

# INTERPRETING DYNAMIC VAPOR SORPTION (DVS) MEASUREMENTS: WHY WOOD SCIENCE NEEDS TO HIT THE RESET BUTTON

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**ABSTRACT:** Water vapor sorption is an important characteristic of wood as an engineering material. In addition to affecting engineering properties such as the heat capacity, thermal conductivity, strength, stiffness, and dimensions, moisture is fundamental to many wood degradation mechanisms including fungal decay. Water vapor sorption isotherms have been gaining increasing attention in the wood literature as more laboratories adopt automated sorption balances, frequently called dynamic vapor sorption (DVS) analyzers. With DVS, the collection of sorption isotherms can be automated, completely transforming what was once a series of tedious manual measurements. The adoption of DVS, however, preceded careful studies of the precision and accuracy of the measurement; as a result, many of the previously published studies have been cast under scrutiny in recent years as the technique has been further studied. This paper summarizes the advancements in understanding of the relationship between experimental method and experimental errors with DVS along with best practices that should be used when collecting DVS data in future studies.

**KEYWORDS:** wood-moisture relations, water vapor sorption isotherms, dynamic vapor sorption analyzers, parallel exponential kinetics (PEK model)

## 1 INTRODUCTION

Water affects nearly every wood property. Likewise, nearly all wood damage mechanisms involve water. Wood decay and fastener corrosion can occur when there is an excess of moisture in the wood [1, 2]. Repeated cycling of the wood moisture content can cause splits and checks to form on the wood surface. Because of their great importance to proper wood utilization, wood-moisture relations have been studied since the dawn of Western Civilizations and these advances in understanding have enhanced our use of wood [3]. While greatly studied, wood-moisture interactions are complex and still are not fully understood.

Water vapor sorption isotherms, often referred to as just “sorption isotherms” are the most fundamental measurement of wood-moisture relations as they describe the relationship between the amount of moisture in the wood for a given relative humidity in the environment at a given temperature. Sorption isotherms are path dependent, and the equilibrium moisture content (EMC) depends on not just the temperature and relative humidity (RH), but the previous RH history of the material. Typically, two isotherms are presented, an “absorption isotherm” where the humidity is increased from the oven-dry state and a “desorption isotherm” where the sample is dried from a moisture saturated

state. When isotherms are collected along a different RH path history, they are called scanning curves. Note that while true desorption isotherms can only be collected from a fully water saturated state (such as vacuum saturated), often in literature desorption isotherms are presented from a starting relative humidity of 95%; however, these should more appropriately be referred to as scanning desorption isotherms [4].

Sorption isotherms have been studied since at least 1451 [5]. For the first several centuries of measurements, the measurements were made by equilibrating a sample in an environment at a constant relative humidity and measuring the mass. Because the mass changes slowly in response to relative humidity it could take up to several months to equilibrate a sample at a given relative humidity step [6]. Therefore, the collection of sorption isotherms, was a long and tedious process, and as a result, these difficulties meant that there were very few sources of high-quality sorption data [7].

Since the beginning of the 21<sup>st</sup> century, automated sorption balances, most commonly called dynamic vapor sorption (DVS) analyzers, have become commonplace in wood laboratories around the world. DVS is an instrument designed to collect water vapor sorption isotherms. In DVS experiments, a small sample (on the order of 20 mg) is placed on a microbalance in the presence of a continuous stream of air with RH maintained by mass flow controllers mixing dry and saturated streams of air. DVS allows for very tight control of temperature and relative humidity and an extremely precise measurement of mass. Furthermore, since the technique is automated, very little labor is required to characterize a sorption isotherm. DVS also

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records the mass over time, which provides data on the rate (or kinetics) of sorption. The adoption of DVS has resulted in a wealth of literature on the sorption kinetics and equilibrium sorption properties of wood in the past decade (e.g., [8-14] among others).

However, we have recently re-examined sorption data published with DVS and conducted detailed studies that have called into question some earlier reported findings. In reviewing the experimental methods used to collect previous data, three major concerns have been found. (1) The data were not collected until equilibrium was reached, resulting in potential systematic errors in the reported EMC [15, 16]. (2) Kinetic models of water vapor sorption do not properly fit experimental data and furthermore do not fully capture the physics of water vapor sorption [17, 18]. (3) The most common equilibrium models that are used to fit sorption isotherm data are not physically meaningful [19].

