EQUILIBRIUM MOISTURE CONTENT UNDER VACUUM CONDITIONS

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Abstract. The equilibrium moisture content (EMC) of three species was measured under vacuum conditions. Temperature, RH, and ambient pressure in a chamber were controlled during the experiments to obtain accurate EMC measurement under vacuum. Based on the experimental results and on the Hailwood–Horrobin model for EMC, the desorption isotherms of wood under vacuum were analyzed. EMC charts and a database under vacuum conditions were also built. Results showed that the desorption isotherms of wood under vacuum conditions also presented a typical sigmoid shape similar to the one at atmospheric conditions. The effect of ambient pressure on EMC was small at high RH ranges and became obvious with decreasing RH. Also, the EMC of ambient pressure from 53.3 to 101.3 kPa was not obvious because the difference in EMC was only 0.1-0.4%. Conversely, the effect of pressure became greater from 53.3 to 13.3 kPa and the difference in EMC was 1.2-1.9%. EMC corresponding to temperature, RH, and

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ambient pressure at vacuum conditions was built with the chart and equations based on experimental results from the real-time MC measurement for vacuum drying and serves as an aid in wood research and drying control under vacuum conditions.

Keywords: Database building, desorption isotherm, equilibrium moisture content (EMC), vacuum condition.

INTRODUCTION

Wood drying is an essential step in the manufacture of wood products. However, conventional kiln drying is a time-consuming and energyintensive process. Compared with traditional drying methods, wood vacuum drying, especially radio-frequency/vacuum (RF/V) and microwave/ vacuum (M/V) drying, has many advanced features such as a significantly shortened drying time, a decreased risk of discoloration, and good energy efficiency (Sasaki et al 1987; Lopatin et al 2014). During the past few decades, the use of vacuum drying has been steadily increasing (especially for drying of valuable species) and is likely to continue increasing as a rapid drying method (Simpson 1987). As with conventional drying, it is important to know the parameters under vacuum condition such as temperature, pressure, RH, and MC of wood during vacuum drying to program appropriate schedules, control costs and quality, as well as to investigate vacuum drying mechanisms.

EMC is used for research both in wood science and wood manufacturing. Much research on EMC under atmospheric pressure has been performed (Stamm 1964; Kollmann and Cote 1968; FPL 1999). EMC data presented in unified description by FPL (1999) serves well in wood conventional drying. With the development of wood vacuum drying, there has recently been some research on wood EMC under vacuum conditions (Chen and Lamb 2002; Yi et al 2008; Chen et al 2009; Xiao and Cai 2009; Liu et al 2010a, b; Zhou et al 2013; Yang et al 2014). Chen and Lamb (2002) found that with vacuum drying, EMC still needs to be controlled to perform the drying run. They also provided a theoretical EMC estimation. Based on the assumption that there is no air in the chamber during vacuum drying, Yi et al (2008) obtained experimental results. Liu et al (2010a) and Cai

and Hayashi (2007) proposed a new method for real-time MC measurement under vacuum conditions using the relationships among temperature, pressure in wood, and EMC under vacuum conditions. The research was a significant attempt to apply EMC to vacuum drying.

MC of wood in equilibrium under constant temperature and RH is termed EMC. Although temperature and RH are the principal factors that determine EMC, it is also affected by the sorption process, mechanical stress, species, and extractive content (Skaar 1988). The theoretical EMC of Chen and Lamb (2002) and the experimental EMC of Yi et al (2008) under vacuum conditions showed obvious differences from the EMC of FPL (1999) as well as each other. Consequently, it appears that other factors might affect EMC under vacuum conditions. In Liu et al (2010a, b), an ambient pressure factor was introduced to investigate the impact on EMC. The results showed that ambient pressure affects EMC of wood under vacuum. EMC increased with decreasing ambient pressure (Liu et al 2010a, b). In addition, in their studies of real-time MC measurement, the precision of MC estimation using EMC was improved after the EMC modification. Although the experiments of Chen et al (2009) and Yi et al (2008) were based on the assumption that there was no air in the chamber during vacuum drying, some air does remain inside the chamber. Consequently, total pressure differs from partial vapor pressure. Also, the condition in the vacuum chamber varies according to ambient temperature and RH in the chamber. The definition of RH under vacuum conditions is the ratio of partial vapor pressure to saturated pressure for a given temperature (Siau 1995). Meanwhile, Xiao and Cai (2009) verified that RH was affected by ambient pressure. Effects on RH by ambient pressure can also be seen from Eq 1 and other equations presented in Liu et al (2010a).

