APPARATUS FOR GRAVIMETRIC MEASUREMENT OF MOISTURE SORPTION ISOTHERMS FOR 1-100 G SAMPLES IN PARALLEL[1]

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Abstract. This article presents a new apparatus for collecting water vapor sorption isotherms in parallel with example data from acetylated and thermally modified loblolly pine. The experimental setup consists of an enclosure that is continuously flushed with a carrier gas of known RH supplied by an RH generator and a balance with precision of 0.1 mg. All samples are placed on the balance and the mass is monitored as a function of time. At a given RH, the measurements are stopped when the change in the total mass of all samples are less than or equal to a change in MC of 0.1 μg g^{-1} min^{-1} (0.000 01%) per minute over a 24 h window. The sorption isotherms effectively differentiated the wood modifications; isotherm models fit the

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data extremely well ($R^2 \geq 0.95$ for all treatments). A comparison of the data with measurements on the same specimens conditioned over saturated salt solutions shows that the method is comparable with isotherms acquired over saturated salts. When compared with the saturated salt solution method for collecting isotherms, this method requires less labor. Furthermore, unlike traditional dynamic vapor sorption methods, this apparatus easily allows multiple macroscopic samples to be run in parallel.

Keywords: Wood-moisture relations, dynamic vapor sorption, water vapor sorption, isotherm.

INTRODUCTION

Many of the important characteristics of wood as a building material such as strength, stiffness, thermal conductivity, and even the dimensions depend upon moisture. Because moisture is such a fundamental variable in describing the properties of wood, sorption isotherms are a key way to characterize and understand the behavior of wood (Simpson 1980; Glass et al 2014).

Measuring sorption isotherms is not trivial. The measurements require keeping the RH and temperature constant over a long period of time until the changes in mass are so small that it is assumed that equilibrium has been reached. Traditionally, sorption isotherms have been collected by conditioning samples to various RH using saturated salt solutions, glycerol–water solutions, or sulfuric acid solutions to fix the RH and this method has been standardized (Greenspan 1977; Anon 2016). In a saturated salt solution, the water vapor pressure above the solution is fixed by the Gibbs phase rule and depends only on temperature. To measure the isotherms, jars with saturated salt solutions in the bottom are made and the samples, suspended above the solution, are periodically removed and weighed. These measurements are very time consuming and labor intensive as they require repeated weighing by the operator. The measurements can also be prone to error if the samples touch the container which often contains salt residue or condensation and affects the mass measurements. When this happens, the sample needs to be discarded from the measurement series. Furthermore, the RH is temperature dependent and can fluctuate as the room temperature changes if the room temperature is not carefully controlled.

Previously, Goulet developed systems to overcome some of these limitations of saturated salt solutions (Goulet 1968; Goulet and Hernández 2007). In this system, the RH was kept constant by placing the desiccators in insulated water baths whose temperature was controlled to within 0.5°C by the use of rotors whose mechanical work heated the water. Furthermore, the samples could be weighed without removing them from the desiccators; the samples were placed on a platform within the desiccator that could be attached to a balance through a small port in the top of the desiccator.

Recently, automated sorption balances, frequently also referred to as dynamic vapor sorption (DVS) measurements have become a popular tool for characterizing the sorption isotherms of wood and other materials (Mangiagli et al 2009; Hill et al 2010a, 2010b; Jalaludin et al 2010; Xie et al 2010; Ceylan et al 2012; Driemeier et al 2012; Volkova et al 2012; Keating et al 2013; Popescu et al 2013). In such equipment, one small ($\leq$100 mg) sample is suspended from a microbalance inside a chamber with a large thermal mass. The RH is controlled by constantly flowing carrier gas (either nitrogen or compressed air) whose water vapor pressure is fixed by mixing streams of dry and saturated gas with mass flow controllers. This technique allows continuous measurement of the mass as a function of time. At each RH step, the "equilibrium moisture content" is determined by reading the mass of the sample when the change in mass as a function of time is below a certain threshold; a 0.002% change in MC per minute (20 μg g^{-1} min^{-1}) over a 10 min window has been the most frequently used mass stability criterion (Hill et al 2009) (In literature, the change in moisture content with respect to time has been traditionally reported in percent change per minute (ie 0.002% per minute). However, because of the small relative changes in mass we examine in this article, we present these in μg g^{-1} min^{-1}, which is unambiguous in what the percentage is and removes most of the leading zeros. For reference, the commonly cited 0.002% per minute becomes 20 μg g^{-1} min^{-1}.