## 2 STOP CRITERIA

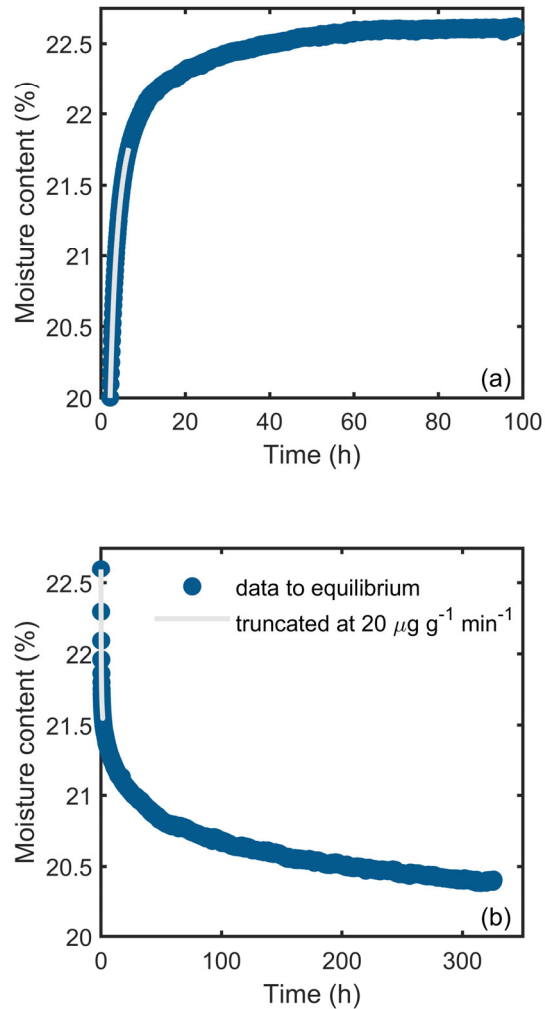
Since DVS involves the continuous collection of data with a balance that has a high mass resolution, the traditional definition of “equilibrium” as identical successive readings cannot work. Instead, a “stop criterion” is used. Stop criteria are based upon the idea that successive mass readings are close enough to ensure that kinetic equilibrium has effectively been reached. Typically, stop criteria are specified so that the mass change is less than or equal to a certain slope over a specified time period [8]. However, it is also possible to specify stop criteria in terms of a time limit (e.g., 60 minutes at each RH step [15, 20]).

In the wood literature, a stop criterion of  $20 \mu\text{g}_{\text{water}} \text{g}_{\text{dry}}^{-1} \text{min}^{-1}$  (or 0.002% MC per minute) over a 10 min window was nearly universally used (see for example [8, 11, 13, 21-23]). Data were collected until the rate of mass change was less than 0.002% MC per minute for 10 minutes, and the final value was assumed to be the equilibrium moisture content. These papers stated the MCs determined in this manner were within  $\pm 0.1\%$  MC of the true equilibrium MC, although no data were given to support this claim or define how true equilibrium MC was determined [8].

Recently, Glass et al. [15, 16] have examined how closely different stop criteria approximate true equilibrium sorption behavior. To do this, they developed an operational definition for equilibrium in a DVS experiment. They determined equilibrium by first measuring the drift limit of their automated sorption balance with an inert specimen; this limit sets the resolution of mass changes that can be observed in an experiment. They observed that the drift limit of their instrument was  $2 \mu\text{g}$  per day. When the change of mass in a sorption experiment was equal to this drift limit ( $2 \mu\text{g day}^{-1}$ ) the experiments were declared to be at equilibrium because it was impossible to separate out potential changes in specimen mass from instrument drift. Once full data sets to equilibrium were collected, it

was then possible to see how closely different stop criteria came to approximating true equilibrium moisture content.

