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Interlaboratory study of automated sorption measurements in wood: method for correcting systematic errors with the commonly used 0.002% min^{-1} stop criterion

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Abstract
Many studies that use an automated sorption balance to determine a water vapor sorption isotherm for wood collect data until the moisture content change is less than or equal to 0.002% min^{-1} (20 µg g^{-1} min^{-1}). This stop criterion has been claimed to give errors in equilibrium moisture content (EMC) predictions of less than 0.001 g g^{-1} but over the past 10 years, studies have shown that the actual errors can be greater than 0.01 g g^{-1} because the measurements are stopped well before equilibrium is reached. Despite the large errors associated with this stop criterion, it remains popular due to the speed at which isotherms can be measured. This paper utilizes data from a worldwide interlaboratory study on automated sorption balances to develop a correction method for estimating EMC of western larch (*Larix occidentalis* Nutt.) from the moisture content corresponding to the 20 µg g^{-1} min^{-1} criterion. The study uses data from 72 relative humidity absorption steps with hold times of 7–10 days from 21 different laboratories and eight different instrument models. EMC is defined based on the inherent mass stability of automated sorption balances determined in the first part of this interlaboratory study. On average the sorption process is less than 80% complete when the 20 µg g^{-1} min^{-1} criterion is reached, resulting in a mean absolute error (MAE) of 0.006 g g^{-1}. The correction equation for estimating EMC reduces the MAE to 0.001 g g^{-1}. The analysis presented in this paper, along with the correction equation, can be considered for certain use cases to reduce systematic errors and shorten measurement times.

Keywords Water vapor sorption · Dynamic vapor sorption · Sorption isotherm · Interlaboratory study · Equilibrium moisture content

1 Introduction

Water vapor sorption isotherms describe the relationship between the equilibrium moisture content (EMC) of wood and the relative humidity at constant temperature and are important for wood science and practical utilization of wood as a building material. In theory, water vapor sorption measurements are easy; however, in practice there are many variables that can affect the accuracy of the measurements [1]. It is becoming increasingly common that sorption isotherms are collected with instruments called automated sorption balances that maintain stable temperature, control relative humidity using mass flow controllers to precisely mix streams of dry and saturated carrier gas, and continuously monitor specimen mass using a microbalance (Fig. 1). Automated sorption balances that mix two streams of gas

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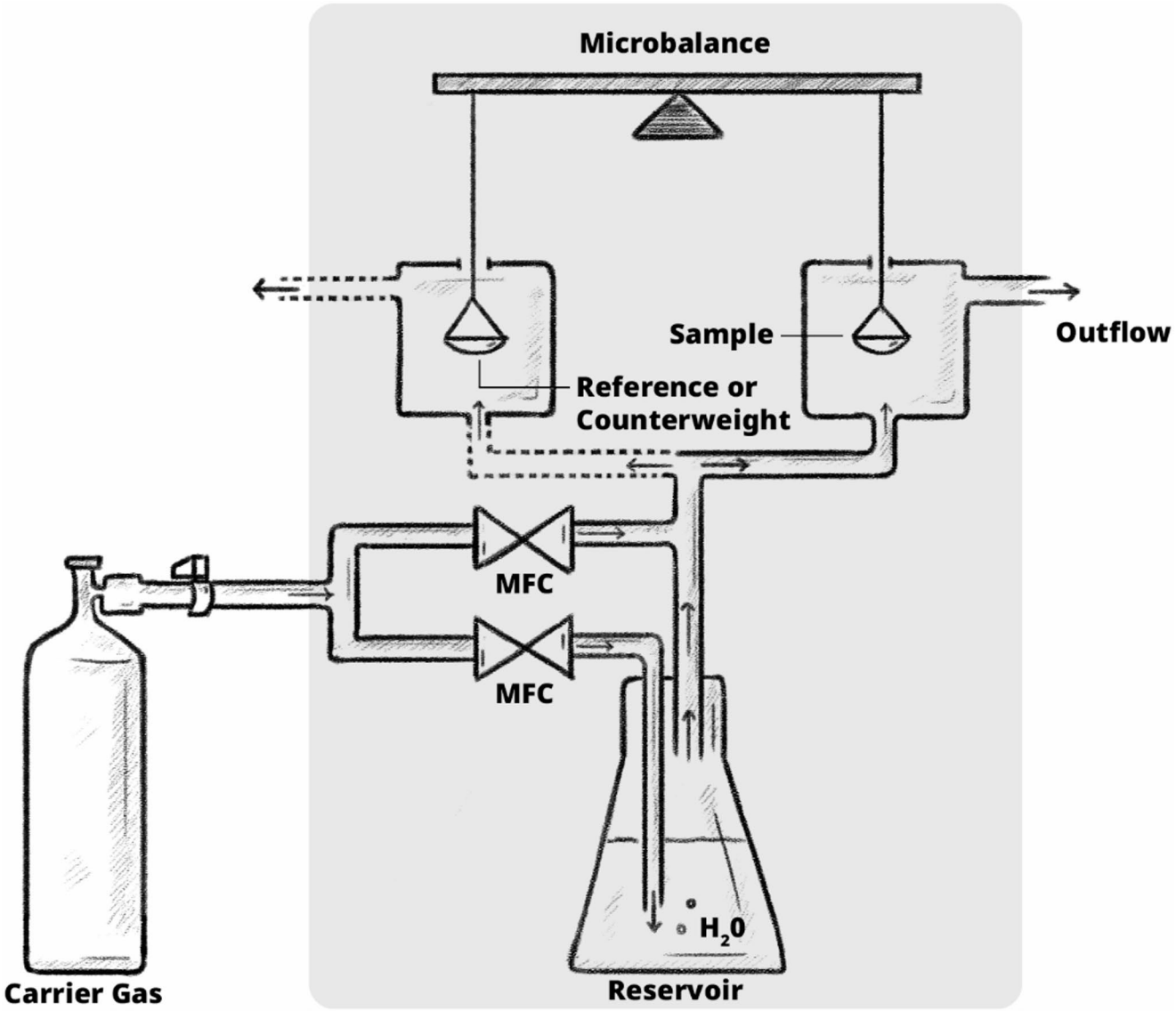

Fig. 1 Generic schematic of an automated sorption balance. Note that some instruments isolate the microbalance counterweight whereas in others an empty reference pan is exposed to the same environment as the sample (illustrated by the dotted lines in the figure). The abbreviation MFC refers to "mass flow controller". Illustration by Dorothy Punderson of the US Forest Service, Forest Products Laboratory

are also often referred to as "dynamic vapor sorption" or "DVS" instruments.

With automated sorption balances, it is possible to acquire an entire sorption isotherm on a sample with no user interaction. For absorption experiments, a sample is placed within an instrument and initially dried. The instrument is programmed to measure the mass while keeping temperature and humidity constant. This continues for a duration (hold time) defined by a stop criterion where the rate of change in relative mass (moisture content) meets a pre-set threshold value. At this point, the instrument ramps up the humidity to the next step (typically in steps of 5% or 10%) and continues to measure the mass under constant environmental conditions until the stop criterion is met at the new humidity level. This goes on until a maximum relative humidity is reached (typically 90% or 95%). In many cases the desorption isotherm is acquired after the absorption isotherm, and desorption is initiated from the last humidity level in the absorption experiment. However, it is important to note that these are scanning isotherms and do not represent true desorption from the saturated state [2]. The quality of sorption isotherms measured with automated sorption balances is strongly dependent on selecting an appropriate stop criterion for the material of interest.

Some of the first manuscripts published using the dynamic vapor sorption technique on wood utilized a stop criterion of a change in moisture content of 0.002% min^{-1} (equivalently, this can be expressed as a mass change of 20

micrograms per gram of dry material per minute, i.e., 20 µg g^{-1} min^{-1}) over a 10-minute window[1]. We notate this stop criterion as S_{10}^{20} where the superscript represents the magnitude of the change in mass relative to dry mass (in µg g^{-1} min^{-1}) and the subscript represents the duration over which the relative mass change was measured (in minutes). Typically, the instrument performs a linear regression of mass vs. time data (looking back) over the specified time window. Hill et al. [3] stated that the S_{10}^{20} stop criterion gives errors in EMC that are less than 0.001 g g^{-1}; however, Glass et al. later found no experimental evidence for this claim [4] and instead showed that absolute errors could be as high as 0.01 g g^{-1} when compared against a measurement where the stop criterion was set equal to the inherent stability of the microbalance over a 24-h duration, which they called "operational equilibrium" [5].

To collect automated sorption measurements with lower errors, Glass et al. proposed two different stop criteria [5]. The first suggestion was to use a S_{120}^{3} stop criterion (that is 3 µg g^{-1} min^{-1} calculated using a 120-minute running slope). The second suggestion was to use a S_{60}^{5} stop criterion (that is 5 µg g^{-1} min^{-1} calculated using a 60-minute running slope) and apply a correction factor for the underprediction caused by the shorter hold times. Glass et al. noted that S_{60}^{5} stop criterion caused an approximately 2% underprediction in the EMC for absorption steps of 10% RH from examining 7 different measurements on holocellulose, microcrystalline cellulose, and loblolly pine (*Pinus taeda*) to equilibrium. Multiplying the mass measured at the S_{60}^{5} stop criterion by this correction factor resulted in mean absolute error of 0.001 g g^{-1} and a maximum error of 0.002 g g^{-1} in the prediction of the EMC.

The papers by Glass et al. [4, 5] highlighting potential issues with less-strict stop criteria yielding short hold times which affect the accuracy of the EMC prediction are highly cited. However, the suggestions are not widely used. For example, of the six papers published in 2023 [6–11] that present sorption data and cite Glass et al. [5], only one paper [7] used a stop criterion of less than 5 µg g^{-1} min^{-1} and none of the papers applied a correction factor for data acquired at short hold times.

One reason that researchers may continue to use less-strict stop criteria, including S_{10}^{20}, can be practical issues with the longer hold times associated with stricter stop criteria[2]. Glass et al. [4] demonstrated that the relationship between absolute error and measurement time is nonlinear with extremely long hold times (3 to 7 days *per RH step*) needed to reach the smallest errors in the EMC prediction. Therefore, there is a need for a correction method for data collected using the commonly used S_{10}^{20} stop criterion along with a better estimation of the errors using this stop criterion with and without correction.