With the 20 μg g^{-1} min^{-1} criterion, water vapor sorption isotherms could generally be collected in under a week (Kohler et al 2006; Hill et al 2009; Engelund et al 2010; Hill et al 2010a; Jalaludin et al 2010; Xie et al 2011a, 2011b; Volkova et al 2012). This represents major time savings over measurements with saturated salt solution which typically take between 6 and 24 mo to complete a full absorption and desorption cycle. This is tempered, somewhat, by the fact that most automated sorption balances can only collect information on a single sample at one time, whereas multiple samples are typically run in parallel over saturated salt solutions.

It has recently been pointed out that the 20 μg g^{-1} min^{-1} criterion mischaracterizes the EMC of cellulosic materials and leads to errors larger than 1% MC (Glass et al 2018). Glass et al (2017) performed sorption measurements with very long hold times (up to 50 h per RH step) and noticed that the predicted MC was off by as much as 2.5% MC (an 18% error in the measurement) when the 20 μg g^{-1} min^{-1} criterion was used. Follow-up work by Glass et al (2018) showed that measured errors only decrease logarithmically with increasing hold time and suggested a new stop criteria of 3 μg g^{-1} min^{-1}. With this new suggested criteria, automated sorption balance measurements will take longer, and with most commercial instruments, still need to be collected in series.

It is desirable to combine some of the aspects of traditional, saturated salt measurements with automated sorption balance measurements. Strømdahl (2000) designed and built an impressive sorption analyzer for macroscopic samples that controlled five environmental chambers in parallel. Hedlin (1968) also developed a system where multiple samples could be placed in a subfreezing environmental chamber and weighed without removing them. Both of these previous measurements combine some of the advantages of traditional measurements (parallel processing, macroscopic samples) with some of the advantages of automated sorption balances (stable RH control, allows automation). Here, we present another approach that combines aspects of both types of measurements built from off the shelf components and demonstrate its use by presenting isotherms of untreated and modified loblolly pine (*Pinus taeda*). The objective of this study was to develop a system that allows for parallel conditioning of macroscopic samples with stable RH and temperature control and does not require manual measurements to determine equilibrium.

MATERIALS AND METHODS

Materials

Isotherms were collected on untreated, acetylated, and thermally modified loblolly pine (*Pinus taeda*), from the southern pine group. All samples were cut from a single board that was obtained in a kiln-dried state from a sawmill in Shaqualak, MS. Sample masses ranged from 1.5 to 3.2 g and sample geometry also varied between samples; the total oven-dry mass of all samples was 15.4591 g (seven treatments with one replicate each). Immediately before starting the absorption isotherm, the samples were oven-dried at 105°C for 48 h.

To produce the acetylated samples, small sections of the board were vacuum-dried at 70°C, weighed, acetylated by submerging them in a solution of acetic anhydride with 4% pyridine, and placed in an oven at 140°C (Rowell et al 1986). Samples were removed at various time points to achieve different weight percent gains (WPG) of acetylation. After removal, the samples were allowed to react in deionized water overnight and vacuum-dried. WPG was calculated as

$$\mathrm{WPG} = \frac{w_{\mathrm{vd}}^{\mathrm{post}} - w_{\mathrm{vd}}^{\mathrm{pre}}}{w_{\mathrm{vd}}^{\mathrm{pre}}} \times 100, \qquad (1)$$

where the subscript “vd” represents vacuum-dried and the superscript “pre” and “post” represent the mass before and after the reaction, respectively. Four different WPGs were tested: 8%, 11%, 13%, and 20%. Two thermal modifications, based off the Thermowood™ process, were also tested (Hill 2006). To produce the heat-treated samples, small sections of the board mentioned previously were first vacuum-dried for 4 h at 60°C and then heated in an oven at 200°C for either 1 or 2 h, after which the oven was switched off; the samples reached a temperature of 50°C 2 h after the oven was turned off.