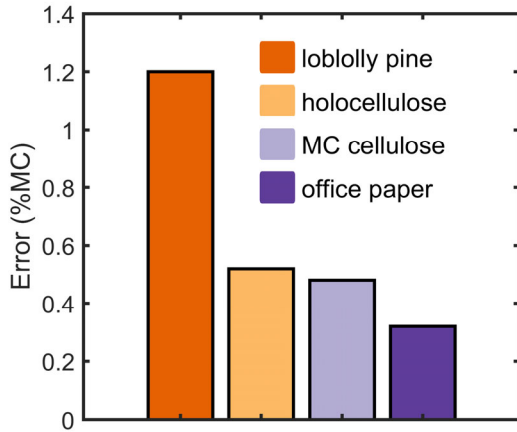
Figure 1 presents examples of this analysis for loblolly pine in absorption and desorption. In both data sets, it took more than 90 h to reach operational equilibrium. In contrast, the data truncated at  $20 \mu\text{g}_{\text{water}} \text{g}_{\text{dry}}^{-1} \text{min}^{-1}$  was finished within 7 h for absorption and 1.7 h for desorption. For the examples shown, the truncated data differed from the true equilibrium moisture content by at least 0.8% MC.



**Figure 1:** Filled circles: loblolly pine sorption data collected to operational equilibrium (where the change in mass was less than the uncertainty in the mass measurement). The top figure represents an absorption step from 90-95% RH and the bottom figure represents a scanning desorption step from 95-90% RH. The white line represents the data truncated at the commonly used  $20 \mu\text{g}_{\text{water}} \text{g}_{\text{dry}}^{-1} \text{min}^{-1}$  criterion. Data replotted from [16].

Glass et al. [16] conducted this analysis for DVS data collected for four cellulosic materials: loblolly pine (*Pinus taeda*), holocellulose, microcrystalline cellulose, and office paper for a total of 21 different RH steps. They found that on average, the  $20 \mu\text{g}_{\text{water}} \text{g}_{\text{dry}}^{-1} \text{min}^{-1}$  criterion resulted in an error in the predicted equilibrium moisture content by 0.5% MC. It should be noted the errors from using a short hold time are systematic; it underpredicts the moisture content in absorption and overpredicts the moisture content in desorption. The maximum error for each material is shown in Figure 2. It can be seen that the error in moisture content is sometimes greater than 1% MC, much greater than the reported accuracy of 0.1% MC.

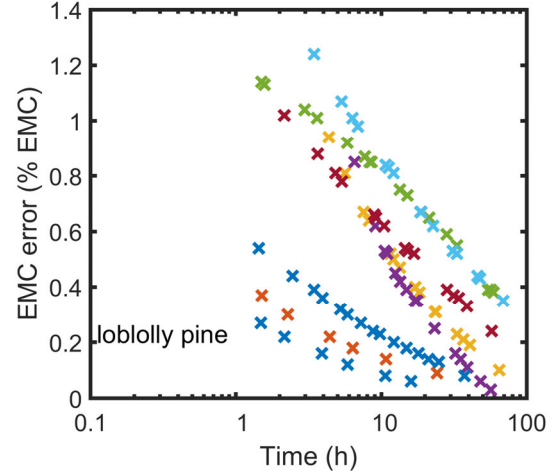
New stop criteria are needed since the errors from using the  $20 \mu\text{g}_{\text{water}} \text{g}_{\text{dry}}^{-1} \text{min}^{-1}$  criterion are unacceptably high. At the same time, it is not practical to hold each RH step to equilibrium using a DVS since this would take many months to collect a single replicate of one isotherm. Glass et al. [16] examined many different stop criteria and found that the reduction in error was roughly inversely proportional to the logarithm of measurement time (Figure 3). As such, it is extremely costly to reduce the measurement errors by decreasing the slope, or  $dM/dt$ , where  $M$  is moisture content and  $t$  is time. They recommended a maximum  $dM/dt$  criterion of  $3 \mu\text{g}_{\text{water}} \text{g}_{\text{dry}}^{-1} \text{min}^{-1}$  over 120 min. This method gives an average error of 0.3% MC.



**Figure 2:** Maximum error in the moisture content prediction for various materials found using the  $20 \mu\text{g}_{\text{water}} \text{g}_{\text{dry}}^{-1} \text{min}^{-1}$  criterion. Further details on the conditioning methods can be found elsewhere [16].

