSIONS

In this work sorption behavior of tropical woods applied on frake, obeche, lotofa and sapele is studied and analyzed using DVS-Intrinsic apparatus. The duration of each adsorption-desorption cycle

is almost 4500min. DVS-Intrinsic apparatus gives satisfactory results on sorption isotherms of studied tropical woods. These sorption isotherms are well fitted with GAB and HH models and the results indicate a type II sigmoid shape for the EMC-RH curves, showing a decrease of the EMC with the increase of temperature, but with a weak influence. Sorption hysteresis most affects the monolayer saturation moisture content, the proof that the cycle desorption-adsorption most affects heat of sorption in the monolayer scale and briefly physical and chemical sorption properties in the scale of the pores. Monolayer saturation moisture content given by X_m is always higher than the moisture content corresponding to RH_m that gives the minimum of the derivative of EMC in relation to the RH. By growth order of magnitude, active sorption sites available within the wood cell walls are fraque, sapele, obeche and lotofa at 20°C; fraque, sapele, lotofa and obeche at 40°C. Air temperature, air relative humidity and wood species affect the sorption behavior and sorption hysteresis of the tropical woods. The predominance of multilayer over monolayer of our woods is from 0,3 to 0,4 RH.

In a future work, it will interesting to study influences on sorption isotherms of extractives content, history of the sample, wood structure and the part of the sample in the log.

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