An apparatus for measuring multiple sorption isotherms in parallel was assembled by combining commercially available parts and equipment (Fig 1). The system consisted of an enclosure, a balance that read to 0.1 mg, an RH generator, and a computer.

The enclosure was fabricated from a plastic sandblasting box. Because the measurements were run at 23°C (ambient laboratory conditions), no effort was made to minimize temperature differentials between the box and the laboratory; however, polystyrene insulation could easily be added to the outside for future experiments. Gasketed holes were added to allow wires, sensors, and a water vapor inlet hose to enter the chamber. The total interior volume of the enclosure was 86.7 L. The top of the chamber was held down with clasps. The chamber had two openings where hands could be inserted. These openings were sealed with overlapping sheets of neoprene and further sealed by using 38 mm plugs of polymeric insulation when no access was required to the chamber. To manipulate samples, the plug was removed, and a hand could be inserted between the neoprene sheets.

The humidity inside the chamber was controlled with an RH generator (Model: HumiSys HF2-1-2-220; InstruQuest, Coconut Creek, FL) and in-house compressed air was used as carrier gas. The humidity generator was run in a feedback control mode (Mode 5). The RH sensor from the RH generator was used to both monitor the RH within the chamber and control the generator. The RH sensor was checked by placing a NIST traceable RH probe with a tolerance of ±1.5% RH and running the system through six different relative humidities (20%, 40%, 50%, 70%, 80%, and 90%). The difference between the two RH readings were within, at most, 1.7% RH (data not shown).

The humidity generator provided a constant flow of 2 L min^{-1} into the chamber. Because there was no deliberate exit path for the air, the system was at positive pressure relative to the room and air exited through leaks in the enclosure to maintain the flow rate. Given the volume of the chamber, the flow rate corresponds with 1.4 air changes per hour. During the experiments, it took approximately 3 h to fully stabilize at the RH target. The temperature of the carrier gas was 24°C. The isotherms were first measured in absorption to 95% RH and then desorption measurements were taken immediately following absorption in the following steps: 10%, 20%, … 90%, 95%, 90%, 80%, … 10%.

The balance was located at the opposite end of the chamber from the gas inlet. The balance door that faced the gas inlet was kept closed to prevent a direct flow of carrier gas influencing the mass

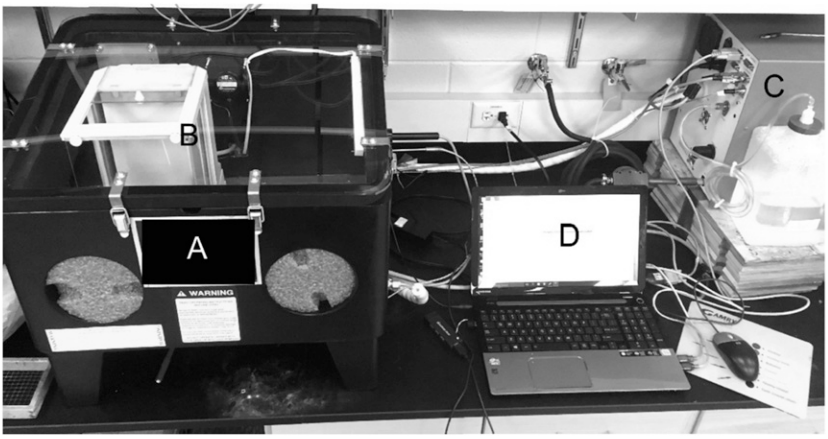

Figure 1. Photograph of the experimental setup including the enclosure (a), balance (b), humidity generator (c), and computer (d) for control and data acquisition.

measurement; the other doors on the balance remained open to allow gas mixing. The mass was outputted to a computer, typically at 60-s intervals. In this experiment, seven samples were run concurrently (one replicate for each treatment).