An alternative to a strict  $dM/dt$  criterion was also presented by Glass et al. [16]. This method takes advantage of the systematic over-/underprediction in the EMC caused by truncating the data before equilibrium. They found that the absolute error in EMC was linearly proportional to the moisture content recorded when the stop criterion was met. The constants of proportionality were different for absorption and desorption and also depended on the size of the RH step. Using these linear regressions, it was possible to reduce the measurement time compared to the time needed to reach  $3 \mu\text{g}_{\text{water}} \text{g}_{\text{dry}}^{-1}$

$\text{min}^{-1}$  over 120 min. They presented data for a  $dM/dt$  criterion of  $5 \mu\text{g}_{\text{water}} \text{g}_{\text{dry}}^{-1} \text{min}^{-1}$  over 60 min and found an average error of 0.1% MC. This method allows data to be collected more quickly and more accurately than the traditional methods, in which data is collected until a  $dM/dt$  criterion is reached where the final moisture content is taken as the EMC.



**Figure 3:** Relationship between the error in EMC and the measurement time for loblolly pine. Different colors represent different RH steps.

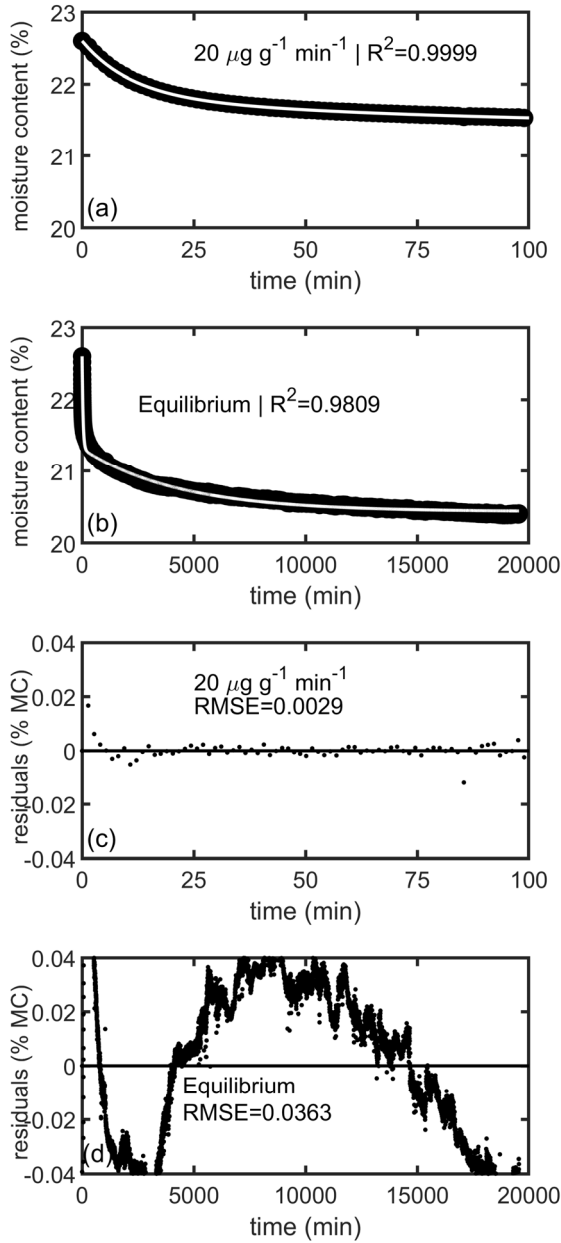
### 3 KINETIC MODELING

Sorption data collected with DVS are commonly fit with the Parallel Exponential Kinetics (PEK) model, which is mathematically the sum of two independent exponential functions, each having a moisture component and a characteristic time constant. The PEK model parameters have often been given physical significance in the literature. For example, some researchers have attributed the two time constants to two different sorption sites within the wood cell wall [23-25]. Others have attributed these time constants to a viscoelastic response in the wood cell wall [26]. Still others have used these time constants to infer activation energies of the wood sorption process [27]. In short, there have been many applications of the PEK model to sorption data in cellulosic materials and many papers have ascribed physical significance to the model fit parameters.

Recently, Thybring et al [17] demonstrated that the PEK model cannot be used to understand the kinetics of water vapor sorption in wood and presented two arguments why it should not be used.

Most importantly, the PEK model does not capture the actual form of the sorption curve. Previous papers, such as [23-27], stopped data collection at  $20 \mu\text{g}_{\text{water}} \text{g}_{\text{dry}}^{-1} \text{min}^{-1}$  and found a good agreement between the model fit and the data with  $R^2$  greater than 0.99. While the PEK model has been shown to fit literature data well, this is because the literature data were not collected to equilibrium. When fit to data collected for various times until equilibrium, the PEK model parameters change drastically and the fitting statistics get worse (Figure 4).