This paper is part of an interlaboratory study whose goal was to develop better information about the relationship between the moisture content when the measurement is stopped at S_{10}^{20} as opposed to operational equilibrium. It builds upon two previous papers from the interlaboratory study that examined the inherent stability of different instruments [12] and the data quality of long hold time sorption measurements on wood samples [13]. The study included 80 measurements on wood with a long hold time (168–240 h) collected from twenty-one different laboratories spanning North America, Europe, and Asia on eight different models of commercially available automated sorption balances. Our previous paper [13] analyzed the data quality of these measurements and found that 72 of the 80 measurements (90%) contained a moisture content uncertainty of less than 0.0005 g g^{-1} which correlates with a rating of "good" based on examinations of previous sorption studies in wood [1].

In this paper, we utilize the 72 good datasets from the interlaboratory study to develop a correction method for data collected with the S_{10}^{20} stop criterion. We first provide an operational definition of equilibrium for data acquired at extended times (7–10 days) with automated sorption balances. We then identify a correction equation for estimating EMC from the moisture content corresponding to the S_{10}^{20} stop criterion and discuss the magnitude of errors using this criterion with and without correction. Additionally, we present a sorption isotherm for western larch (*Larix occidentalis* Nutt.) based on data from 21 laboratories.

2 Materials and methods

2.1 Materials

Laboratories were given two different types of samples: a non-hygroscopic reference material to test the stability of the instrument and wood samples to conduct sorption experiments. Laboratories were provided with aluminum Differential Scanning Calorimetry (DSC) sample pans (Tzero® sample pan, TA Instruments, New Castle, DE, USA) as a non-hygroscopic reference material. DSC sample pans were chosen as a reference because they have a relatively uniform mass (ca. 41 mg), are low cost, easily obtainable, and non-hygroscopic.

Wood samples consisted of 6 mm by 6 mm by 1 mm cross sections of western larch (*Larix occidentalis* Nutt.)

[1] Note that this stop criterion also had a 10-minute "stability period".

[2] Another complication may be that some commercial software packages do not allow a slope window longer than 10 min, thus prohibiting the use of stricter mass change criteria such as S_{60}^{5}; however, it is still possible to achieve any arbitrarily strict stop criterion by running the experiments for a fixed time as we did in this interlaboratory study.

heartwood. Samples were cut from a single US nominal "2 × 4", a board with dimensions of 38 mm by 89 mm by 2.44 m. The board was chosen from a large pallet of visually graded #1 boards of the "Douglas Fir-Larch" sawn lumber commercial species combination [14]. The board was chosen because it was free of knots and had a straight grain pattern. Samples were cut along the true anatomical directions with the shortest dimension (1 mm) in the longitudinal direction to allow direct access for water vapor to all cell wall surfaces [15, 16]. Samples had a dry mass of around 20 mg. Full details of how the cross sections were obtained can be found elsewhere [13].

2.2 Experimental methods

The major objective of the interlaboratory study was to acquire sorption data to "operational equilibrium" (defined below in Sect. 2.3) on a broad range of instruments and relative humidity steps. Other objectives and data from the interlaboratory study can be found in our earlier papers on instrument stability [12] and data quality for western larch specimens [13]. Only details relevant to the analysis presented in this paper are included here.

2.2.1 Instrument stability

To measure the instrument stability, each laboratory was asked to perform a measurement on the non-hygroscopic reference material (DSC sample pan). The measurement consisted of a 3000-minute measurement at 25 °C and 50% RH with a carrier gas flow rate of 200–250 cm^3 min^{-1} and a data recording interval of less than or equal to 1 min. The instrument stability was characterized by several different metrics, including a 24-hour running slope, first suggested by Glass et al. [5] for characterizing instrument stability. The maximum 24-hour slope over the 3000-minute window was recorded for each laboratory and tabulated. Across the participating laboratories, the mean, median, maximum, and minimum values were 2.8 µg day^{-1}, 2.3 µg day^{-1}, 8.7 µg day^{-1}, and 0.4 µg day^{-1}, respectively [12].

Table 1 Number of datasets with long hold time taken at each relative humidity (RH) step along with the number of instruments used to collect the data

Initial RH (%)	Final RH (%)	Instruments	Datasets	Acceptable Datasets
0	10	4	5	5
10	20	6	16	15
20	30	4	6	6
30	40	4	6	6
40	50	4	7	7
50	60	4	4	4
60	70	4	8	6
70	80	4	6	6
80	90	5	11	8
90	95	5	11	9

2.2.2 Sorption measurements on wood samples

The same data acquisition rate, carrier gas flow rate, and temperature were used for sorption measurements on western larch specimens. Prior to the sorption steps, the sample was first held at 0% RH (dry carrier gas) at 25 °C for 24 h, yielding a dry mass, m_{dry}. This m_{dry} may not be the complete anhydrous state of the wood cell wall (see [7]); however, the starting conditions should be similar across laboratories and the dry mass would not affect the sorption kinetics which was the goal of the study, rather it would only influence the moisture content calculations. Instruments were programmed to use the mass after this first step as the dry mass of the sample when calculating relative mass changes. Sorption sequences had one "long" step held at a final RH value listed in Table 1, preceded by anywhere from zero to nine "short" steps at each of the RH levels below the final RH. The "long" step had a specified duration of seven days for RH values less than 90% and 10 days for RH values at 90% and 95%. The stop criterion for the "short" steps was a relative mass change of 0.002% min^{-1} within a 10-minute window (i.e. S_{10}^{20}), with a maximum time of 600 min per step. An example protocol for a "long step" at 40% RH is as follows:

- Target: 0% RH, Stop criterion: Time, Period: 24 h (=1440 min).
- Target: 10% RH, Stop criterion: Relative mass change, Value: 0.002% min^{-1}.
- Target: 20% RH, Stop criterion: Relative mass change, Value: 0.002% min^{-1}.
- Target: 30% RH, Stop criterion: Relative mass change, Value: 0.002% min^{-1}.
- Target: 40% RH, Stop criterion: Time, Period: 168 h (=10080 min).

The original study plan called for at least five replicates of long hold times for each RH step and at least one replicate from each instrument manufacturer. A list of the instruments used in the study is included in Table 2. As is common in interlaboratory studies, some laboratories were unable to complete the full range of measurements, but other laboratories provided additional data. In addition, some laboratories were asked to repeat measurements with noticeable abnormalities in the sorption data. In total 80 measurements were taken to operational equilibrium. For each relative humidity step, at least four datasets (replicates) were collected, from at a minimum of four different instruments (laboratories).

Table 2 List of instruments used in the interlaboratory study

Manufacturer	Model(s)
Hiden Isochema Ltd.	IGAsorp
Surface Measurement Systems Ltd.	DVS Advantage, DVS Advantage Plus, DVS Endeavour, DVS Intrinsic, DVS Resolution
TA Instruments – Waters LLC	Discovery SA, Q5000 SA

The distribution of measurements across the different RH steps is given in Table 1. Of these 80 measurements, 72 contained a moisture content uncertainty of less than 0.0005 g g^{-1} which was considered acceptable [13]. Table 1 lists both the number of datasets collected and the number of "acceptable datasets" at each RH condition.

The interlaboratory study focused on the relationship between moisture content at short hold times and operational equilibrium within the same dataset. Because of this focus, laboratories were not asked to validate the accuracy of the relative humidity control within the instruments. Therefore, while the data from all laboratories can be combined and plotted as a sorption isotherm, it should not be considered a "reference isotherm" for western larch.

2.3 Data analysis

For this paper, data analysis consisted of comparing the moisture content for each dataset at the S_{10}^{20} criterion against the equilibrium moisture content as defined below. Only datasets with an acceptable level of uncertainty in the moisture content determination were included [13].

Glass et al. [5] defined operational equilibrium for an automated sorption balance to be a mass change that is less than or equal to the inherent mass stability of the instrument over a 24-h duration. In the first leg of the interlaboratory study, laboratories measured the stability of the microbalance using an inert sample pan (Sect. 2.2.1). The mean 24-hour slope was 2.8 µg day^{-1}, which was rounded up to 3 µg day^{-1} to set the operational equilibrium definition for this study. For comparison with operational definitions in the broader literature on water vapor sorption in wood [1] not only those using automated sorption balances, this 24-h slope of 3 µg day^{-1} for a specimen dry mass greater than 20 mg corresponds with a relative mass change of less than 0.1 µg g^{-1} min^{-1} at the end of the sorption process. This qualifies as "very good" according to the evaluation criteria of Zelinka et al. [1], and it is stricter than the values used for two of the three literature datasets that were considered reliable.

Figure 2 illustrates the procedure for identifying operational equilibrium. On the top panel of the figure the mass is plotted in blue, and a linear fit over a 24-hour window is plotted in red. The bottom panel shows the slope of the linear fit based upon this 24-hour running window along with the point at which the operational equilibrium criterion of 3 µg day^{-1} is achieved. In three of the 72 datasets, the 24-h slope did not reach this 3 µg day^{-1} criterion during the measurement period; however, the 24-h slopes in these three cases were all below 4 µg day^{-1}, and the mass at the end of the measurement period was used as the equilibrium value.

The S_{10}^{20} criterion was implemented as a relative mass change of 20 µg g^{-1} min^{-1} calculated with a 10-minute running slope based on linear regression of mass vs. time data, relative to dry mass. It should be noted that some instruments also require a 10-minute "stability period" for the S_{10}^{20} criterion, meaning that the stopping point is delayed until the running slope remains less than or equal to 20 µg g^{-1} min^{-1} for a 10-minute period. The delay effect of the stability period depends on the noise level. In some cases, the stop criterion was reached 10 min after the running slope was less than 20 µg g^{-1} min^{-1} (Fig. 3, right side). However, in other cases where the data contained more noise, the relative mass change did not stay below 20 µg g^{-1} min^{-1} continuously for 10 min after first reaching this value (Fig. 3, left side). Throughout this paper we examine both stop criteria - the first time at which the slope reached 20 µg g^{-1} min^{-1} and also the case where a 10-minute stability period was required.

Finally, the data analysis requires an examination of the error with shorter hold times. We define the absolute error in the EMC prediction as the difference between moisture content at operational equilibrium and the moisture content at the stop criterion.