Generally speaking, from the isothermal process, RH decreases with decreasing pressure based on the premise that pressure is maintained only by water vapor. In fact, there is air in the chamber as explained previously. For a certain ambient pressure of the vacuum chamber, the total pressure was maintained by air pressure and water vapor pressure. RH in the vacuum chamber was determined by water vapor pressure not by total pressure. Evaporation of water in wood is governed also by temperature and humidity of surrounding conditions in the vacuum chamber. Therefore, RH as well as temperature must be considered to determine EMC of wood even under vacuum.

EMC relationships with temperature and RH under atmospheric pressure have been well established by FPL (1999) and serve well in wood conventional drying processes and wood research. Many equations have been proposed and tested for describing the moisture sorption isotherms of wood (Hailwood and Horrobin 1946; Simpson 1973; Avramidis 1989). Among them, the Hailwood-Horrobin sorption theory has been applied for many years. This model representing sorption isotherms is relatively simple and is in excellent agreement with the experimental results in a broad range of RH (Simpson 1973). In addition, wood has generated sorption isotherms under vacuum conditions similar to the one at atmospheric condition. This was also demonstrated by Voigt et al (1940). Thus, the Hailwood-Horrobin model used for normal atmospheric pressure can also be applied to vacuum conditions by revising some coefficients for the effect of ambient pressure on EMC (Liu et al 2010b).

With the development of vacuum drying procedures, EMC data with sufficient precision under vacuum conditions became more and more important for wood vacuum drying both in theory and practice application. In this study, EMC of three species of wood under vacuum conditions were tested and EMC charts and database under vacuum conditions were built based on the test results and the regression affecting coefficient using the Hailwood–Horrobin model.

MATERIALS AND METHODS

Materials

Materials used were Russian larch (*Larix gmelinii*, 510 kg·m⁻³ basic density, 47.8% initial MC), Hinoki (*Chamaecyparis obtusa* 400 kg·m⁻³ basic density, 37.1% initial MC), and Sugi (*Cryptomeria japonica*, 320 kg·m⁻³ basic density, 90.2% initial MC). Each species was processed into 25 end-matched EMC test samples with dimensions of 5 (L) \times 30 (T) \times 30 (R) mm for every temperature level.

Methods

A vacuum chamber with inside dimensions of 800 mm \times 800 mm \times 1.1 m (YASUJIMA Co., Ltd., Kanazawa-Shi, Japan) (Fig 1) was used for the EMC test under vacuum conditions. The chamber consisted mainly of a pressure control system, a steam generator, and an online monitoring system for dry-bulb and wet-bulb temperature measurement. The performance characteristics of this vacuum chamber, such as precise dry-bulb and wet-bulb temperature measurement, air velocity adjustment under different vacuum conditions, were tested by Myojin et al (2006) and fully met the requirements of the experiment. An electronic balance (AJ100, Mettler-Toledo



Figure 1. Test chamber: 1) leak valve, 2) manometer, 3) condenser, 4) hot water tank, 5) heating pipe, 6) circulating fan, 7) vacuum pump, 8) steam generator, 9) samples, 10) Dry- and wet-bulb temperature sensors, 11) dehumidifier, 12) water collector, and 13) control cabinet.

International, Inc., Columbus, OH) with a precision of 0.1 mg was used to measure the weight of EMC samples.

