Effects of Removal of Chemical Components on Moisture Adsorption by Wood

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To investigate the effects on moisture sorption behavior of wood caused by the removal of chemical components, *Populus euramericana* flour (40 to 60 mesh) was divided into four groups: untreated, extractives removed, hemicellulose removed, and matrix removed. The samples at the fiber saturation point and under oven-dried conditions were separately exposed to relative humidities of 11%, 45%, and 75% at 25 °C for desorption and adsorption. Additionally, moisture changes were measured during the processes. The results showed that moisture changed rapidly for all four groups at the initial stage during sorption, after which the rate gradually decreased until a steady-state equilibrium was reached. Among the four groups, the samples from which extractives had been removed exhibited the highest moisture content and moisture sorption coefficient, followed by the untreated samples, hemicellulose removed, and matrix removed samples. With increasing relative humidity, the hysteresis ratio A/D of the samples increased, indicating a reduction in sorption hysteresis, which was further decreased by hemicellulose extraction.

Keywords: Wood flour; Chemical component removing; Adsorption; Moisture content; Sorption hysteresis

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INTRODUCTION

Wood is a natural hygroscopic material with a great number of free hydroxyl groups in the cell wall. When the temperature or humidity changes, wood adsorbs moisture from the external environment, thereby forming hydrogen bonds, or releases moisture through the cutting of hydrogen bonds. This results in corresponding variations in the moisture content (MC) of wood, with its dimensional stability, mechanical properties, environment-humidifying ability, and other properties changing accordingly. Therefore, the study on the moisture sorption of wood plays an essential role in the understanding of wood properties as well as the use of wood products.

The hygroscopicity of wood is indirectly evaluated by a sorption isotherm that can either be measured experimentally from equilibrium moisture content (EMC) of wood at different relative humidities under a certain temperature, or calculated for the full hygroscopic range through moisture sorption theory, which is further capable of separating out the amount of adsorbed water for monolayer and multilayers, respectively. The moisture sorption theories proposed by Dent, Brunauer-Emmett-Teller (BET), and Hailwood-Horrobin (H-H) are the most classic among those in current use (Ma and Zhao 2012). Simpson (1973) evaluated 10 early moisture sorption theories and found that the H-H theory had the best simulation of the experimental data.

Various studies (Zsigmondy 1911; Urquhart 1929; Liu and Zhao 2004) have shown that the water sorption isotherms obtained for wood adsorption and desorption under the same temperature do not overlap, implying that water vapor sorption by wood is an irreversible process. The phenomenon in which adsorption MC is always lower than desorption MC at certain equilibrium states below 75 °C (Engelund *et al.* 2013) is called sorption hysteresis (Liu and Zhao 2004). The ratio of adsorption EMC to desorption EMC, defined as the hysteresis ratio (Liu and Zhao 2004), represented by A/D, has been found to generally range from 0.75 to 0.9 (Skaar 1988). It has been reported that the hysteresis ratio is affected by relative humidity (RH) (Spalt 1958; Okoh 1976). Specifically, as RH increases, A/D tends to increase, suggesting a reduction in sorption hysteresis.

There are many interpretations for this hysteresis. One is the “ink bottle theory” believing that water is much easier to move out of wood during desorption than to enter it when adsorption happens, based on the shape of a “ink bottle”. Another considers it as the difference in contact angle between water and the internal surface of wood substance during adsorption and desorption. Both of the two theories are raised from capillary condensation, while the most widely accepted theory is the “effective hydroxyl group theory” proposed by Urquhart (Liu and Zhao 2004), which suggests that the amount of free hydroxyl groups during adsorption process is fewer compared with desorption process. On the other hand, Engelund *et al.* (2013) attributed sorption hysteresis to stress relaxation, pointing out that moisture equilibrium of wood would change along with the rate of stress relaxation depending on a transition from glassy state to rubbery state by the softening of amorphous hemicellulose. As a result, hemicellulose was thought to have a significant effect on the sorption hysteresis of wood.