Additional datasets for water vapor absorption in holocellulose, loblolly pine, microcrystalline cellulose, and office paper published previously [5] were reanalyzed using the same methods described above to provide test cases for comparison with the western larch datasets in this study.

3 Results and discussion

3.1 Water vapor sorption isotherm for Western larch

The absorption isotherm at 25 °C is plotted in Fig. 4. The relative humidity values are plotted as the nominal target values. Equilibrium moisture content values are based on the criterion of 3 µg day^{-1}. The sorption isotherm is sigmoidal, as expected for water vapor sorption in wood. These results and all subsequent results are based on 72 out of 80 datasets that had an acceptable level of moisture content uncertainty as defined previously (Table 1; [13]; see Appendix for all data used to create this graph, i.e., the "Equilibrium"

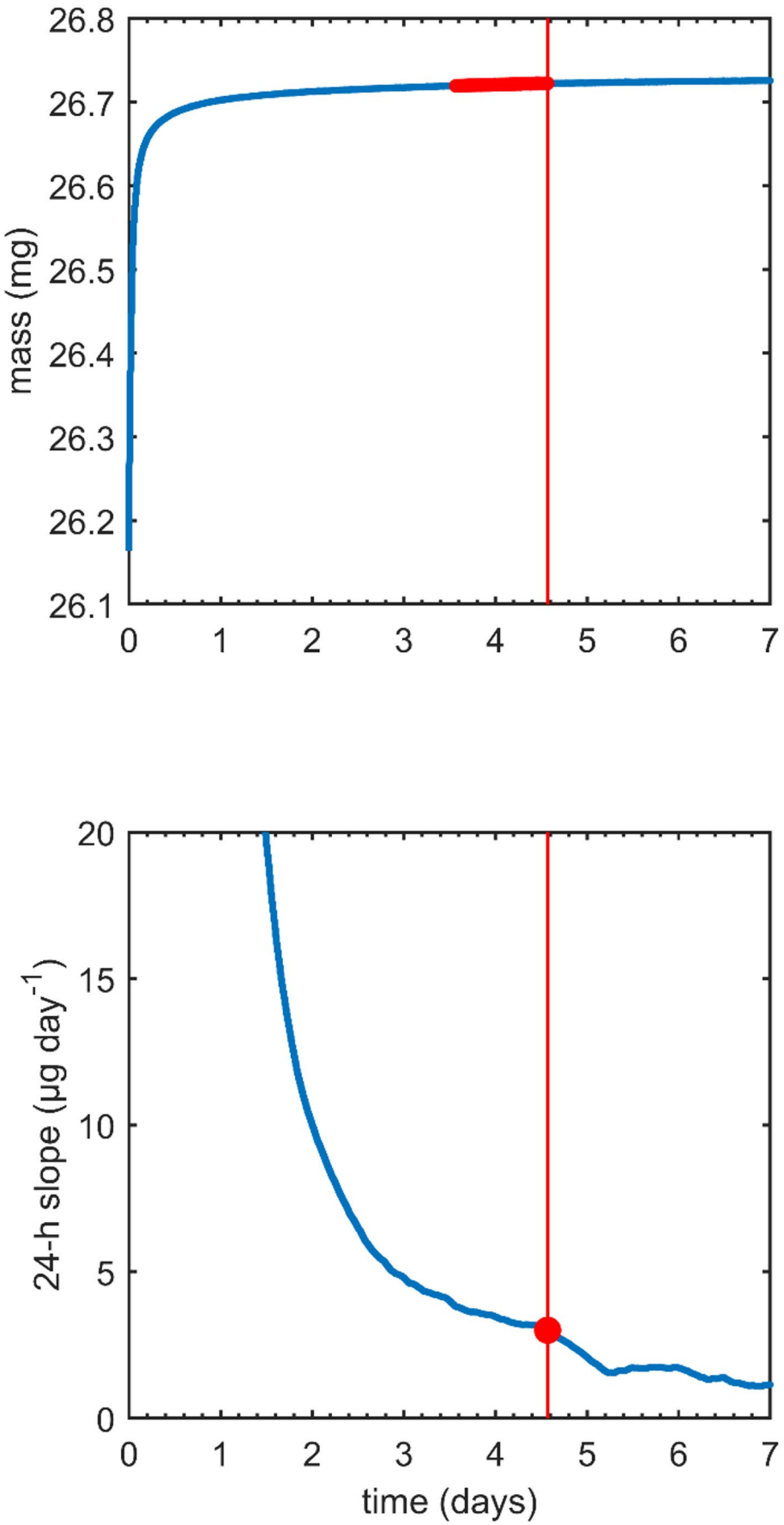

Fig. 2 Example showing how operational equilibrium was identified. (top) Mass as a function of time; the thick red line represents a linear fit to a 24-hour window of data. (bottom) 24-hour running slope. The red dot represents operational equilibrium where the 24-hour running slope first reaches 3 µg day^{-1}

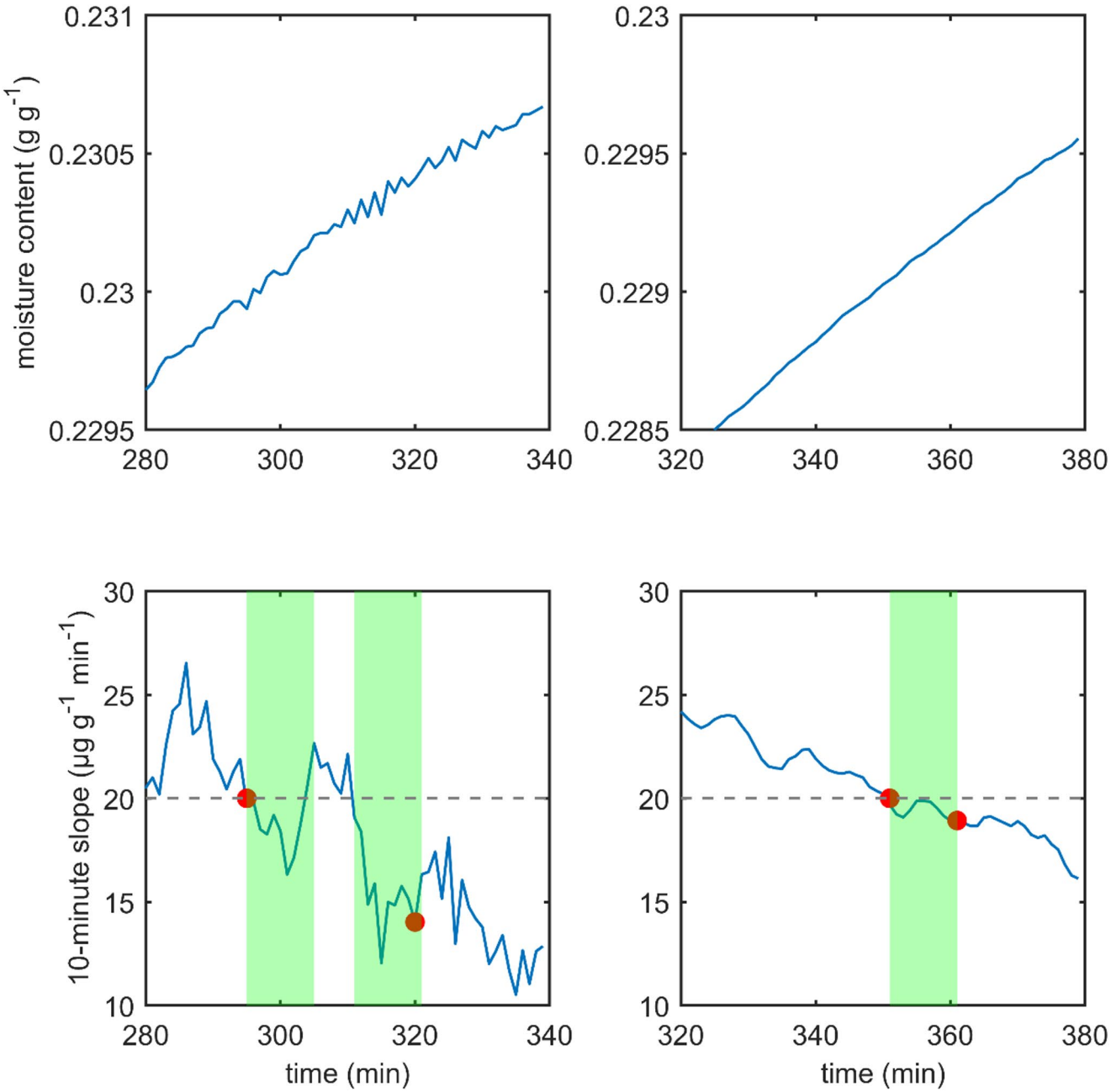

Fig. 3 Examples of how the stop criterion was implemented with the moisture content plotted in the top graphs and the 10-minute slope plotted on the bottom. The green region represents a 10-minute "stability period" and red dots represent the time at which the stop criterion was reached with and without a stability period. The left and right columns show data with different levels of noise

column in Table 6). The curve fit in Fig. 4 is the empirical ABC isotherm [17]

$$\frac{h}{u_{\mathrm{eq}}} = Ah^2 + Bh + C \tag{1}$$

where h is the relative humidity (Pa Pa^{-1}), u_{eq} is the equilibrium moisture content (g g^{-1}), and A, B, C are fitting parameters with no physical significance (–13.64, 14.63, and 2.40, respectively); note that the ABC isotherm is mathematically equivalent to the GAB, Hailwood-Horrobin, and Dent isotherms [18–23]. While the quality of data is evident,