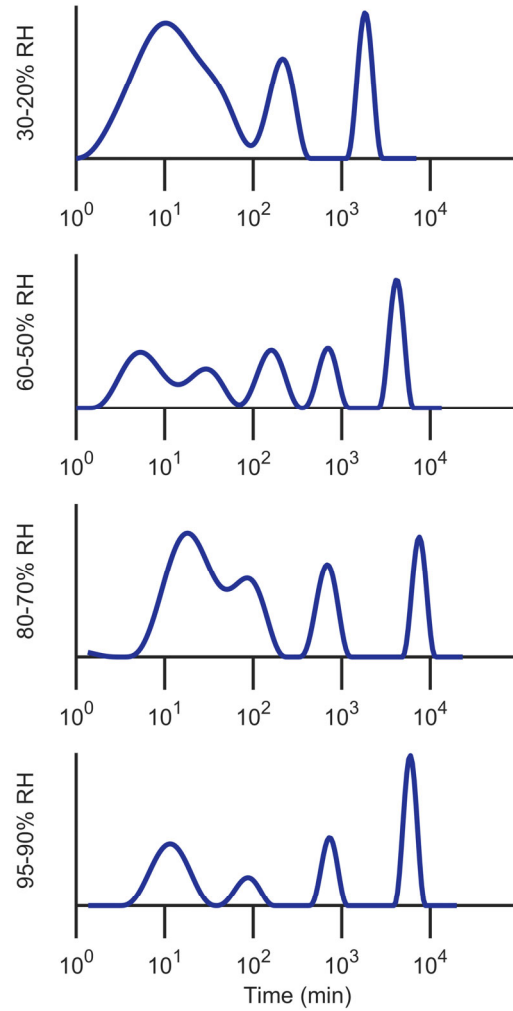
In addition, the residuals are not random but instead show trends (Figure 4d). This suggests that the model is not properly capturing the physics of the sorption process.



**Figure 4:** Figure 4(a),(b): kinetic sorption data with the PEK fit overlaid. Figure 4a presents data truncated with the 20  $\mu\text{g}_{\text{water}} \text{g}_{\text{dry}^{-1}} \text{min}^{-1}$  criterion whereas Figure 4b presents the entire data set to operational equilibrium. In Figure 4(c),(d) the residuals are plotted. Note that the PEK model gives a poor fit to data collected to equilibrium. Data replotted from [17].

Sorption kinetics and the PEK model were further explored by using multi-exponential decay analysis (MEDEA). In this technique, the data are fit to hundreds of exponential functions with logarithmically distributed time constants whose amplitude is allowed to vary to minimize the residuals. The result, shown in Figure 5, is

a smooth function with peaks that describe the important exponential time constants in the data.



**Figure 5:** MEDEA fits to loblolly pine sorption data for different RH steps. Peaks in these spectra indicate time constants. Note that there are more than two time constants for all RH steps and that the number of time constants changes with RH step. Data replotted from [17].

From the data in Figure 5, it is clear that loblolly pine exhibits more than two time constants. The number of time constants varies from three time constants for 30%-20% RH in desorption to as many as five time constants for 60%-50% RH.

The data in Figure 5 clearly demonstrate why the data collected to equilibrium cannot be fit by the PEK model. It appears that when the data are truncated at short hold times, only the first two (of many) time constants are captured in the data. However, as more data are collected, more time constants can be resolved at longer times. Because the PEK model does not capture the

physics of the sorption process, the model fit parameters cannot be physically meaningful.

#### 4 EQUILIBRIUM SORPTION MODELS

It has been known for many years that commonly used water vapor sorption isotherm models do not correctly predict thermodynamic quantities [19, 28]. Therefore, while useful for interpolating data, they cannot yield insight into the physical mechanism of moisture sorption in wood [28]. Despite this fact, many papers are still published where the model fit parameters are ascribed a physical meaning.