For each RH condition, the drift in the balance was measured by placing a stainless steel cylinder (mass approximately 6 g) on the balance after each RH step and measuring its mass for 24 h. This drift depended on RH and was higher at low RH. The maximum drift in the balance was 1.6 mg over a 24 h period at 10% RH. With the total dry mass of all samples equal to 15.4591 g, this corresponds with a dM/dt on the sample MC of 0.07 μg g^{-1} min^{-1} over a 24 h window. Above 20% RH, the balance was much more stable and did not exhibit a linear trend with time.

In typical automated sorption balance measurements, a mass stability criterion is used to determine when to stop collecting data at a given RH and move to the next RH. The most widely used criterion, promoted by Hill et al (2009, 2010c) was 20 μg g^{-1} min^{-1} over 10 min. However, it has recently been noted that this criterion mischaracterizes the EMC (Glass et al 2017; Glass et al 2018). In the current experiment, the mass of all seven samples was monitored because they were all on the balance at the same time. The mass was monitored until the change in the overall MC (as calculated from the readout on the balance and the sum of the individual oven-dry masses) was less than 0.1 μg g^{-1} min^{-1} over a 24 h period. In this experiment, that was equivalent to a change of less than or equal to 2.5 mg over a 24 h period. For comparison with other macroscopic measurements, ASTM C1498 suggests maintaining a mass change of less than 0.1% over 5 d; this is equivalent to 0.14 μg g^{-1} min^{-1}. It should be noted that at the beginning of the experiment (10% RH, 20% RH in absorption), a less stringent criterion of 0.3 μg g^{-1} min^{-1} over a 24-h period was used.

The total mass of all samples was used to determine when equilibrium was achieved. Once the stability criteria was reached, each individual sample was weighed manually, in situ. These equilibrium weights were used to determine the water vapor sorption isotherms.

RESULTS

Figure 2 presents a representative output from the balance as a function of time for two RH steps, 60-50% and 90-80% RH, in desorption. In Fig 2(b) and 2(c), the corresponding measured RH

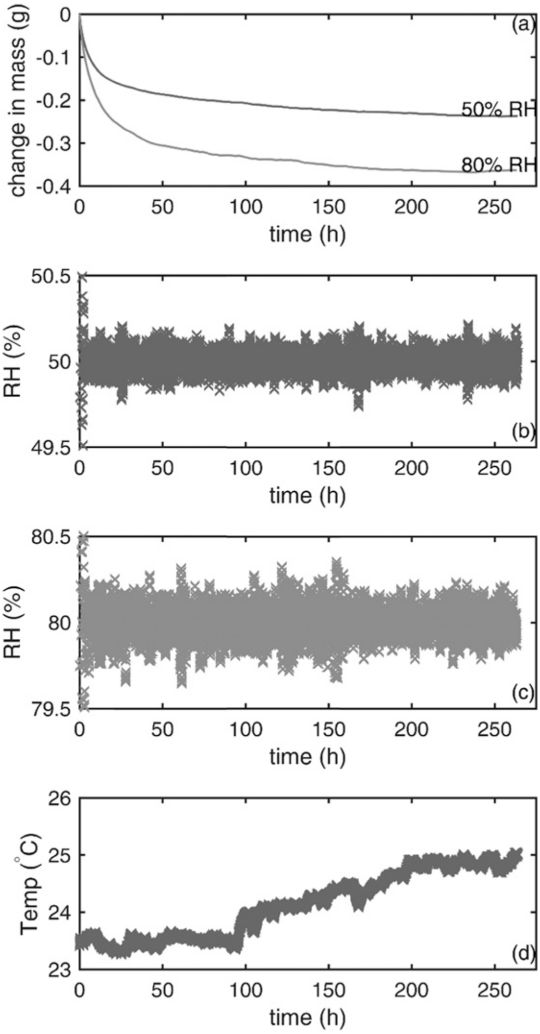

Figure 2. Example of representative change in mass as a function of time for the 60-50% RH and 90–80% RH steps. (b and c) Measured RH within the chamber during the desorption steps and (d) measured temperature within the chamber during the 50% RH desorption step.