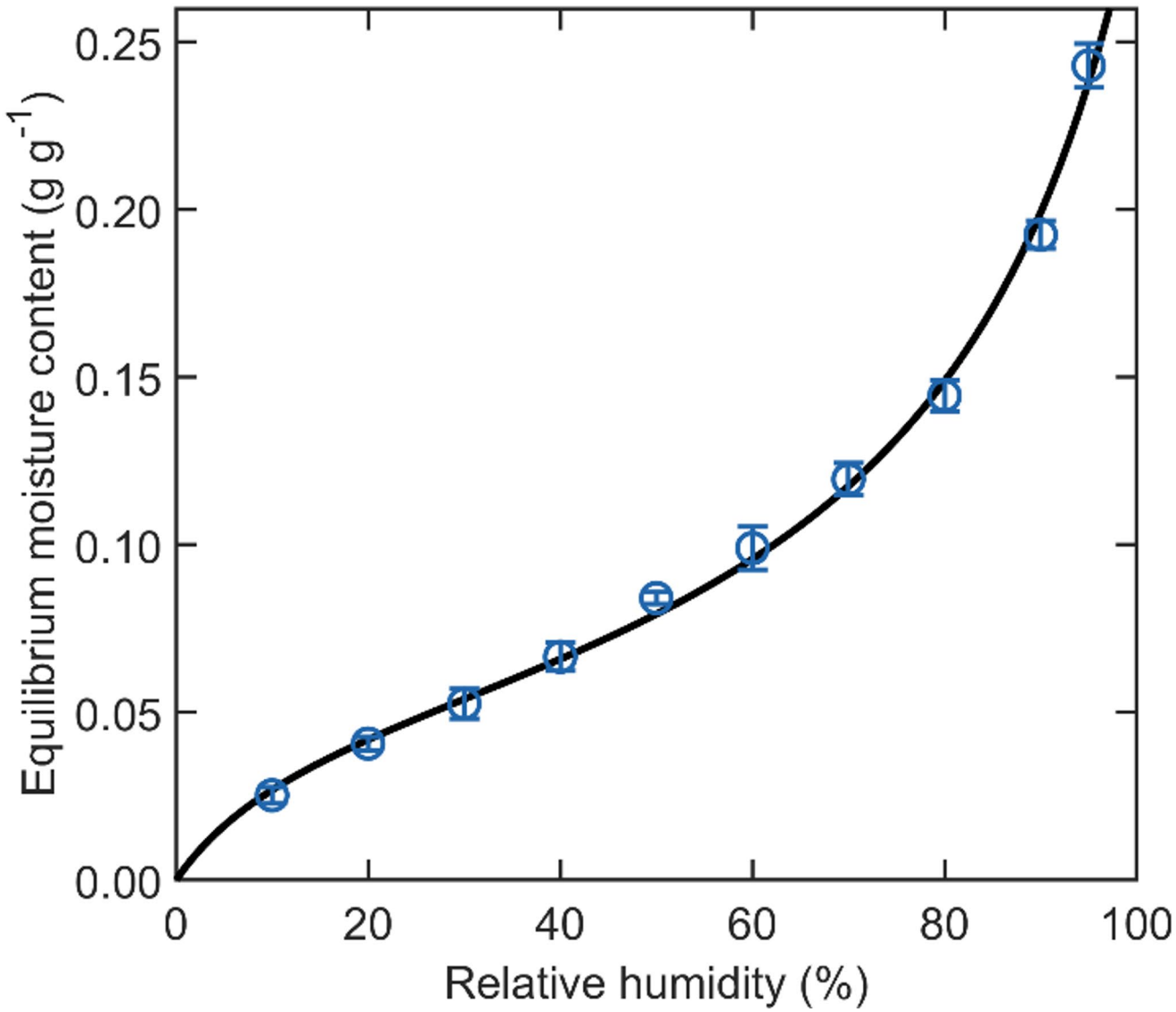

Fig. 4 Absorption isotherm of western larch at 25 °C (mean equilibrium moisture content ± standard deviation); curve fit is the empirical ABC isotherm; note that the standard deviation at each RH level is based on different numbers of replicates (Table 1)

this should not be considered a reference isotherm because the RH accuracy of each instrument was not verified.

3.2 Equilibrium moisture content prediction error for short hold times

3.2.1 Incomplete sorption at short hold times

Four example datasets are plotted in Fig. 5 in the form of $E(t)$, the fractional change in moisture content, defined as

$$E(t) = \frac{u_t - u_0}{u_{\mathrm{eq}} - u_0} \tag{2}$$

u_t is the moisture content (g g^{-1}) at time t, u_0 is the moisture content (g g^{-1}) at the beginning of the RH step, and u_{eq} is the equilibrium moisture content (g g^{-1}) defined by the operational equilibrium criterion of 3 µg day^{-1} and indicated by a red dot. Values corresponding to the S_{10}^{20} stop criterion with a 10-minute stability period are indicated by a red "×". These four plots clearly indicate that the moisture content is far from equilibrium when this lenient, yet commonly used stop criterion is met. This is not surprising given the relatively short hold time [4, 5]. Across all datasets, the S_{10}^{20} stop criterion took 2–8 h per RH step, whereas operational equilibrium required 36–194 h per RH step.

Mean values of the fractional change in moisture content at the S_{10}^{20} stop criterion for all datasets are shown in Fig. 6 as a function of relative humidity. This quantity is

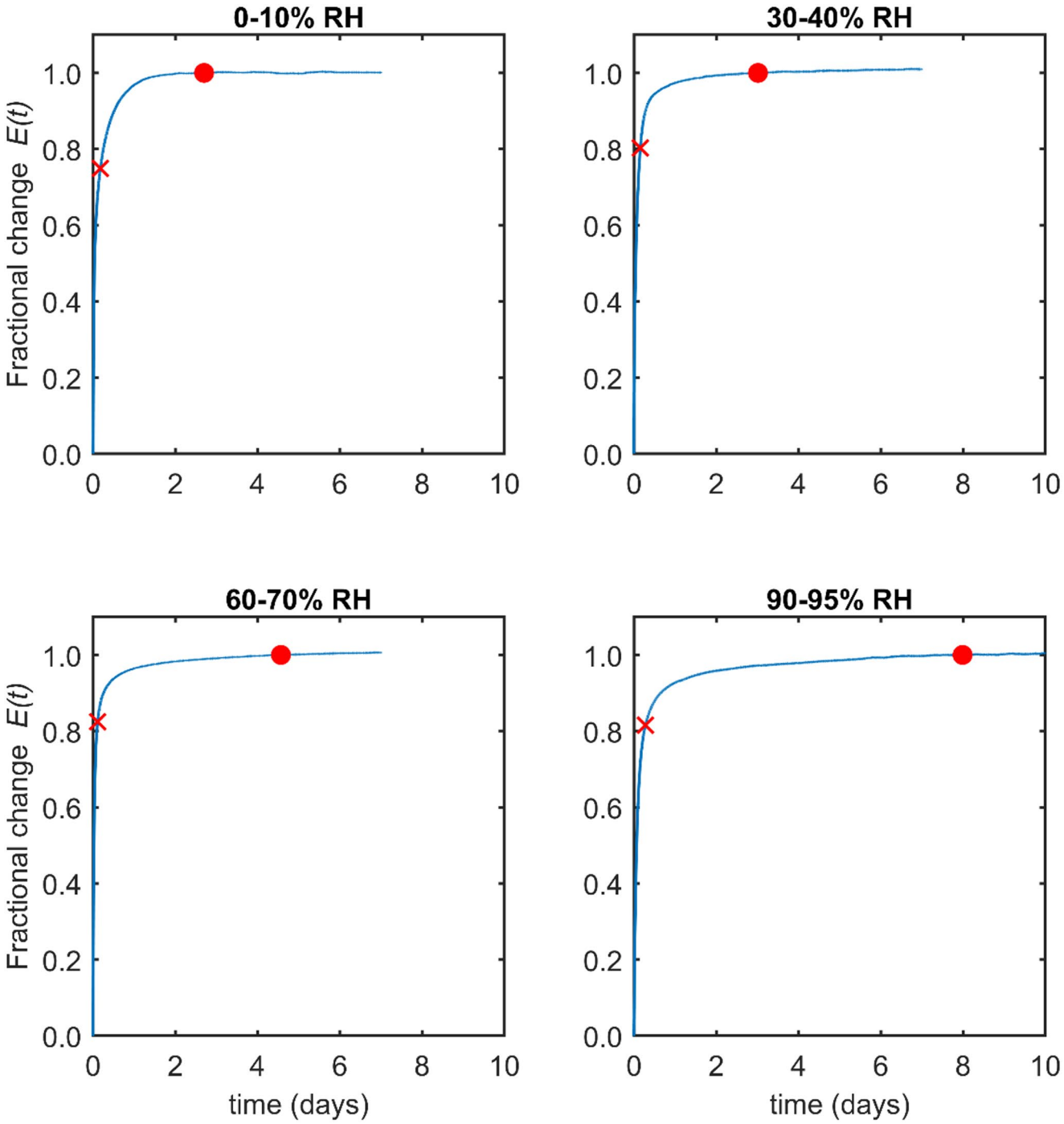

Fig. 5 Fractional change in moisture content E(t) vs. time for four example datasets- the red dot represents operational equilibrium (3 μg day^{-1}) and the red × represents the S_{10}^{20} stop criterion with a 10-minute stability period

abbreviated as $E\left(t_{\text{stop}}\left(S_{10}^{20}\right)\right)$. The mean values for all datasets in the 10%–95% RH range are 0.79 (with a 10-minute stability period) and 0.78 (without it), shown as dashed horizontal lines. The S_{10}^{20} stop criterion clearly fails to give valid approximations of equilibrium; on average the sorption process is less than 80% complete.