In fact, the most commonly used isotherm models used to describe wood are mathematically equivalent [19, 29]. Table 1 presents the mathematical expressions for the Guggenheim Anderson DeBoer (GAB) isotherm [30-32], Dent isotherm [33], and Hailwood-Horrobin isotherm [34]. These models can all be arranged into a parabola of the form

$$\frac{a_w}{M} = C + Ba_w + Aa_w^2 \quad (1)$$

where  $a_w$  is the activity of water (fractional relative humidity),  $M$  is the fractional moisture content, and  $A$ ,  $B$ , and  $C$  are fitting parameters.

**Table 1:** Mathematical expressions for the GAB, Hailwood-Horrobin, and Dent isotherms.

Expression for $M/M_m$	Reference
$\frac{c_0 k a_w}{(1 - k a_w)(1 + (c_0 - 1)k a_w)}$	GAB [30-32]
$\frac{(1 + K_1 K_2)(1 + K_2)a_w}{K_2(1 - K_1 a_w)(1 + K_1 K_2 a_w)}$	HH [34]
$\frac{b_0 a_w}{(1 - b_1 a_w)(1 + b_0 a_w - b_1 a_w)}$	Dent [33]

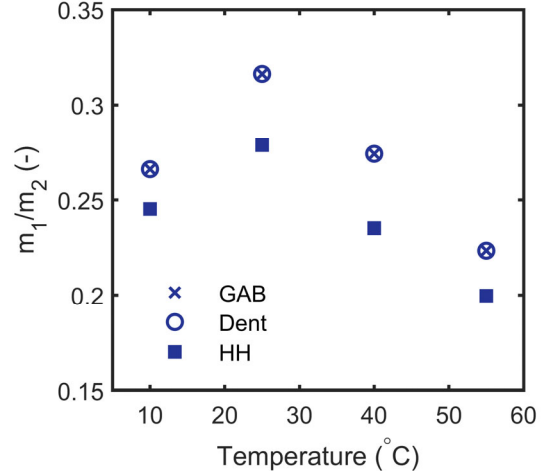
Each of these models has a parameter for a monolayer moisture capacity ( $M_m$ ); that is, water molecules interacting directly with specific locations in the solid material, e.g. adsorbed onto specific sites on the surface of a pore or adsorbed on specific functional groups on a polymeric material. Table 2 shows how the monolayer capacity can be calculated from the fitting parameters  $A$ ,  $B$ , and  $C$ . Note that the parameter  $Z$  arises from the parabolic nature of the fits and can be described by  $Z = (-B + \sqrt{B^2 - 4AC})/2C$ .

**Table 2:** Expression for the monolayer moisture content for the models in Table 1. Expressions are given in terms of the parabolic fit given in Equation (1).

Monolayer expression	Sorption isotherm model
$1/(B + 2CZ)$	GAB and Dent [30-33]
$(B + CZ)/[(B + C + CZ)(B + 2CZ)]$	HH [34]

These three models yield two different predictions for the monolayer moisture capacity. Furthermore, because the models are mathematically equivalent but derived from different physical assumptions, at most only one of these models can be correct. However, several authors [19, 35, 36] have shown that all three of these models yield incorrect predictions of the monolayer moisture

content as a function of temperature. While the ratio of monolayer water to “secondary” water (water that interacts with other water molecules in the cell wall) should increase with temperature (because secondary water is supposedly less tightly bound), in fact the ratio decreases (Figure 6).



**Figure 6:** Ratio of primary to secondary water as predicted by the GAB [30-32], Dent [33], and Hailwood Horrobin isotherms [34]. Replotted from [19].

Therefore, it must be concluded that while providing a good fit to the data these parabolic models do not yield physically meaningful parameters of the wood cell wall.

#### 5 RECOMMENDATIONS: DVS RESET

The following best practices are suggested when using automated sorption balances to acquire sorption isotherm data:

- A stop criterion of  $20 \mu\text{g}_{\text{water}} \text{g}_{\text{dry}}^{-1} \text{min}^{-1}$  will result in large systematic errors in the equilibrium moisture content. A more strict stop criterion should be used when collecting data for scientific purposes.
- The PEK model is not physically meaningful. The model parameters change drastically depending on data collection time. New kinetic models are needed to understand sorption kinetics.
- Commonly used “parabolic” isotherm models are useful for interpolating data points. However, the model parameters from these fits are not physically meaningful and cannot be used to understand the thermodynamics of water in wood.

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