Temperature and ambient pressure ranges were 45°C, 50°C, and 60°C, and 13.3, 33.3, 53.3, and 101.3 kPa, respectively. For every temperature level, RH ranges were 40%, 50%, 60%, 70%, and 80%. For each temperature and RH condition, the temperature and RH were held constant throughout the test. To obtain the curves of the specimen weight changes and constant weight corresponding to the fixed condition, the specimens were taken out of the vacuum chamber. intermediate mass within a 3-da interval was measured at every pressure condition, and the specimens were considered at EMC, whereas the mass difference was within 2 mg between the last two intervals. First, five samples of each species were put in an ambient pressure of 13.3 kPa until all samples reached EMC at this pressure. After the samples were weighed, the ambient pressure was changed to and held at 33.3 kPa until all samples reached EMC again. After the samples were reweighed, the pressure was changed to and held at 53.3 kPa until all samples reached EMC at this pressure, and the same process was carried out until samples reached EMC under atmospheric pressure. Finally, after weighing, all samples were dried in an oven at $103 \pm 2^{\circ}$ C for 24 h.

The pressure inside the chamber was measured by a diaphragm pressure gauge. The pressure was maintained within 0.26 kPa of the controlling value with the vacuum pump. The internal RH was controlled using dry-bulb and wet-bulb temperatures. To control the wet-bulb temperature, the chamber was equipped with a steam generator, condenser, and cooling pipe, and an air circulating fan was used to equalize the temperature and humidity. A variable-frequency motor was used to ensure the airflow velocity over the wet-bulb probe was more than 1 m/s at various ambient pressure conditions.

RH was calculated from Eq 1 according to the previous study (Liu et al 2010a). Equation 1 shows that RH was affected by ambient pressure.

Therefore, to control the same RH under different ambient pressures, the wet-bulb temperature should be adjusted. For example, to obtain the condition of 45°C and 60% RH under pressures of 13.3, 53.3, and 101.3 kPa, the wet-bulb temperature should be changed from 35.6° C to 36.3° C and then to 37.0° C, respectively.

$$\phi = \frac{P_{\rm w} - \frac{(P - P_{\rm w})(t - t_{\rm w})}{1546 - 1.44t_{\rm w}}}{P_{\rm s}} \times 100\% \qquad (1)$$

where ϕ is RH (%), P_w is saturated vapor pressure at the wet-bulb temperature (kPa), P is ambient pressure (kPa), t is dry-bulb temperature (°C), t_w is wet-bulb temperature (°C), P_s is saturated vapor pressure at the dry-bulb temperature (kPa).

RESULTS AND DISCUSSION

Experimental EMC and Wood Desorption Property Under Constant Vacuum Condition

The experimental results are shown in Table 1 at the specific vacuum conditions. Based on these results, the desorption isotherms of Russian larch, Hinoki, and Sugi at 45°C, 50°C, 60°C, 13.3 kPa, and three ambient pressure levels (13.3, 53.3, and 101.3 kPa) at 50°C are plotted in Figs 2 and 3, respectively. The desorption isotherms in these two figures at any ambient pressure presented the typical sigmoid shape of moisture sorption isotherms of wood (Skaar 1988; Simpson 1979). Figures 2 and 3 and Table 1 show that under vacuum and at any ambient pressure, the effect on EMC was similar for all three species: at the same temperature for a given ambient pressure, EMC increased with an increase in RH, and at a constant RH for a given ambient pressure, EMC decreased with an increase in temperature. We can also see the effects on EMC of ambient pressure in Fig 3. EMC increased with decreasing ambient pressure as described in previous reports (Liu et al 2010a,b). The effect was small at high RH ranges and became significant with decreasing RH. In addition, the effect on EMC of ambient pressure from 53.3 to 101.3 kPa was not obvious (the difference in EMC was only