In addition to the large magnitude of this systematic error, two further issues should be noted. First, $E\left(t_{\text{stop}}\left(S_{10}^{20}\right)\right)$ varies considerably across different RH steps, which may reflect different underlying physical mechanisms in the sorption kinetics. Second, $E\left(t_{\text{stop}}\left(S_{10}^{20}\right)\right)$ varies considerably within a given RH step as shown by the standard deviation. This may reflect specimen heterogeneity as well

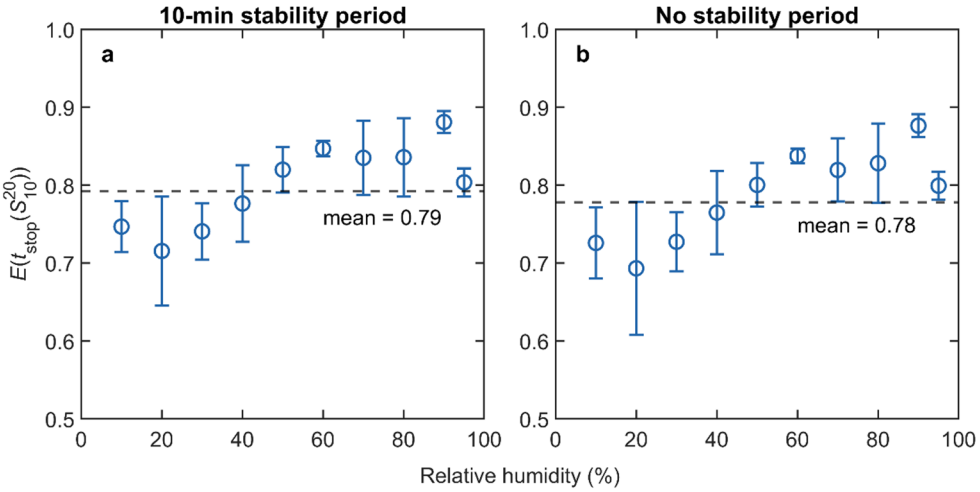

Fig. 6 Fractional change in moisture content with the S_{10}^{20} stop criterion with a 10-minute stability period (**a**) and without a stability period (**b**), plotted as mean ± standard deviation, for each relative humidity step; note that the standard deviation at each RH level is based on different numbers of replicates (Table 1); horizontal dashed lines represent mean values across all datasets in the 10–95% RH range

as differences in instrument mass stability between laboratories. The use of a 10-minute window for determining relative mass changes can produce appreciable fluctuations, as shown in Fig. 3 and noted previously [5], which translates into variation in the time needed to reach the stop criterion and therefore causes variation in the resulting moisture content at the stop criterion and the value of $E\left(t_{\text{stop}}\left(S_{10}^{20}\right)\right)$.

These considerations suggest that stricter stop criteria (i.e., longer time window and smaller threshold value of the relative mass change) would increase the value of $E(t_{\text{stop}})$ and reduce its variation for a given RH step. These improvements might yield a more straightforward RH-dependence, thereby making $E(t_{\text{stop}})$ more predictable. While this paper focuses on the S_{10}^{20} criterion, the relationship between stop criteria and the RH dependence of the error in the EMC prediction requires further study.

3.2.2 Absolute and relative errors in the EMC prediction

The difference between the EMC and the moisture content at the S_{10}^{20} stop criteria (i.e., the absolute error, $u_{\text{eq}} - u_{\text{stop}}$, where u_{stop} is the moisture content (g g^{-1}) at the stop criterion) is plotted in Fig. 7 (top row). Absolute errors of 0.01 g g^{-1} or greater can be observed in both the low moisture content range and the high moisture content range. Absolute errors of this magnitude (or larger) resulting from the same S_{10}^{20} stop criteria were previously reported in the high moisture content range for lignin [4] and loblolly pine [5]. However, the large absolute errors in the low moisture content range observed here differ from previous results for loblolly pine, holocellulose, microcrystalline cellulose, and office paper [5]. That study found small absolute errors in the low moisture content range and an apparent linear relationship between absolute error and moisture content using a stricter S_{60}^{5} stop criterion. The relationship between absolute error and moisture content in Fig. 7 is clearly nonlinear. The absolute errors are fitted with a parabola in Fig. 7a and c.

Relative errors in the EMC prediction are shown in the bottom row of Fig. 7. For low moisture contents, the relative errors are on the order of 30%. Such large relative errors in this range stem from the combination of large absolute error (large numerator) and small equilibrium moisture content (small denominator). As the moisture content increases to approximately 0.10 g g^{-1}, the absolute error decreases, yielding a dramatic decrease in the relative error to approximately 5% (the numerator decreases while the denominator increases). With further increase in moisture content, the relative error changes only slightly.

The reason for nonlinearity in the relationship between absolute error and moisture content can be explained based on two factors. The first is how complete the sorption process is when the stop criterion is reached. The normalized values in Fig. 6 quantify the extent of completion from the initial state to the equilibrium state. The fractional sorption deficit (or extent of incompleteness) would then be given by the quantity $1 - E(t_{\text{stop}})$, as shown in Fig. 8a with mean values at each RH level for the S_{10}^{20} stop criterion with a 10-minute stability period. The second factor is the sigmoidal shape of the sorption isotherm (see Fig. 4). Each RH step (0–10, 10–20, and so on) corresponds to a different incremental increase in moisture content, as shown in Fig. 8. This increase is moderate at low RH, reaches a minimum in the 30–60% RH range, and then increases with RH. The absolute error in the EMC prediction is the product of the two factors above (as shown explicitly below), which is plotted

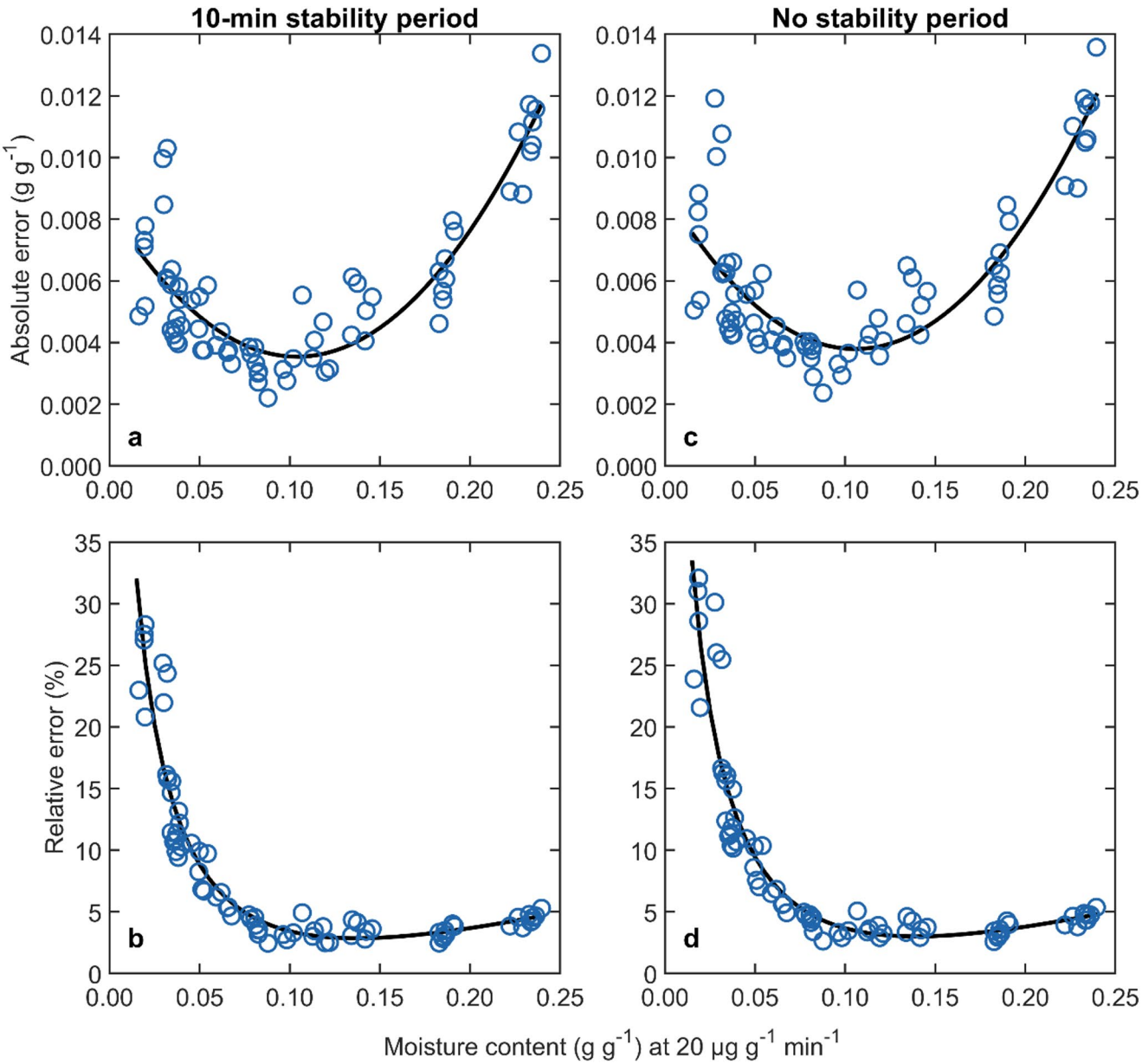

Fig. 7 Absolute error (top row) and relative error (bottom row) in the EMC prediction as a function of moisture content with the S_{10}^{20} criterion with (**a**, **b**) a 10-minute stability period and without a stability period (**c**, **d**). Curve fits to absolute error are parabolic (**a**, **c**); curve fits to relative error (**b**, **d**) are calculated based on those in (**a**, **c**)

in Fig. 8c. The RH dependence of these two factors (and the fact that moisture content increases monotonically with RH) explains why absolute error is large in the low and high moisture content ranges and has a minimum in the medium moisture content range.

The relationships discussed above are justified mathematically as follows. The fractional change in moisture content at the stop criterion, $E(t_{\text{stop}})$, is given by

$$E\left(t_{\text{stop}}\right) = \frac{u_{\text{stop}} - u_0}{u_{\text{eq}} - u_0} \tag{3}$$

where u_{stop} is the moisture content (g g^{-1}) at the stop criterion, u_0 is the moisture content (g g^{-1}) at the beginning of the RH step, and u_{eq} is the equilibrium moisture content (g g^{-1}). It then follows that

