

Analysis Of Water Desorption In Purple Ipê (*Handroanthus Impetiginosus* Mart. Ex DC. Mattos) Husk, Endosperm and Whole Seeds

¹Leandro, C. H. S., ¹Freire, J. T., *¹Maia, G. D.

¹Chemical Engineering Department, Federal University of Sao Carlos
Via Washington Luis, Km235, P.O. Box 676, 13565-905, São Carlos, SP, Brazil
Corresponding author: Maia, G. D.

Abstract: Purple Ipê (*Handroanthus impetiginosus* Mart. ex DC. Mattos) is a tree of South America, known for reforestation use, medicinal use and as hardwood. Its seeds are very sensitive and need to be processed and stored under strict conditions to ensure their germination capacity. Analysis of their hygroscopic behaviour serves as a basis for studies on drying processes and storage conditions. The aim of this paper is to determine the desorption isotherms of Purple Ipê whole seeds, husk and endosperm at 20°C, 30°C and 40°C with subsequent analysis of the multilayer Guggenheim, Anderson and de Boer sorption model (GAB) parameters, as well as liquid isosteric heat and desorption entropy calculations. To determine the isotherms, the static gravimetric method using saturated solutions of known water activity was used. The isotherms presented good reproducibility, although the influence of temperature is slight in the range of values studied. The GAB model fitted well to the experimental data obtained for the desorption isotherms for whole seeds, but presented incoherent values for the husk and endosperm due to the experimental deviations resulting from the nature of the samples. The isosteric heat and desorption entropy calculations showed deviations from those predicted by the GAB model for monolayer moisture, but were considered adequate within the order of magnitude.

Keywords: reforestation, seeds storage conditions, sorption isotherms

Date of Submission: 25-02-2019

Date of acceptance: 11-03-2019

I. Introduction

In Brazil, the Ministry for the Environment estimates that 35 million hectares will have to be restored - replanted or induced to be recovered - so that rural producers comply with the forest code norms [1]. However, there are no seeds available to meet this demand. It is difficult to find labour to collect seeds and there are few regularized native plant nurseries in Brazil, which clearly shows there is a niche in the market. On the other hand, for rural producers, reforesting Legal Nature Reserves can be a clever way to diversify property and earns a profit from trading certified hardwoods, extracting oil, selling seeds and even providing environmental services.

In this context, the Purple Ipê (*Handroanthus impetiginosus* Mart. ex DC. Mattos) is one of the species that is known for having a high economic value, considering the purposes of its wood and leaf extracts, and alarming decrease in the number of individuals still found in areas of natural occurrence. It originates from the Brazilian Atlantic Forest, found both in the Atlantic rain forest and in the semideciduous rain forest. Sometimes it also occurs in the Brazilian Cerrado. It also occurs in Argentina, Bolivia, Colombia, French Guiana, Paraguay, Peru, Suriname and Venezuela, in South America; in El Salvador, Costa Rica, Guatemala, Honduras, Nicaragua and Panama, in Central America, and in Mexico in North America.

Employed as a reforesting tree, usually live in riparian forests in the Brazilian Cerrado, and in areas near to rivers. The tree is widely used in urban afforestation in Brazil. The wood presents good durability and resistance against organisms that feed on it (xylophages), being difficult to saw or nail. Used in construction civil, musical instruments and bowling balls. From the bark, are extracted tannic and lapachic acids, alkaline salts and dye which is used to dye cotton and silk. Extracts of bark, leaves and flowers are used in medicine due to its antioxidant, antibiotic, bactericidal, antiviral, antifungal and cicatrization activities.

Its seed viability ranges from 3 to 15 months in cold/dry chambers and drastically reduces in cold chambers and in a laboratory environment. Purple Ipê seeds are winged, orthodox and have a germination rate of 60%. There are 13,500 to 35,000 seeds per kilogram [2]. Studying the storage of seeds, the authors identified a small influence of the dry-chamber condition on the conservation of Yellow Ipê seeds [3]. According to the authors, the critical moisture content can be around 10% to 11%. It was found that the effective conditions for storage comprise a combination of relative humidity of 10 to 50% and a temperature of 0°C to 10°C [4]. For

short periods, temperatures between 0 and 5°C were recommended, while for long periods the recommended temperatures are between -4°C and -18°C [3].