Species	Pressure (kPa)	Temperature (°C)/RH (%)				
		45/50	45/60	50/40	50/60	60/50
Russian larch	13.3	10.3	11.6	8.6	10.8	9.2
	33.3	9.7	10.8	7.9	10.2	8.4
	53.3	8.7	9.7	6.8	9.3	7.5
	101.3	8.6	9.6	6.4	9.0	7.1
Hinoki	13.3	9.9	10.5	7.2	10.2	8.7
	33.3	9.2	9.8	6.7	9.6	7.9
	53.3	8.2	8.9	6.0	8.7	7.0
	101.3	8.0	8.8	5.7	8.6	6.5
Sugi	13.3	9.2	10.1	7.5	9.8	8.5
	33.3	8.7	9.6	6.6	9.3	7.5
	53.3	8.0	8.9	6.2	8.6	7.2
	101.3	7.6	8.6	5.8	8.5	6.5
USDA FPL	101.3	8.2	9.7	6.5	9.5	7.3

Table 1. Experimental EMC of Russian larch, Hinoki, and Sugi under various conditions.

0.1-0.4%), whereas it became greater from 53.3 to 13.3 kPa (the difference in EMC was 1.2-1.9%). This shows that the extent of effects was significant at low ambient pressure (13.3 kPa) compared with that at medium ambient pressure

(53.3 kPa). Ambient pressure is another factor that affects EMC in addition to temperature and RH. A reason for this effect of ambient pressure could be that at low ambient pressure conditions, air in the microscopic capillaries in the cell wall is





Figure 2. Desorption isotherms of Russian larch, Hinoki, and Sugi wood at three temperatures at 13.3 kPa.

Figure 3. Desorption isotherms of Russian larch, Hinoki, and Sugi wood for three ambient pressure conditions at 50°C.

replaced with bound water resulting in higher MC at low ambient pressure compared with MC at atmospheric conditions. Also, Table 1 shows that the EMC of three species at atmospheric pressure was different from not only the value in FPL (1999) but also from each other, which verified that EMC was affected by species.

Regression Equations of EMC for Larch, Hinoki, and Sugi Under Vacuum Conditions

Experimental EMC results under vacuum conditions for special conditions at 45°C/50%, 45°C/ 60%, 50°C/60%, 50°C/40%, and 60°C/50% at four levels of ambient pressure at 13.3, 33.3, 53.3, and 101.3 kPa are shown in Table 1. EMC in broad ranges of temperature and RH could be obtained by the regression equations based on the previously mentioned test results. According to the results in Table 1, EMC at 60°C/50% at various ambient pressures were graphed in Fig 4. The regression equations of EMC for Russian larch, Hinoki, and Sugi in the special conditions



Figure 4. EMC and regression equations of Russian larch, Hinoki, and Sugi wood at 60°C/50% under vacuum.

were obtained according to Fig 4 and listed in Table 2. As previously explained, EMC increased with decreasing ambient pressure at middle and low RH ranges. Therefore, we can calculate any EMC values corresponding to 60°C/50% below 13.3 kPa because they could not be tested using the equipment in this study. For other special conditions, the regression equations of EMC are also listed in Table 2. These can be used for EMC calculations at the corresponding conditions for any ambient pressures.

EMC Database Building at 13.3-kPa Vacuum Condition

As mentioned in the Introduction, the Hailwood– Horrobin model representing sorption isotherms is relatively simple and is in excellent agreement with the experimental results in a broad range of RH. The Hailwood–Horrobin formula (Simpson 1973) to calculate EMC is

$$EMC = \left(\frac{KK_1h + 2K^2K_1K_2h^2}{1 + K^2K_1K_2h^2 + K_1Kh} + \frac{Kh}{1 - Kh}\right) \times \frac{1800}{W}$$
(2)

$$W = 349 + 1.29 \times T + 0.0135 \times T^{2}$$

$$K = 0.805 + 0.000736 \times T - 0.00000273 \times T^{2}$$

$$K_{1} = 6.27 - 0.00936 \times T - 0.000303 \times T^{2}$$

$$K_{2} = 1.91 + 0.0407 \times T - 0.000293 \text{ T}^{2}$$

where EMC is EMC (%), *T* is temperature (°C), and *h* is RH (%/100). The EMC under vacuum conditions can be calculated from Eq 2, which was used to compare the experimental EMC results (three species) in Table 1 at 13.3 kPa and the results are presented in Fig 5. We can see that the relationships are linear. The Hailwood–Horrobin formula for EMC calculation may be revised using the regression equations. The coefficient can be obtained as the revising coefficient for the Hailwood–Horrobin formula to calculate EMC at 13.3 kPa. According to the regression equations in Fig 5, EMC