As a natural polymer, wood is mostly composed of cellulose, hemicellulose, and lignin (the latter two collectively known as the matrix), and a minor amount of extractives. Cellulose and hemicellulose are rich in free hydroxyl groups, components that contribute to the hygroscopicity of wood, whereas lignin is a hydrophobic heteropolymer. As a result, delignification will weaken the water resistance capabilities of wood. Removing extractives will encourage wood sorption due to the fact that extractives deposit in cell cavity which could block the passage for water (Ma and Zhao 2012). Studies have shown that heat treatment can effectively reduce hygroscopicity (Zhou *et al*. 2012), moisture uptake rate (Bak and Németh 2012), and sorption hysteresis (Tu *et al*. 2010). These reductions are related to the degradation of hemicellulose, and some researchers also believe that other chemical components of wood, like lignin, play an important role (Repellin and Guyonnet 2005).

In a previous study (Zhou *et al*. 2014), the moisture and dimensional changes of lignin-removed wood during adsorption processes were investigated. Based on this, the effect of lignin on moisture sorption and hygroexpansion of wood was discussed.

This paper focused on the impact of hemicellulose. Wood samples were divided into four groups: untreated, extractives removed, hemicellulose removed, and matrix removed. FTIR analyses were used to study the chemical transformations during the chemical-removing treatments. The adsorption processes of the samples were measured at three different relative humidities. Meanwhile, the sorption isotherms of all samples were calculated by Hailwood-Horrobin sorption theory (Hailwood and Horrobin 1946), from which an attempt to explain the mechanism of sorption hysteresis was made using the stress relaxation theory proposed by Engelund *et al.* (2013). This study not only can provide some fundamental data for the physical properties of chemical components of wood, and enrich the content of wood chemistry and wood physics, but also can prompt a new insight into wood sorption through the interactions between the main components of wood.

EXPERIMENTAL

Materials

Poplar (*Populus euramericana*), selected as the study species, was ground into flour (40 to 60 mesh) by a grinding miller.

Chemical Treatment

The samples were dried at 105 °C until a constant weight (m_0) was achieved, and then divided into four groups: untreated (Control), extractives removed (DE), hemicellulose removed (DHC), and matrix removed (DM), whose chemical treatments were conducted as follows.

Extractives removed: the samples were extracted with a mixture of ethanol and benzene (1:2 volume ratio) for 48 h and then boiled for 3 h at 60 °C in the same solution heated by water bath to remove soluble extractives. They were then washed with distilled water, air-dried, and finally dried at 105 °C until a constant weight (m_1) was achieved (Xie 2006).

Hemicellulose removed: the DE was further extracted by liquid hot-water at 170 °C in a reactor for 2 h with a water-to-solid ratio of 20:1 (w/w) and then washed with distilled water, air-dried, and finally dried at 105 °C until a constant weight (m_2) was achieved (Hosseinaei *et al*. 2012a,b).

Matrix removed: the DE was first delignified with a mixture of 967 mL of distilled water, 20 g of $NaClO_2$, and 13 mL of CH_3COOH at 40 °C in a water bath for 30 h and then washed with distilled water, air-dried, and dried at 105 °C until a constant weight (m_3) was achieved (Zhang *et al*. 2006a,b,c). This was followed by an extraction of hemicellulose based on the liquid hot-water approach above to obtain a constant weight (m_4).

The ratio of extractives loss (P_1), hemicellulose loss (P_2), matrix loss (P_3), and lignin loss (P_4) was calculated according to Eqs. 1 through 4.

$$P_1 = \frac{m_0 - m_1}{m_0} \times 100\% \tag{1}$$

$$P_2 = \frac{m_0 - m_2}{m_0} \times 100\% \tag{2}$$

$$P_3 = \frac{m_0 - m_4}{m_0} \times 100\% \tag{3}$$

$$P_4 = \frac{m_0 - m_3}{m_0} \times 100\% \tag{4}$$

Approximately 3% of extractives in DE and 9% of the hemicellulose in DHC were removed, each based on the oven-dried weight (m_0) of wood flour. In DM, the matrix loss was 38%, wherein lignin and hemicellulose losses were 14% and 24%, respectively. After chemical treatment, each group was divided into three parts, with each part comprising approximately 0.5 g. They were then stored in a sealed container for use.