Thus, the aim of this paper is to analyse the desorption of water in Purple Ipê (*Handroanthus impetiginosus* Mart. ex DC. Mattos) husk, endosperm and whole seeds. The desorption isotherms were determined at 20°C, 30°C and 40°C and analyzed using the Guggenheim, Anderson and de Boer (GAB) model evaluating the physical meaning of their parameters. For this purpose, Purple Ipê whole seeds, its husk and endosperm were used. Its husk is extremely important in terms of distributing it in nature and cannot be separated from the seed for storage and commercialization purposes, which requires studies concerning its influence on the process of hygroscopic balance of the seeds. Furthermore, this paper analysed the liquid isosteric heat and desorption entropy and compared it with the analyses obtained from evaluating the GAB model parameters.

II. Material And Methods

II.1. The GAB model:

The GAB model represents a refined extension of the Langmuir theories [5] and the extension proposed by Brunauer, Emmett and Teller in the BET model [6]. The theoretical basis for the GAB model is the assumption of localised physical sorption in multilayers, where the first layer of water evenly covers the surface of the adsorbate and is tightly bound to it, forming what is called a monolayer. The molecules arranged in the multilayer interact with the adsorbate at levels varying from monolayer to liquid water, or bulk water [7]. Thus, successive layers of water have more and more properties similar to those of liquid water [8]. Three considerations are used to develop the GAB model: that the adsorbate presents independent and distinguishable sites of the same activity, isothermal and open to the adsorbent vapor [9]. Statistical thermodynamics leads to the expression of the GAB model, represented by:

$$\frac{X}{X_m} = \frac{C_g \cdot K \cdot a_w}{(1 - K \cdot a_w) \cdot (1 - K \cdot a_w + C_g \cdot K \cdot a_w)} \quad (1)$$

In this equation, the three parameters of the GAB model, C_g , K and X_m have a physical meaning. C_g is defined as the ratio between the partition function of the first molecule sorbed at one site and the partition function of molecules adsorbed beyond the first molecule in the multilayer. It is a measure of the binding force of water to the primary binding sites and is intrinsically enthalpic in nature. Thus, the higher the C_g value, the stronger the water is bound in the monolayer and, consequently, the greater the difference in enthalpy between the monolayer and multilayer molecules [7]. Considering that monolayer molecules have low mobility and, therefore, a limited number of possible configurations, the entropic effect associated with C_g is lower than the enthalpic effect [10].

K is defined as the ratio between the partition function of the liquid water molecules and the partition function of the adsorbed molecules in the multilayer. The entropic content associated with K can be explained considering that liquid water molecules have more possibilities of configuration and mobility when compared to those in the multilayer [10].

When K approaches the unit value, no distinction is made between the liquid water and the water present in the multilayer, and the GAB model is reduced to the BET model, which considers the characteristics of the water in the multilayer equal to those of the liquid water. X_m is known as the monolayer value and is a measure of the availability of active sites for water sorption by the material [10]. The physical meaning of the X_m parameter can be described as the number of molecules in the monolayer or simply the moisture content of the monolayer [11]. The qualitative analysis of the GAB parameters makes it possible to evaluate the behaviour of the adsorbed moisture by combing the C_g and K values, as described in Table 1.

Table 1. Combination of the magnitude of the C_g and K parameters and the corresponding sorption/desorption mechanism.