Condition	Russian larch	Hinoki	Sugi
45°C/50%	$0.0003x^2 - 0.0605x + 11.116$	$0.0004x^2 - 0.0629x + 10.71$	$0.0002x^2 - 0.0429x + 9.7822$
45°C/60%	$0.0004x^2 - 0.0735x + 12.599$	$0.0004x^2 - 0.0619x + 11.277$	$0.0002x^2 - 0.0445x + 10.699$
50°C/40%	$0.0004x^2 - 0.0679x + 9.5495$	$0.0002x^2 - 0.045x + 7.838$	$0.0003x^2 - 0.0488x + 8.0206$
50°C/60%	$0.0003x^2 - 0.0559x + 11.573$	$0.0004x^2 - 0.0634x + 11.097$	$0.0003x^2 - 0.046x + 10.431$
60°C/50%	$0.0004x^2 - 0.0673x + 10.101$	$0.0003x^2 - 0.0625x + 9.4927$	$0.0002x^2 - 0.0511x + 9.0787$

Table 2. EMC regression equations for Russian larch, Hinoki, and Sugi at different conditions.

data calculation formulas were modified as subsequently shown. EMC in broad ranges of temperature and RH at 13.3 kPa can be calculated from modified Eqs 3-5 for Russian larch, Hinoki, and Sugi: to 99% at 13.3-kPa ambient pressure. The EMC data in Fig 6 can serve not only for the real-time measurement of MC in previous studies (Liu et al 2010b; Cai and Hayashi 2007) but also as an aid in wood vacuum drying control.

$$EMC_{L} = 0.9578 \times EMC_{Hailwood} + 1.9623 = 0.9578 \times \left(\frac{KK_{1}h + 2K^{2}K_{1}K_{2}h^{2}}{1 + K^{2}K_{1}K_{2}h^{2} + K_{1}Kh} + \frac{Kh}{1 - Kh}\right)$$
(3)
$$\times \frac{1800}{W} + 1.9623$$

$$\operatorname{EMC}_{\mathrm{H}} = 0.9192 \times \operatorname{EMC}_{\mathrm{Hailwood}} + 0.5716 = 0.9192 \times \left(\frac{KK_{1}h + 2K^{2}K_{1}K_{2}h^{2}}{1 + K^{2}K_{1}K_{2}h^{2} + K_{1}Kh} + \frac{Kh}{1 - Kh}\right)$$
(4)
$$\times \frac{1800}{W} + 1.5716$$

$$EMC_{S} = 0.9472 \times EMC_{Hailwood} + 1.0711 = 0.9472 \times \left(\frac{KK_{1}h + 2K^{2}K_{1}K_{2}h^{2}}{1 + K^{2}K_{1}K_{2}h^{2} + K_{1}Kh} + \frac{Kh}{1 - Kh}\right)$$
(5)
$$\times \frac{1800}{W} + 1.0711$$

where *K*, K_1 , K_2 , and *W* are the same as those in Eq 2, *T* is temperature (°C), and *h* is RH (%/100). Based on these modified equations, EMC charts were graphed in Fig 6 for the three species, temperature from 30°C to 90°C, and RH from 30%

EMC of Larch, Hinoki, and Sugi at Various Vacuum Conditions

EMC corresponding to temperature and pressure conditions in Table 1 below 13.3 kPa could be



Figure 5. Relationship between experimental EMC at 13.3 kPa and that calculated from the Hailwood model.