within the chamber is presented. The RH within the chamber was extremely stable after reaching equilibrium in the chamber; after 3 h, the maximum deviation from the set point was 0.25% RH for the 50% RH condition and 0.35% for the 80% RH condition. During the first 3 h, the RH fluctuated around the set point and briefly overshot the target RH by as much as 10%. For the 50% RH condition, the samples were weighed when the total mass change of the sample was 0.3 mg over a 24-h period. Because the oven-dry mass of all of the samples was 15.4591 g, this represented a dM/dt of 0.1 μg g^{-1} min^{-1} over a 24-h period. For the 80% RH condition, the mass actually slightly increased at the end of the experiment, resulting in a minimum mass change of 0.0 mg over a 24-h period.

The absorption isotherm was started on December 13, 2016, and completed (at 95% RH) on April 3, 2017, a duration of 111 d. The (scanning) desorption isotherm immediately followed and July 25, 2017, a duration of 113 d; the total duration of the entire experiment was 224 d. Figure 3(a)-3(e) presents the isotherm for the different levels of acetylation; Fig 3(f) presents all absorption isotherms on the same graph to show differences between treatments. The ratio between the untreated and 20 WPG acetylated specimens at 95% RH was 2.7. The acetylation affected the MC in a similar manner to previously

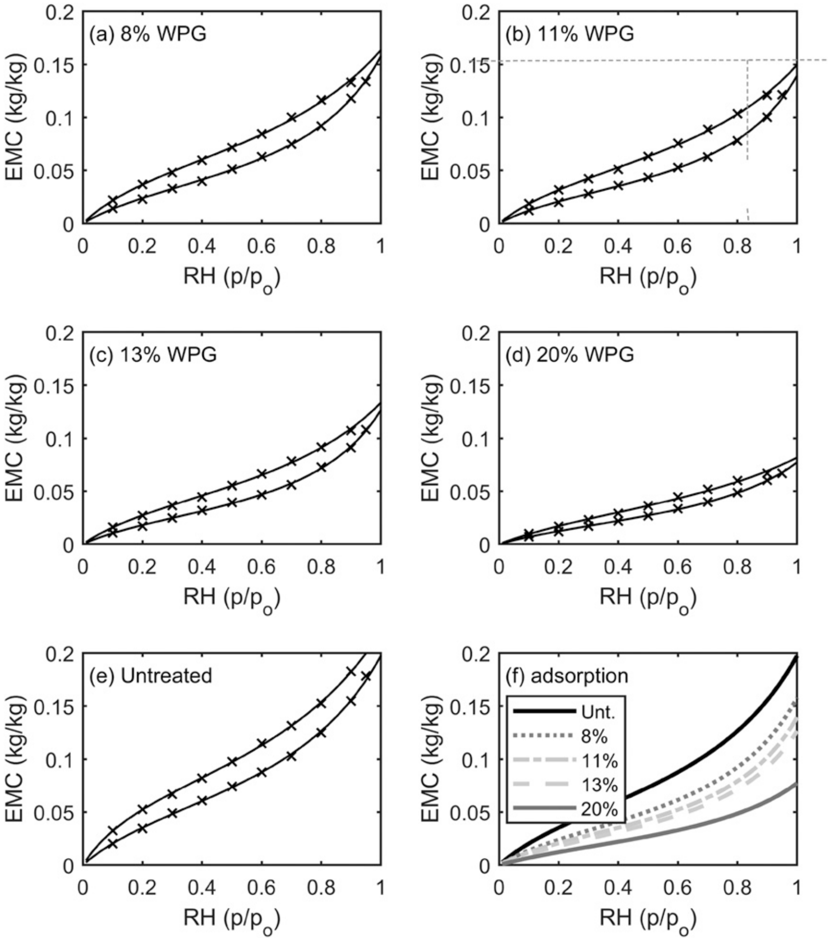

Figure 3. (a-e) Sorption isotherms for the acetylated and untreated samples. (f) Comparison of the absorption isotherms.