$Mo \approx Mu \approx Liq$	$Mo \approx Mu \neq Liq$	$Mo \neq Mu \neq Liq$	$Mo \neq Mu \approx Liq$
$C_g \approx 1$	$C_g \approx 1$	$C_g \gg 1$	$C_g \gg 1$
$K \approx 1$	$K \ll 1$	$K \ll 1$	$K \approx 1$

II.2. Liquid isosteric heat and desorption entropy:

An important thermodynamic parameter obtained through isotherms at various temperatures is isosteric heat, which measures the energy of the bonding forces between the water molecules and the solid [12]. Its measurement provides information on the nature of the sorption/desorption mechanism and helps in the process of determining water types in adsorbate [13].

The difference between the amount of energy required to remove water from a material and the energy required for the vaporization of liquid water at the same temperature and pressure of the material is defined as liquid isosteric heat (ΔH_{is}) and can be calculated using the Clausius-Clapeyron equation:

$$\Delta H_{is} = -\mathfrak{R} \cdot \left(\frac{\partial \ln a_w}{\partial T^{-1}} \right)_x \quad (2)$$

This relation is more adequate for qualitative analyses on the nature of ΔH_{is} as its values are calculated through a series of algebraic manipulations, leading to considerable errors, besides those already associated to the water activity measures [7]. In addition, (2) takes into account that ΔH_{is} is invariant with temperature. In this regard, it should be mentioned that the results obtained for ΔH_{is} represent an average value in the evaluated temperature range. Some authors suggest using desorption data alone, disregarding possible hysteresis effects, for the determination of ΔH_{is} [7, 10, 11]. According to the authors, desorption represents the phenomena as a whole. In addition, the authors argue that the study of isotherms mainly aims to provide practical information for drying processes, therefore, a desorption process.

Desorption entropy is the measure of the entropy difference between the adsorbed water and the liquid water for sorption processes and vice versa, in the case of desorption. It can be calculated considering the relationships described in Equations 3 and 4 [8].

$$\Delta G_{is} = \mathfrak{R}T \ln a_w \quad (3)$$

$$\Delta S_s = \frac{\Delta H_{is} - \Delta G_{is}}{T} \quad (4)$$

Considering ΔH_{is} regardless of temperature, substituting (3) in (4), we obtain the expression for the calculation of ΔS_s :

$$\ln a_w = \frac{\Delta H_{is}}{\mathfrak{R}} \cdot \frac{1}{T} - \frac{\Delta S_s}{\mathfrak{R}} \quad (5)$$

II.3. Sample preparation:

Purple Ipê seeds (*Handroanthus impetiginosus* Mart. ex DC. Mattos) were purchased from the Xingu Seed Network and the respective purity test report of the sample was obtained. To determine desorption isotherms, seeds were submitted to rehumidification by submersion in water at 5°C for 24 h, followed by a rest period of 2h in a single layer at a temperature of 25°C. Afterwards, the amount of humidified seeds was distributed in two batches in which one remained with the whole seed and the second batch had the husk separated from its endosperm, as shown in Fig 1. The humidity of the samples was determined by oven drying them at 105°C at the end of the procedure to determine the sorption isotherm.



Figure 1. Purple Ipê Seeds: whole, husk and endosperm respectively.

II.4. Methodology to determine the isotherms:

The static gravimetric method was adopted using saturated solutions under constant temperature conditions. In this method, the ambient temperature and relative humidity are kept constant until the mass of the samples remains constant between two successive weightings. Flasks were filled with a saturated solution according to the data available in Table 2. A perforated container with the sample was attached to the top of each vial so that the solution did not come in contact with the sample.

The sample mass was determined using an analytical balance with an accuracy of 0.001g. The tests were carried out in a chamber (Binder) with temperature control. The tests were performed in triplicate. The

samples were incubated for 30 days. After this period, the mass was determined and the samples were placed in the oven for another 7 days to weigh them again to determine if there was any change in the mass.

Table 2. Relative humidity for saturated solutions at different temperatures [14].