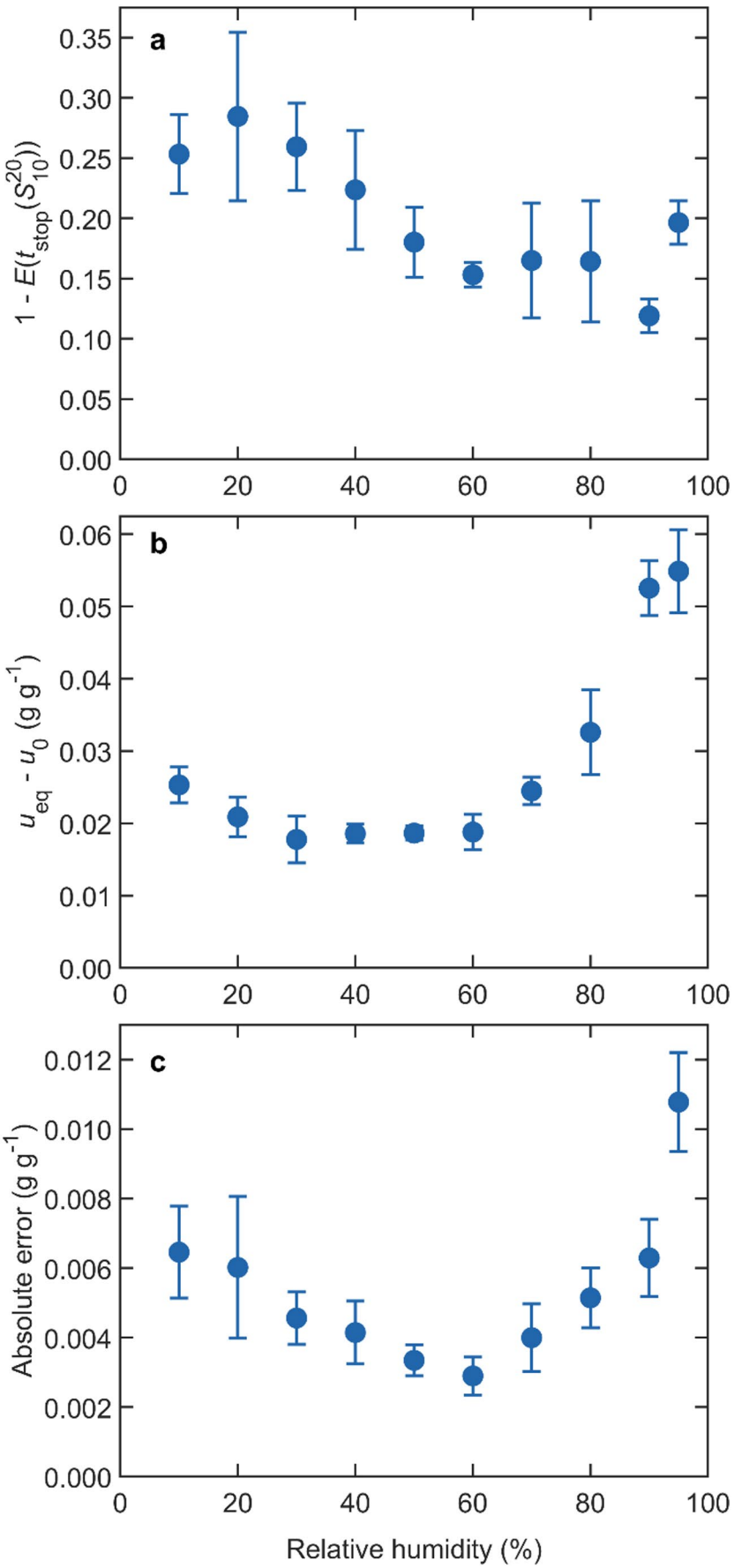

Fig. 8 Fractional sorption deficit from the S_{10}^{20} stop criterion with a 10-minute stability period (**a**), incremental change in moisture content from initial state to equilibrium state (**b**), and resulting absolute error (**c**) for each relative humidity step. Error bars represent the standard deviation

$$1 - E\left(t_{\text{stop}}\right) = \frac{u_{\text{eq}} - u_{\text{stop}}}{u_{\text{eq}} - u_0} \tag{4}$$

The absolute error in EMC for a given RH step is equal to the numerator on the right side of Eq. (4). Rearranging Eq. (4) gives

$$u_{\text{eq}} - u_{\text{stop}} = \left[1 - E\left(t_{\text{stop}}\right)\right]\left(u_{\text{eq}} - u_0\right) \tag{5}$$

Thus Eq. (5) expresses the same relationship discussed above and illustrated in Fig. 8 between the fractional sorption deficit, incremental change in moisture content, and absolute error.

As mentioned above, an apparent linear relationship between absolute error and moisture content was previously noted for the S_{60}^{5} criterion [5]. The analysis here implies that, for this relationship to be linear, the RH-dependence of $E\left(t_{\text{stop}}\right)$ would need to compensate to effectively cancel the nonlinearity of the sorption isotherm. The influence of the strictness of the stop criterion on the RH-dependence of $E\left(t_{\text{stop}}\right)$ is yet to be determined. Along these lines, we suggest that the apparent linear behavior previously noted [5] be further investigated.

3.3 Correction method for accurate estimation of the EMC with the S_{10}^{20} stop criterion

The time required for reaching equilibrium has an associated cost. One of the features of automated sorption balances is the ability to stop data collection at a given RH step based on a pre-programmed criterion. As shown above, the S_{10}^{20} criterion requires much less measurement time than the operational equilibrium criterion but yields large errors. However, the fact that these errors can be fit with a curve (Fig. 7) means that a correction can be applied to data acquired with the S_{10}^{20} criterion to give a reasonably accurate estimate of the equilibrium moisture content.

The parabolic curve fit to the absolute error in Fig. 7 can be expressed as

$$u_{\text{eq}} - u_{\text{stop}} = k_0 + k_1 u_{\text{stop}} + k_2 {u_{\text{stop}}}^2 \tag{6}$$

where k_0, k_1, and k_2 are empirical fitting parameters. Equation (6) can then be rearranged so that the equilibrium moisture content is simply a function of the moisture content at the stop criterion. This estimated EMC is denoted $\hat{u}_{\text{eq}}$ and is given by:

$$\hat{u}_{\text{eq}} = k_0 + \left(k_1 + 1\right) u_{\text{stop}} + k_2 {u_{\text{stop}}}^2 \tag{7}$$

For the case with the 10-minute stability period, $\{k_0, k_1, k_2\}$ = {0.0084, −0.0926, 0.4448}. For the case without the stability period, $\{k_0, k_1, k_2\}$ = {0.0089, −0.0970, 0.4593}.

Applying the correction in Eq. (7) to the measured value of u_{stop} for each data point yields a set of $\hat{u}_{\text{eq}}$ values that can be compared with the measured u_{eq} values. These are plotted for the S_{10}^{20} stop criterion in Fig. 9, along with residuals. The residuals appear to be randomly distributed. For data with the 10-min stability period, the root mean square error (RMSE) is 0.0012 g g^{-1}, the mean absolute error (MAE) is 0.0009 g g^{-1}, and 95% of the residuals fall within the interval [–0.0022 g g^{-1}, +0.0022 g g^{-1}]. For data without the stability period, the RMSE is 0.0014 g g^{-1}, the MAE is 0.0010 g g^{-1}, and 95% of the residuals fall within the interval [–0.0025 g g^{-1}, +0.0025 g g^{-1}]. The parabolic equation in Eq. (7) is a significant improvement over linear correction equations, but correction equations with four or more fitting parameters do not give significant improvement over the three-parameter parabolic equation (see Appendix).

3.4 Applicability of the correction equation to other materials

The method for developing an empirical correction for systematic error in the EMC prediction described here for western larch could be replicated for other materials if they used the same 10% RH steps. Development of a correction equation requires quality data at extended times across a range of moisture contents for the specific material of interest. Such data are rare and require a lengthy amount of time to acquire, so it is reasonable to ask whether the correction equation in this paper is directly applicable to other materials. Although the data in this study are broad in terms of the number of laboratories and number of replicates across each RH step, the data are limited in the sense that all specimens were cut with the same orientation and dimensions from one piece of western larch lumber and measurements were taken at one temperature, in absorption starting from the dry condition with a single carrier gas flow rate.

We test the applicability of Eq. (7) with the fit parameters from western larch using data on four materials from Glass et al. [5] using similar RH steps: holocellulose, loblolly pine, microcrystalline cellulose (MCC), and office paper. The absolute errors in the EMC prediction using the S_{10}^{20} criterion are plotted in Fig. 10, along with the parabolic

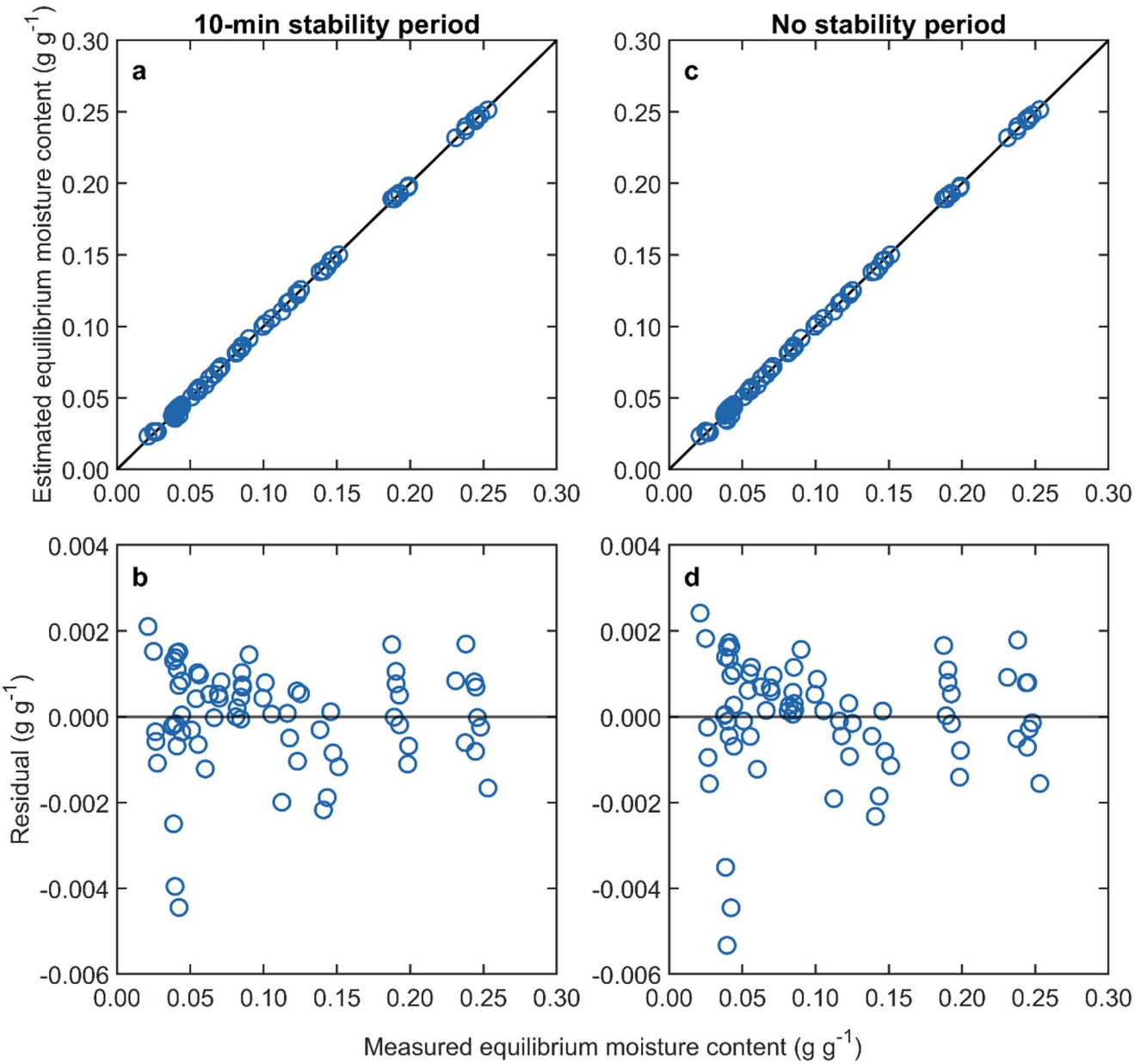

Fig. 9 Estimated equilibrium moisture content calculated from the moisture content with the S_{10}^{20} criterion and corrected with Eq. (7) (top row) and residuals (bottom row) vs. measured equilibrium moisture content, with a 10-minute stability period (**a**, **b**) and without a stability period (**c**, **d**); solid lines in top row represent y = x

curve fits for western larch. At low moisture contents, these materials all had lower absolute errors than western larch.