Table 1. Fit parameters (Eq 1) for interpolating absorption and desorption isotherms.

		Absorption			Desorption		
		A	B	C	A	B	C
	Untreated	−9.19	10.25	4.00	−7.01	9.29	2.25
Acetylated	8%	−15.03	15.46	5.91	−8.40	10.89	3.62
	11%	−19.20	19.88	6.48	−9.69	12.06	4.31
	13%	−20.34	20.34	7.90	−11.00	13.37	5.11
	20%	−22.54	22.78	12.70	−12.13	15.15	9.19
Heat treatment	1 h	−11.27	12.42	3.79	−7.59	10.10	2.01
	2 h	−9.75	9.38	6.18	−6.64	8.97	3.08

reported data where the MC of the untreated sample was between two and three times that of the maximally acetylated sample at 95% RH (Thygesen et al 2010; Popescu et al 2013; Himmel and Mai 2015).

Isotherms were fit with the parabolic model

$$\frac{h}{m} = \mathrm{A}h^2 + \mathrm{B}h + \mathrm{C}, \qquad (2)$$

where h is the fractional RH; m is the EMC (g/g); and A, B, and C are the fitting parameters with no physical significance (Zelinka and Glass

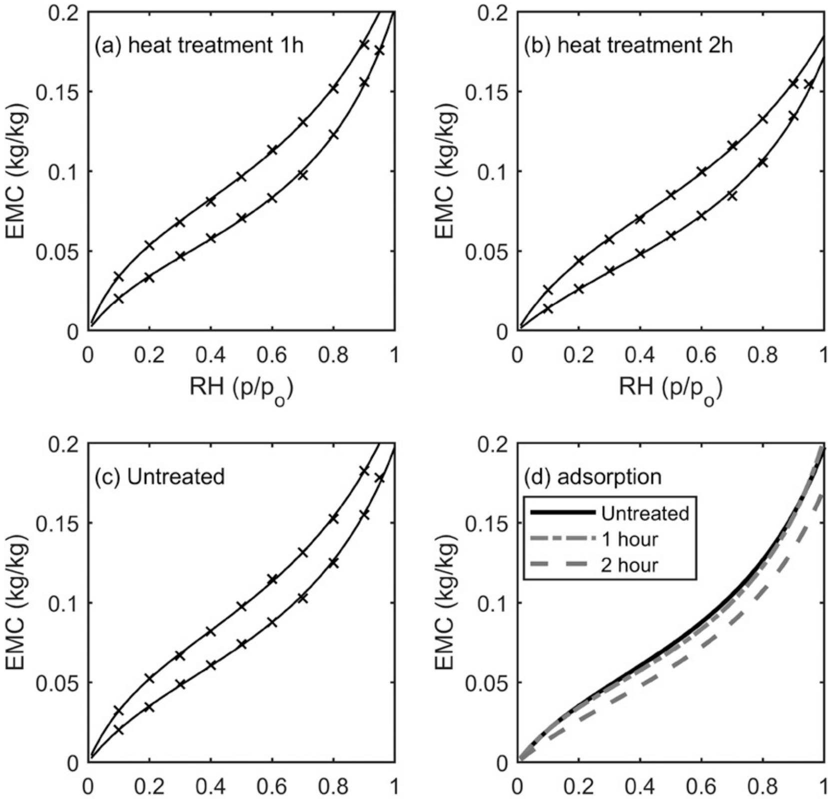

Figure 4. (a-c) Water vapor sorption isotherms for the heat-treated and untreated specimens. (d) Absorption isotherms from (a-c) overlaid.

2010). The model is mathematically equivalent to the Hailwood–Horrobin sorption isotherm (Hailwood and Horrobin 1946). Dent sorption isotherm (Dent 1977), and the Guggenheim–Anderson–DeBoer sorption isotherm (Anderson 1946), and is useful for interpolation but does not ascribe physical significance to the model fit parameters. The model fit parameters are given in Table 1 and can be used to determine the EMC of acetylated southern pine at any given RH at room temperature (23°C).