Compound	20°C	30°C	40°C
KOH	0.08	0.07	0.06
CH ₃ COOK	0.21	0.20	0.19
MgCl ₂	0.33	0.32	0.31
K ₂ CO ₃	0.43	0.43	0.43
Mg(NO ₃) ₂	0.55	0.48	0.47
NaNO ₂	0.65	0.62	0.60
NaCl	0.76	0.75	0.75
KCl	0.85	0.82	0.80

III. Results and Discussion

III.1 Desorption isotherms:

Desorption isotherms for the Purple Ipê whole seeds, husk and endosperm, separately, at temperatures of 20°C, 30°C and 40°C are shown at Fig. 2. The standard deviation was less than 5% for all experimental data. Fig. 2 shows the mean that the whole seeds and their parts present similar hygroscopic behaviour in the temperature range evaluated. It can be observed that the husk has a greater capacity of water sorption per kilogram than the respective whole seed and its endosperm in the evaluated temperature range. In water activities around 0.4, the equilibrium moisture for the husk at 20°C is approximately double that observed for the endosperm and the whole seed: the first is approximately 0.08 kg/kg and the second is 0.05 kg/kg.

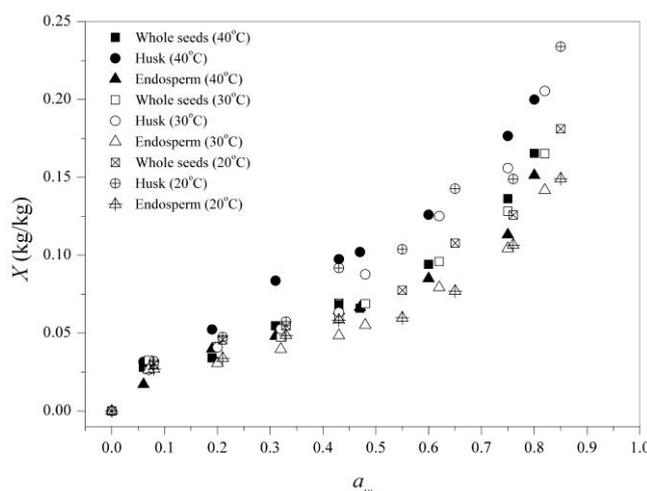


Figure 2. Desorption isotherms for Purple Ipê whole seeds, husk and endosperm.

This discrepancy is less evident in the water activity region up to 0.2 where the equilibrium moisture for the seed parts are similar to those of the whole seed and are not very affected by the temperature. This can be explained by the fact that the husk generally presents hygroscopic behaviour that enables it to protect the endosperm in cases where there is a great availability of water for the seed, i.e., the husk would be soaked, sparing the structure of the endosperm of an amount of water that makes its biological activities unviable.

Fig. 2 also shows that in the temperature range studied, the hygroscopic behaviour of whole seeds and endosperm are similar and are not very affected by the temperature. This suggests that the husk contributes less effectively when the whole seed is analysed. This fact occurs because the husk mass represents about 5% of the total mass of the seed, and its sorption and desorption capacity follow this proportion in quantitative terms. Figs. 3, 4 and 5 show the GAB model adjustments for the desorption isotherms of whole seeds, husk and endosperm at temperatures of 20°C, 30°C and 40°C, respectively.

When analysing the parameters of the GAB model for desorption isotherms of whole seeds, it can be observed that there is a conformity of the behaviour of these parameters with what is expected in relation to the increase in temperature. However, Quirijns et al. (2005a) reinforce that the desorption isotherm analysis must be predominantly qualitative, as the C_g parameter presents little sensitivity in the adjustment. For whole seeds, the proximity of the isotherms to the different temperatures suggests that Purple Ipê seeds are hygroscopically unresponsive to the variation of temperature within the studied range.

This result is not compatible with those observed in studies such as the thermodynamic analysis for the hygroscopic behavior of barley seeds [15], where the seeds showed significant differences in water desorption in

the temperature range of 15°C and 50°C. The authors justified this behaviour since the rise in humidity caused an increase in the availability of sorption sites, and consequently the X_m parameter went up significantly when the temperature was increased. Considering the physical meaning of the X_m parameter [11], we can observe its decrease when the temperature is increased.