The results of applying the western larch correction to these different materials are summarized in Table 3, where prediction error refers to the difference between the estimated equilibrium moisture content using the correction in Eq. (7) and the measured EMC of the material. A summary of these results is shown in Fig. 11 which plots the mean absolute error in the prediction of EMC with the S_{10}^{20} stop criterion with and without the Eq. (7) correction applied. While the correction dramatically improves the western larch data that was used to develop Eq. (7), the correction also reduces the mean absolute error for the other materials

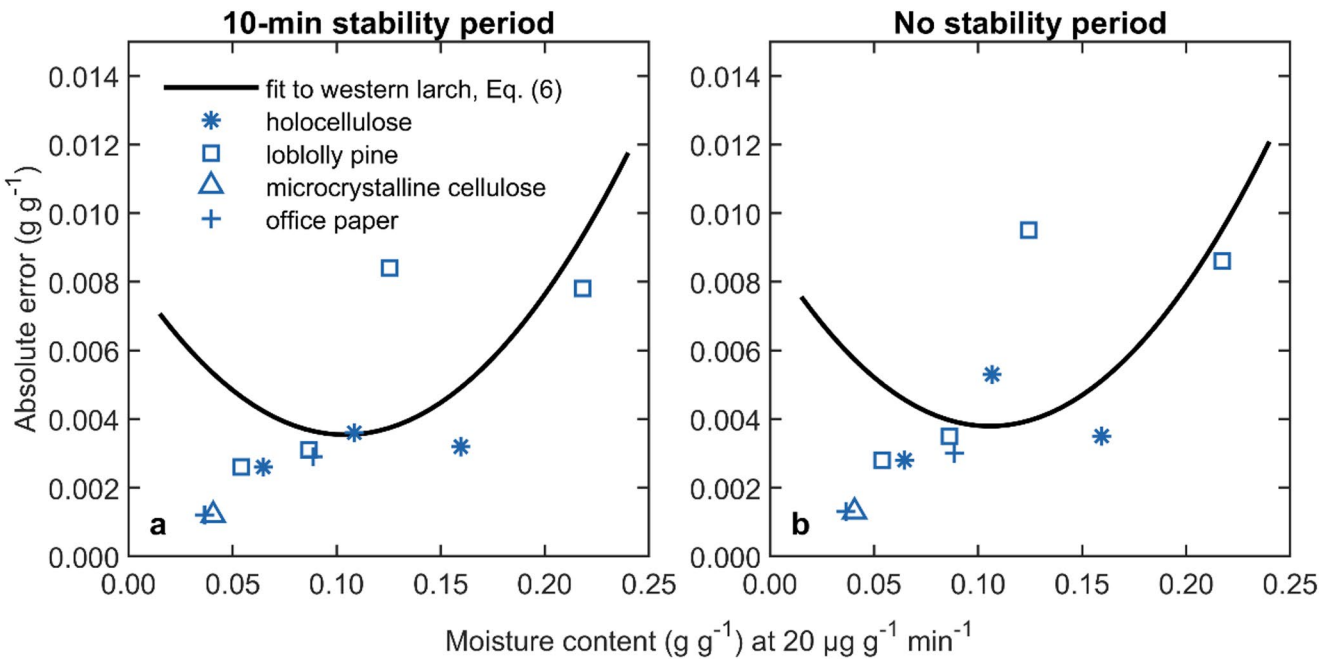

Fig. 10 Absolute error in the EMC prediction as a function of moisture content with the S_{10}^{20} stop criterion with a 10-minute stability period (**a**) and without a stability period (**b**) for various materials. Data from Glass et al. [5]. The "fit to western larch" curve uses data from Fig. 7

Table 3 Application of the Western larch correction to other materials

Material	RH step	u_{eq} (g g^{-1})	10-min stability period			No stability period		
			u_{stop} (g g^{-1})	$\hat{u}_{eq}$ (g g^{-1})	Prediction error (g g^{-1})	u_{stop} (g g^{-1})	$\hat{u}_{eq}$ (g g^{-1})	Prediction error (g g^{-1})
Holocellulose	20–30%	0.0674	0.0648	0.0690	0.0016	0.0646	0.0692	0.0018
	50–60%	0.1120	0.1084	0.1119	–0.0001	0.1067	0.1105	–0.0015
	70–80%	0.1628	0.1596	0.1645	0.0017	0.1593	0.1644	0.0016
Loblolly pine	20–30%	0.0567	0.0541	0.0588	0.0021	0.0539	0.0590	0.0023
	50–60%	0.0898	0.0867	0.0903	0.0005	0.0863	0.0903	0.0005
	70–80%	0.1338	0.1254	0.1291	–0.0047	0.1243	0.1282	–0.0056
	90–95%	0.2260	0.2182	0.2275	0.0015	0.2174	0.2270	0.0010
MCC	20–30%	0.0419	0.0407	0.0461	0.0042	0.0406	0.0463	0.0044
Office paper	20–25%	0.0377	0.0365	0.0421	0.0044	0.0364	0.0424	0.0047
	70–75%	0.0916	0.0887	0.0923	0.0007	0.0886	0.0925	0.0009

tested by Glass et al. [5]. Further work is needed where data are collected to operational equilibrium, to see how well the correction in Eq. (7) works on other wood species or materials.

4 Conclusions

This paper examined the systematic errors in the equilibrium moisture content prediction from the commonly used S_{10}^{20} stop criterion with automated sorption balances (relative mass change less than 20 µg g^{-1} min^{-1} calculated with a 10-minute interval). Seventy-two data sets from twenty-one laboratories were analyzed on absorption steps across the 10–95% RH range on samples from western larch with hold times of 7–10 days. EMC was defined based on the operational mass stability determined in the first part of this interlaboratory study. On average, the S_{10}^{20} criterion was met before the fractional change in moisture content from the initial state to the equilibrium state had reached 0.8 (i.e., the sorption process was less than 80% complete). The resulting absolute error in the EMC prediction was as

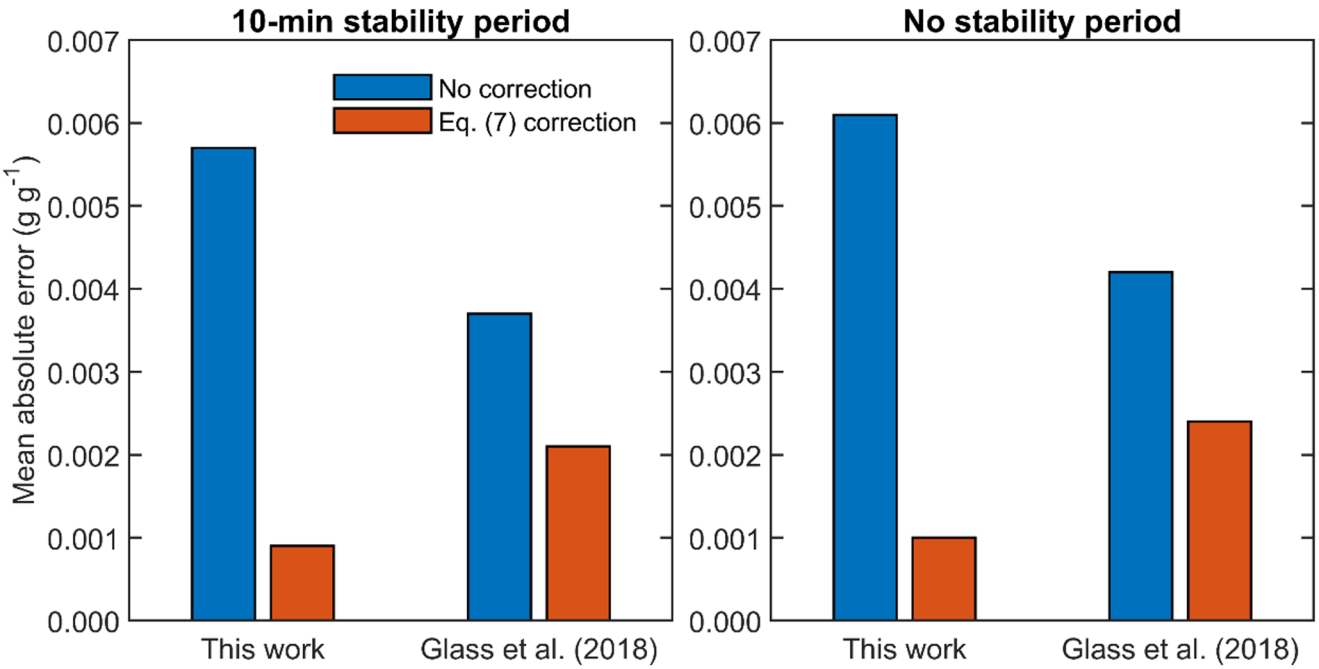

Fig. 11 Mean absolute error in the EMC prediction with the S_{10}^{20} stop criterion for western larch and other materials with and without the Eq. (7) correction applied

high as 0.0136 g g^{-1} with a mean absolute error of 0.0057 g g^{-1} or 0.0061 g g^{-1} depending on whether the S_{10}^{20} criterion utilized a 10-minute stability period.