Figure 4 presents the isotherms collected for the heat-treated wood along with the parabolic model fit to the data. The curve fit parameters for the heat-treated wood are also included in Table 1. The difference between the 1-h heat treatment and the untreated wood was negligible.

DISCUSSION

This article presents a method for collecting multiple sorption isotherms in parallel using a sorption balance DVS technique. One advantage of this method over traditional isotherm techniques is that it does not require saturated salt solutions to reduce labor, and the temperature and RH can be held within very tight tolerances over the experiment. Although these advantages are similar to commercial automated sorption balance measurements, using our system gives much more flexibility on the sizes and number of samples as compared with the commercial systems which are limited to small (<100 mg) sample masses.

Although the current method has some advantages in collecting isotherms, it is worthwhile to examine its accuracy as compared with other isotherm methods. For the 8% WPG acetylated sample, a sorption isotherm was also collected over saturated salt solutions. In these measurements, the specimen was allowed to equilibrate for at least 1 mo at each RH condition and then weighed; this was more than twice the time the sample took to reach equilibrium in the parallel sorption balance (Fig 1). The saturated salt data are compared with the parallel sorption balance data in Fig 5. It can be seen that the sorption isotherms for both methods fall along a single curve. When fit with Eq 2 the combined data set had an R^2 of 0.96 and a mean (absolute) residual of 0.2% MC.

The method presented in this article seems to have good agreement with traditional, saturated salt sorption isotherms. Several researchers have compared automated sorption balance and saturated salt measurements. Although some researchers have found no differences between the methods, others have found that the method does

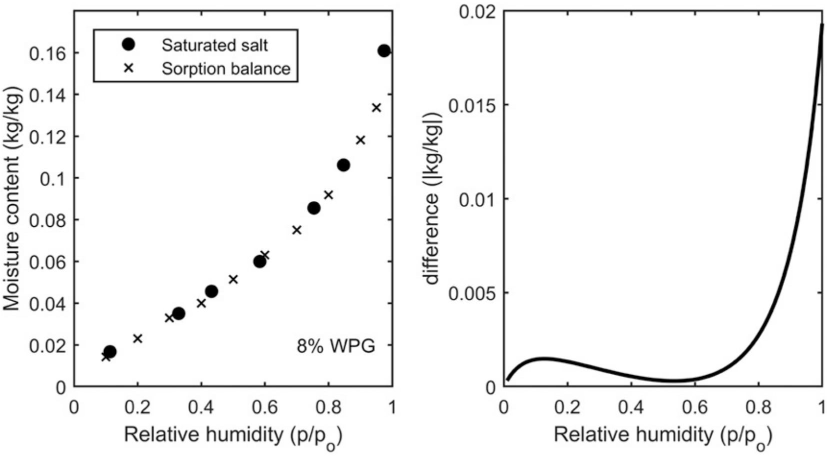

Figure 5. Comparison of the water vapor sorption isotherm of the 8% WPG sample collected with the saturated salt solution method (●) and with the parallel DVS apparatus presented in this article (×).

affect their results (Arlabosse et al 2003; Peuhkuri et al 2005; Simón et al 2017). It appears that the method presented in this article gives similar results to the saturated salt solutions. Further direct comparisons between the saturated salt method, automated sorption balance measurements, and the current method could yield further insights into sorption techniques.

CONCLUSIONS

An apparatus was presented for collecting multiple sorption isotherms in parallel in a similar manner to automated sorption balances DVS using entirely off-the-shelf laboratory parts. A comparison of sorption isotherms collected on the same sample using this method is in good agreement with data collected using saturated salt solutions. One major advantage of this apparatus over most automated sorption balance systems is that it allows for multiple samples of arbitrary sizes to be run in parallel. Furthermore, such a system can be assembled using commercially available laboratory equipment for the fraction of the cost of a commercial automated sorption balance.

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