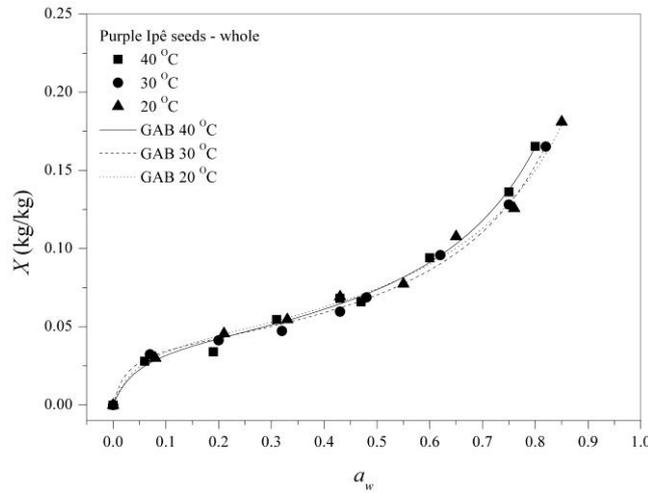


Figure 3. Adjustment of the GAB model for whole seeds desorption isotherms at 20, 30 and 40°C.

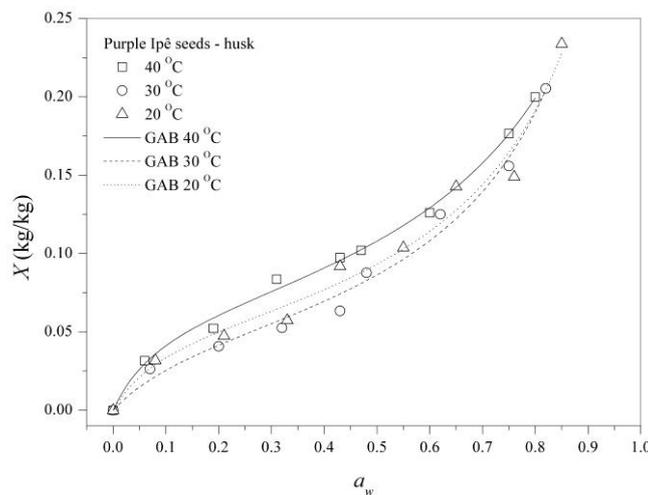


Figure 4. Adjustment of the GAB model for husk desorption isotherms at 20, 30 and 40°C

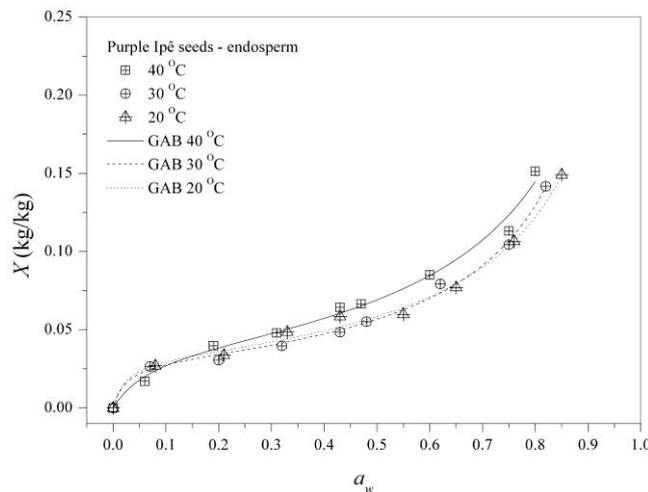


Figure 5. Adjustment of the GAB model for endosperm desorption isotherms at 20, 30 and 40°C

The literature often shows the values of X_m as constant [16]. However for better accuracy and physical consistency of the values of the other GAB model parameters, X_m should not be considered constant for different temperatures [11]. However, in Purple Ipê whole seeds, what is observed is a slight increase in the X_m values as a function of the temperature rise, starting from 0.043 kg/kg at 20°C to 0.041 kg/kg at 40°C. This variation, although expected from the theoretical point of view, is within the range of experimental error, which reinforces the use of these data from the qualitative point of view. This effect can be explained considering that, insofar as the system has a higher energy content, more molecules have properties compatible with those of the multilayer, therefore decreasing the value of X_m when the temperature increases.