The systematic underprediction of EMC exhibited a parabolic dependence on moisture content. An equation was developed to correct the systematic underprediction and estimate EMC from the moisture content corresponding to the S_{10}^{20} criterion. This correction equation reduced the mean absolute error to less than or equal to 0.0010 g g^{-1} for the western larch data.

The applicability of the correction equation was tested against sorption data for other materials from the literature, where extended hold times allowed for determining EMC using the same operational definition. The correction equation developed for western larch reduced the mean absolute error in the EMC prediction for these materials as well but not to the same extent. Further measurements for other wood species and materials are recommended.

While the correction equation can reduce the systematic error in measurements stopped at the S_{10}^{20} criterion, it should be emphasized that this stop criterion is far from equilibrium. Extending the measurement time may be necessary to estimate EMC more accurately. Further analysis of alternative stop criteria is recommended. Development of scientifically informed and robust stop criteria could lead to more standardization of stop criteria used within the scientific community and allow better comparison between studies.

On the other hand, for applications where minimizing measurement time is a primary concern and the S_{10}^{20} criterion is used, the correction equation presented in this paper can greatly improve the prediction of equilibrium moisture content in absorption.

Appendix

See Tables 4, 5, 6.

Table 4 Models for correcting absolute error in equilibrium moisture content

Model	Equation
A: linear, zero intercept	$y = k_1 u_{stop}$
B: linear, variable intercept	$y = k_0 + k_1 u_{stop}$
C: parabolic	$y = k_0 + k_1 u_{stop} + k_2 {u_{stop}}^2$
D: 3rd degree polynomial	$y = k_0 + k_1 u_{stop} + k_2 {u_{stop}}^2 + k_3 {u_{stop}}^3$
E: 4th degree polynomial	$y = k_0 + k_1 u_{stop} + k_2 {u_{stop}}^2 + k_3 {u_{stop}}^3 + k_4 {u_{stop}}^4$
F: linear + exponential	$y = k_0 + k_1 u_{stop} + k_2 e^{-k_3 u_{stop}}$
G: linear + hyperbolic	$y = k_0 + k_1 u_{stop} + \frac{k_2}{u_{stop}+k_3}$

y is absolute error; u_{stop} is moisture content at stop criterion; k_i are empirical constants

Table 5 Results for models listed in Table 4

Model	k_0	k_1	k_2	k_3	k_4	RMSE (g g^{-1})	R^2
10-min stability period							
A	—	0.0460	—	—	—	0.0031	<0.1
B	0.0038	0.0186	—	—	—	0.0022	0.267
C	0.0084	-0.0926	0.4448	—	—	0.0012	0.774
D	0.0089	-0.1135	0.6483	-0.5388	—	0.0012	0.776
E	0.0105	-0.2032	2.109	-9.335	17.42	0.0012	0.783
F	-0.0709	0.2258	0.0799	4.346	—	0.0012	0.777
G	-0.1456	0.2504	0.0622	0.4011	—	0.0012	0.776
No stability period							
A	—	0.0460	—	—	—	0.0033	<0.1
B	0.0043	0.0175	—	—	—	0.0023	0.220
C	0.0089	-0.0970	0.4593	—	—	0.0014	0.730
D	0.0097	-0.1292	0.7741	-0.8360	—	0.0013	0.735
E	0.0118	-0.2465	2.699	-12.49	23.15	0.0013	0.747
F	-0.0358	0.1591	0.0458	6.652	—	0.0013	0.737
G	-0.1459	0.2541	0.0611	0.3920	—	0.0013	0.737

stop criterion: relative mass change≤20 μg g^{-1} min^{-1}, 10-min slope (S_{10}^{20}), with specified stability period; k_i are empirical constants; RMSE is root mean square error; R^2 is coefficient of determination

Table 6 Summary of 72 "acceptable" datasets on water vapor sorption in Western larch (moisture content uncertainty less than 0.0005 g g^{-1})

Laboratory	RH step	Moisture content (g g^{-1})			
		Initial	Equilibrium	Stop criterion 1[a]	Stop criterion 2[b]
A	20–30%	0.0293	0.0443	0.0396	0.0398
B	10–20%	0.0158	0.0386	0.0285	0.0301
	10–20%	0.0152	0.0396	0.0277	0.0296
	10–20%	0.0213	0.0421	0.0378	0.0381
	60–70%	0.0929	0.1162	0.1123	0.1127
	60–70%	0.0958	0.1231	0.1183	0.1185
C	30–40%	0.0525	0.0710	0.0675	0.0677
D	70–80%	0.1296	0.1512	0.1455	0.1457
E	50–60%	0.0823	0.1011	0.0982	0.0984
F	20–30%	0.0404	0.0550	0.0508	0.0512
	20–30%	0.0386	0.0539	0.0493	0.0495
	50–60%	0.0842	0.1055	0.1018	0.1020
	60–70%	0.0974	0.1227	0.1191	0.1197
	60–70%	0.0997	0.1252	0.1211	0.1220
G	0–10%	0.0000	0.0275	0.0187	0.0197
	0–10%	0.0000	0.0266	0.0183	0.0193
	40–50%	0.0662	0.0854	0.0816	0.0824
	40–50%	0.0676	0.0858	0.0819	0.0827
H	10–20%	0.0214	0.0442	0.0376	0.0384
	10–20%	0.0212	0.0410	0.0367	0.0369
	90–95%	0.1909	0.2375	0.2265	0.2267
I	70–80%	0.1065	0.1384	0.1338	0.1342
	70–80%	0.1068	0.1408	0.1343	0.1347
K	60–70%	0.0903	0.1124	0.1067	0.1069
L	30–40%	0.0416	0.0602	0.0540	0.0544
	40–50%	0.0651	0.0820	0.0782	0.0784
M	30–40%	0.0459	0.0663	0.0618	0.0619
	30–40%	0.0468	0.0631	0.0590	0.0592
	80–90%	0.1394	0.1983	0.1898	0.1903
N	0–10%	0.0000	0.0263	0.0188	0.0192
	10–20%	0.0159	0.0423	0.0315	0.0320
	20–30%	0.0333	0.0554	0.0497	0.0499
	20–30%	0.0372	0.0562	0.0522	0.0524
	30–40%	0.0509	0.0697	0.0658	0.0660
	30–40%	0.0503	0.0691	0.0652	0.0654
	40–50%	0.0652	0.0845	0.0804	0.0806
	40–50%	0.0633	0.0812	0.0772	0.0774
	50–60%	0.0799	0.0995	0.0962	0.0963
	60–70%	0.0944	0.1177	0.1135	0.1137
	70–80%	0.1144	0.1473	0.1421	0.1423
	80–90%	0.1411	0.1989	0.1910	0.1913
	80–90%	0.1405	0.1928	0.1859	0.1861
	90–95%	0.1884	0.2459	0.2342	0.2347
	90–95%	0.1895	0.2438	0.2333	0.2336
	90–95%	0.1894	0.2480	0.2362	0.2364
	90–95%	0.1884	0.2449	0.2343	0.2345
	90–95%	0.1895	0.2446	0.2327	0.2328
	90–95%	0.1826	0.2310	0.2220	0.2221
O	10–20%	0.0177	0.0401	0.0338	0.0342
	10–20%	0.0202	0.0410	0.0363	0.0365
	10–20%	0.0191	0.0409	0.0343	0.0345
	10–20%	0.0223	0.0442	0.0386	0.0388

Table 6 (continued)

Laboratory	RH step	Moisture content (g g^{-1})			
		Initial	Equilibrium	Stop criterion 1[a]	Stop criterion 2[b]
P	10–20%	0.0216	0.0386	0.0338	0.0341
	10–20%	0.0223	0.0421	0.0372	0.0374
	10–20%	0.0204	0.0382	0.0320	0.0322
	10–20%	0.0223	0.0398	0.0353	0.0355
	10–20%	0.0204	0.0378	0.0315	0.0317
	80–90%	0.1391	0.1875	0.1826	0.1829
	80–90%	0.1387	0.1903	0.1847	0.1849
	80–90%	0.1400	0.1904	0.1846	0.1847
	80–90%	0.1419	0.1926	0.1864	0.1865
	80–90%	0.1390	0.1891	0.1826	0.1828
Q	0–10%	0.0000	0.0212	0.0161	0.0163
	70–80%	0.1059	0.1434	0.1373	0.1375
	70–80%	0.1082	0.1458	0.1415	0.1417
R	50–60%	0.0746	0.0901	0.0877	0.0879
S	0–10%	0.0000	0.0249	0.0195	0.0197
T	40–50%	0.0647	0.0845	0.0810	0.0812
	40–50%	0.0660	0.0852	0.0823	0.0825
U	20–30%	0.0303	0.0509	0.0453	0.0455
	90–95%	0.1869	0.2380	0.2290	0.2292
	90–95%	0.1873	0.2530	0.2394	0.2396

[a] Stop criterion 1: relative mass change≤20 µg g^{-1} min^{-1}, 10-min slope (S_{10}^{20}), no stability period

[b] Stop criterion 2: relative mass change≤20 µg g^{-1} min^{-1}, 10-min slope (S_{10}^{20}), 10-min stability period

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Author contributions Conceptualization: SLZ, SVG, EET, Investigation: All authors, Data Curation: SLZ, SVG, EET, NF, Formal Analysis: SLZ, SVG, EET, NF, Visualization: SLZ, EET, SVG, NF, Writing - Original Draft: SLZ, SVG, EET, NF Writing - Review & Editing: All authors, Project administration: SLZ, SVG, EET, NF.

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Data availability Raw data were submitted to the US Forest Service Data Archive. Data can be found at the following URL [https://www.fs.usda.gov/rds/archive/catalog/RDS-2025-0010].

Declarations

Competing interests Several coauthors work for companies that sell automated sorption balances. All data have been anonymized to prevent a comparison across instrument manufacturer or model to address potential conflicts due to commercial interests.

Ethical approval This study did not involve human or animal participants.

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