Figure 6. EMC chart of Russian larch (a), Hinoki (b), and Sugi (c) at 13.3 kPa.

calculated from the corresponding equations in Table 2. Table 3 shows the calculated EMC at 6.7 kPa corresponding to the temperature and RH conditions. The effective coefficient at 6.7 kPa (Fig 7) can be obtained at 13.3 kPa by comparing the EMC in Table 3 with the one calculated from Eq 2. Therefore, EMC in broad ranges of temperature and RH at 6.7 kPa can be calculated from the modified Hailwood Eqs 6-8 for Russian larch, Hinoki, and Sugi.

In this study, EMC tests below 13.3 kPa could not be carried out because of the limitations of the test equipment. However, an EMC below 13.3 kPa corresponding to the temperature and RH in this experiment could be calculated from the regression equations in Table 2. The effective coefficient below 13.3 kPa can be obtained by comparing EMC obtained from the regressions in Table 2 with EMC calculated from Hailwood Eq 2. Finally, all EMC database values in broad ranges of temperature and RH at vacuum conditions can be obtained from the modified Hailwood equations. The EMC charts shown in Fig 6 also could be established based on the modified Hailwood equations at various vacuum conditions to serve as an aid in wood research and drying control at vacuum conditions.

$$EMC_{L} = 0.9499 \times EMC_{Hailwood} + 2.4584 = 0.9499 \times \left(\frac{KK_{1}h + 2K^{2}K_{1}K_{2}h^{2}}{1 + K^{2}K_{1}K_{2}h^{2} + K_{1}Kh} + \frac{Kh}{1 - Kh}\right)$$
(6)

$$\times \frac{1800}{W} + 2.4584$$

$$EMC_{H} = 0.9097 \times EMC_{Hailwood} + 1.9878 = 0.9097 \times \left(\frac{KK_{1}h + 2K^{2}K_{1}K_{2}h^{2}}{1 + K^{2}K_{1}K_{2}h^{2} + K_{1}Kh} + \frac{Kh}{1 - Kh}\right)$$
(7)
$$\times \frac{1800}{W} + 1.9878$$

$$EMC_{S} = 0.922 \times EMC_{Hailwood} + 1.6501 = 0.922 \left(\frac{KK_{1}h + 2K^{2}K_{1}K_{2}h^{2}}{1 + K^{2}K_{1}K_{2}h^{2} + K_{1}Kh} + \frac{Kh}{1 - Kh} \right)$$

$$\times \frac{1800}{W} + 1.6501$$
(8)

		RH (%)				
Species	Temperature (°C)					
		40	50	60	70	80
Russian larch	45	9.4	10.7	12.1	13.6	17.2
	50	9.1	10.3	11.2	12.9	16.1
	60	8.6	9.6	10.7	12.6	15.4
Hinoki	45	8.8	10.3	10.8	12.4	15.8
	50	7.5	9.4	10.6	12.1	15.1
	60	8.1	9.0	9.9	11.6	14.7
Sugi	45	8.4	9.5	10.4	12.4	15.3
	50	7.7	9.0	10.1	12.3	14.9
	60	7.8	8.8	9.9	11.9	14.5

Table 3. Obtained EMC at 6.7 kPa for different conditions from the regression equations.



Figure 7. Relationships between experimental EMC at 6.7 kPa and EMC calculated from Hailwood model.

CONCLUSIONS

A database with charts and equations related to temperature, RH, and ambient pressure was built based on experimental EMC results. The property of wood sorption under vacuum conditions was also investigated. Under vacuum, wood performed similar desorption isotherms to those at normal pressure conditions. MC of wood also decreased with temperature decrease at constant RH and increased with RH increase at constant temperature. Ambient pressure affected EMC of wood. The effect was not obvious at high RH ranges although it was greater when RH became low. The extent to which EMC at ambient pressure became significant decreased with ambient pressure. The difference in EMC was from 0.1-0.4% at relative high ambient pressure to 1.2-1.9% at low ambient pressure. The charts and equations were obtained based on the experimental results and regression equations to build EMC corresponding to temperature, RH, and ambient pressure. The database could be used widely in wood vacuum drying and research.

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