Another effect that occurs when the temperature is increased is that a decrease in the value of X_m may occur due to a reduction in the number of active sites for water binding due to the structural changes caused in the material by the increase in temperature [7]. Considering that for Purple Ipê whole seeds, this variation is not significant, it can be inferred that in this temperature range the seed does not undergo significant structural changes. The C_g parameter decreases when the temperature increases, as observed in other studies [7, 11, 15]. Table 3 shows the qualitative conjugate effect of the C_g and K parameters in order to determine the water classes in the material.

Table 3. Relative Humidity for Saturated Solutions at different temperatures

Parameters	Temperatures		
	<i>Whole</i>		
	20°C	30°C	40°C
X_m	0.043	0.042	0.041
C_g	92	42	22
K	0.91	0.93	0.94
R^2	0.99	0.99	0.99
	<i>Husk</i>		
	20°C	30°C	40°C
X_m	0.043	0.038	0.041
C_g	25	41	22.5
K	0.9	0.94	0.94
R^2	0.99	0.99	0.99
	<i>Endosperm</i>		
	20°C	30°C	40°C
X_m	0.057	0.05	0.042
C_g	21.68	8.27	8.87
K	0.89	0.91	0.92
R^2	0.97	0.91	0.99

Similar to that reported for starch cylinders [7], GAB model adjustments for barley isotherms showed relatively higher C_g values than the unit, and K values smaller than the unit for the whole temperature range. According to the authors, and as shown in Table 1, this indicates that water molecules are arranged in a monolayer, with molecules tightly attached to their sorption sites and a multilayer, in which the water molecules have considerably different properties compared to liquid water at the same temperature and pressure.

The higher the C_g value, the more strongly attached the monolayer molecules are. Considering that C_g decreases with the temperature, this analysis also explains the decrease in X_m values, so that the molecules are less and less bound to their monolayer sites as the temperature increases. As the temperature increases, an increase in K can be evaluated. This result is physically coherent, considering that a rise in temperature increases the entropic effect on the system, so that the water molecules have greater possibilities of configuration and mobility. Considering that for Purple Ipê seeds the K values were higher than 0.91, it can be inferred that the multilayer region is narrow, so that above the monolayer there is a small region where the water presents a differentiated behaviour of liquid water, but which moves quickly as the multilayer develops.

When we analyse the parameters of the GAB model for the husk and endosperms, it can be observed that there is a physical incoherence in relation to the values of the coefficients of the GAB model. It can be observed in the graphs in Figs. 4 and 5 that, on average, the isotherms determined at 40°C show higher equilibrium moisture values on a dry basis than those observed at 30°C and 20°C in the same water activity.

This contradicts studies another studies [7, 11, 15]. In all cases, the increase in temperature led to a decrease in the hygroscopic capacity of the analysed samples in the same water activity. By analysing the data obtained for the husk and the endosperm it can be observed that the isotherms are significantly close, in the temperature range studied. Considering the inherent deviations from the methodology, the effects of temperature on the equilibrium moisture of the seed structures could not be identified separately.

Thus, a temperature rise should not produce an increase in the values of C_g and K , as observed in Table 3 for husk and endosperm, respectively. Although the X_m values are consistent, experimental tests carried out on the husk may present additional deviations due to the reduced mass of the sample used in the experiments,

resulting from the low density of the husk. Concerning the endosperm, a degradation of the sample was also observed during the experiment, which may account for the errors associated with this measure in particular. However, these effects, were not observed in the tests carried out with the whole seed, suggesting that the husk, although not having a significant hygroscopic effect on the equilibrium moisture of the seed as a whole, provides protection to the degradation of the endosperm.

The qualitative analysis of the sorption is still adequate due to the reproducibility of the data and the satisfactory adjustments of the parameters of the GAB model [7, 11]. Although the analyses of the GAB parameters are not quantitatively possible for the husk and the endosperm, it was observed that the behaviour of the desorption curves follows the same trend as the whole seeds, which suggests that the husk does not have a significant influence on the hygroscopic capacity of the seeds and that a monolayer is formed with the binding force of water, followed by a multilayer with water having similar characteristics to those of free water. The analyses also demonstrate that all seeds parts have the same energetic relation with water, so that the seeds drying process tends to occur homogeneously in all tissues.

III.2 Isostatic heat and desorption entropy:

Considering the experimental data, the GAB model was used to calculate values of water activity as a function of temperature for specific values of equilibrium moisture. Using (5), the ΔH_{is} and ΔS_s values were calculated, shown in the graphs of Figs. 6 and 7. As predicted, the ΔH_{is} values were positive, in agreement with the endothermic character of the desorption. It was observed that ΔH_{is} significantly decreased from an equilibrium moisture of 0.02 kg/kg with increasing moisture content starting from values of approximately 3000 kJ/kg.

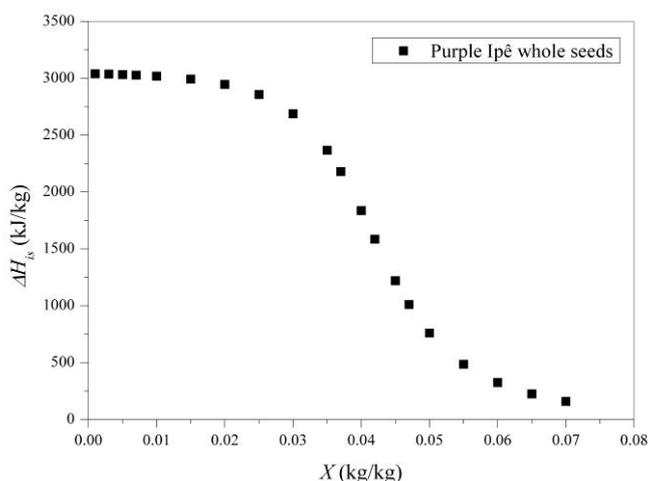


Figure 6. Isostatic heat for Purple Ipê whole seeds.

This is indicative of intermolecular attraction forces between the sorptive sites and water vapour molecules [7]. According to the authors, at moisture contents in the monolayer region, water is tightly bound to the material, corresponding to the high interaction energy. Under these conditions, the total heat of sorption is equal to the heat of vaporization and the water molecules in the adsorbate behave in a similar way energetically to those of liquid water.

The same can be observed for the ΔS_s values shown in Fig. 7. Coherence was observed for positive values for ΔS_s . This behaviour was expected since, as the moisture increases in the monolayer, the water entropy decreases, considering that the sites are filled and the degrees of freedom of the water molecules decrease until the whole monolayer is filled. The graph in Fig. 7 shows the point of maximum entropy variation between liquid water and adsorbed water, of approximately 8.0 kJ/kg.K for a moisture content of 0.02 kg/kg.

This point coincides with the point of water content for the monolayer formation. This is due to the fact that the maximum desorption entropy occurs when water molecules form the complete monolayer, thus decreasing the number of degrees of freedom that water molecules have. From this point, any increase or decrease in moisture in the humidity of the material increases the degrees of freedom, causing the entropy variation to decrease. This result is more evident in graphical representations of desorption entropy than in isosteric heat.

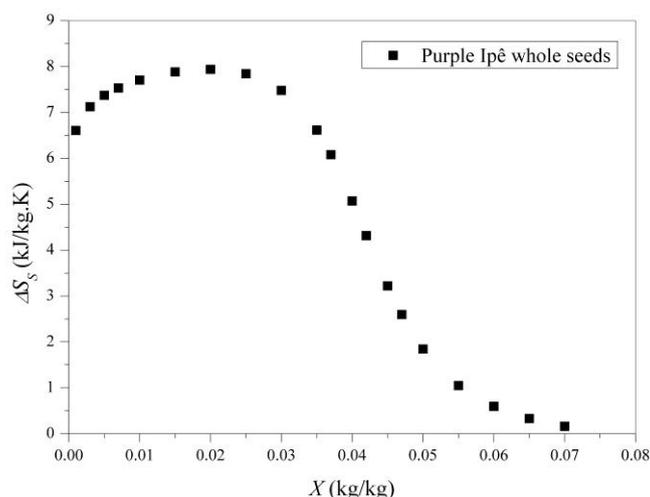


Figure 7. Desorption entropy for Purple Ipê whole seeds

In addition, the monolayer moisture values obtained from the GAB model adjustments can be compared with those observed in the graph of Fig. 7. While for whole seeds, the mean value of X_m was 0.042 for the studied temperature range, the graph in Fig. 7 indicates a value for the monolayer moisture of 0.02 kg/kg, half of that found by the GAB model. However, considering the same order of magnitude and the fact that the isosteric heat and desorption entropy data show deviations associated with algebraic manipulation for their determination, the values can be considered coherent. It should be noted that no statistical analysis was performed in the determination of the parameters of the GAB model, as well as isosteric heat and desorption entropy, since the determination of these parameters allows a qualitative and non-quantitative analysis of the phenomenon [7]. Therefore, their order of magnitude and tendency to increase or decrease were evaluated.

IV. Conclusion

Considering the above, for Purple Ipê whole seeds and their parts, it can be concluded that there is little influence of the husk on the equilibrium moisture for whole seeds, although the husk has a higher sorption capacity in the studied temperature range.

The endosperm presented similar behaviour to that of the whole seed. For whole seeds, the temperature had a slight influence on the equilibrium moisture values.

The GAB model parameters were coherent in terms of values and behaviour for the whole seed isotherms, although the qualitative approach is more significant than the quantitative one in this type of analysis.

It was not possible to conclusively analyse the parameters of the GAB model for the husk and the endosperm separately.

Concerning the husk, it was believed that the deviations were caused by the reduced sample mass and by the subtle behaviour of the isotherms in relation to the temperature variation.

Regarding the endosperm, the deviations were believed to be caused by the degradation of the sample during the experiment. The isosteric heat values were calculated satisfactorily for the experimental data, with a maximum value of about 3000 kJ/kg.

The maximum desorption entropy was observed in the formation of the monolayer at a humidity of 0.02 kg/kg, the same order of magnitude of the value 0.042 kg/kg obtained by the GAB model.

The GAB model fitted satisfactorily to all the experimental curves obtained in this study.

All seeds parts have nearly the same energetic relation with water, so that the seeds drying process tends to occur homogeneously in all tissues.

Acknowledgements

The authors would like to thank the Xingu Seed Network, which provided the samples that made this study possible. This research received no specific grant from any funding agency, commercial or not-for-profit sectors.

V. Nomenclature

X	- equilibrium moisture dry basis [kg/kg]
X_m	- monolayer moisture [kg/kg]
C_g	- enthalpic parameter of GAB model
K	- entropic parameter of GAB model

a_w	- water activity
Mo	- monolayer
Mu	- multilayer
Liq	- liquid water
ΔH_{is}	- liquid isotheric heat [kJ/kg of water]
ΔS_s	- desorption entropy [kJ/kg of water.K]
ΔG_{is}	- Gibbs Free Energy [kJ/kg of water]
\mathfrak{R}	- gas constant [kJ/kg of water.K]
T	- temperature [K]

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Maia, G. D. "Analysis Of Water Desorption In Purple Ipê (*Handroanthus Impetiginosus* Mart. Ex DC. Mattos) Husk, Endosperm and Whole Seeds." *IOSR Journal of Agriculture and Veterinary Science (IOSR-JAVS)* 12.3 (2019): PP- 32-40.