Fourier Transform Infrared Spectroscopy (FTIR)

The samples were pulverized (100 mesh), mixed with potassium bromide (KBr), and pressed as pellets before being examined by FTIR (Bruker VERTEX 70V, Germany) in the scanning range of 400 to 4000 cm^{-1}, and a resolution of 4 cm^{-1}, and 32 scans per sample were used.

X-Ray Diffraction (XRD)

XRD analysis of the samples were carried out on an X-ray diffractometer (Bruker D8 Advance, Germany). The X-ray beam was Cu-Kα radiation (λ= 0.1540 nm) and operated at 40 kV and 40 mA. The scanning rate and step size were 0.5s/step and 0.02^{o} with 2θ varying from 5° to 40°.

Desorption and Adsorption Test

Samples at the fiber saturation point (conditioned at 100% RH) and the over-dried condition were separately exposed to three RH conditions, 11%, 45%, and 75% at 25 °C, for desorption and adsorption, and the moisture changes were measured during the processes.

The three RH conditions were obtained using saturated salt solutions of lithium chloride, potassium carbonate, and sodium chloride, respectively (Macromolecule Academy 1958).

Hailwood-Horrobin Sorption Theory

The sorption behavior of chemical component-removed wood was explored using the H-H sorption theory (Hailwood and Horrobin 1946), and the results were compared with the Control. In the theory, the sorption isotherm of wood can be described as a parabola by,

$$\boldsymbol{h/m = A + Bh - Ch^2} \tag{5}$$

$$\boldsymbol{A = \frac{W}{18}\frac{1}{K_2(K_1+1)}, B = \frac{W}{18}\frac{K_1-1}{K_1+1}, C = \frac{W}{18}\frac{K_1K_2}{K_1+1}} \tag{6}$$

where h is the relative humidity, m is the moisture content of wood, K_1 is the equilibrium constant, where the hydrated wood is formed from the dissolved water and dry wood, K_2 is the equilibrium constant between dissolved water and water vapor, $18/W$ is the moisture content corresponding to complete hydration of all of the available sorption sites.

Hydrated water (m_h) and dissolved water (m_s) can be further figured out through Eqs. 7 and 8:

$$\boldsymbol{m_h = \frac{18}{W} K_1K_2h/(1+K_1K_2h)} \tag{7}$$

$$m_s = \frac{18}{W} K_2 h/(1 - K_2 h) \tag{8}$$

RESULTS AND DISCUSSION

Analysis of Chemical Components Based on FTIR

The adsorption peaks of the FTIR spectrum for poplar wood are listed in Table 1. Figure 1 displays the FTIR spectra for various chemical component-removed samples (all of them were normalized). The peaks at 3414 and 2922 cm^{-1} played a limited role in the analysis of changes in wood chemical components because the three main chemical constituents of wood all contain hydroxyl and methylene (Cheng 2011). Therefore, changes in cellulose and hemicellulose are usually investigated by the intensity variation for the characteristic peaks of oxygen-containing functional groups (1165, 1115, and 1051 cm^{-1}). It is worth noting that the peak of carbonyl at 1739 cm^{-1} is characteristic of hemicellulose, which is different than other chemical components (Deng *et al.* 2008). In addition, lignin can be confirmed by the characteristic peaks of benzene rings at 1608, 1506, 1427, and 1270 cm^{-1}.

Table 1. FTIR Absorption Peak Location and Assignment of Poplar Wood

Wavenumber (cm^{-1})	Absorption peak location and assignment
3414	O-H stretching in hydroxyl groups (Zhu *et al.* 2012)
2922	C-H asymmetric stretching in methylene groups (Zhu *et al.* 2012)
1739	Carbonyl stretching (Moniruzzaman and Ono 2013)
1608, 1506, 1427	Benzene ring stretch (Huang *et al.* 2015)
1270	Ar-O stretching in oxygen bond of benzene ring (Cheng 2011)
1165	C-O-C stretching (Chang *et al.* 2014)
1115	OH association absorption band (Müller *et al.* 2008)
1051	C-O stretching (Müller *et al.* 2008)

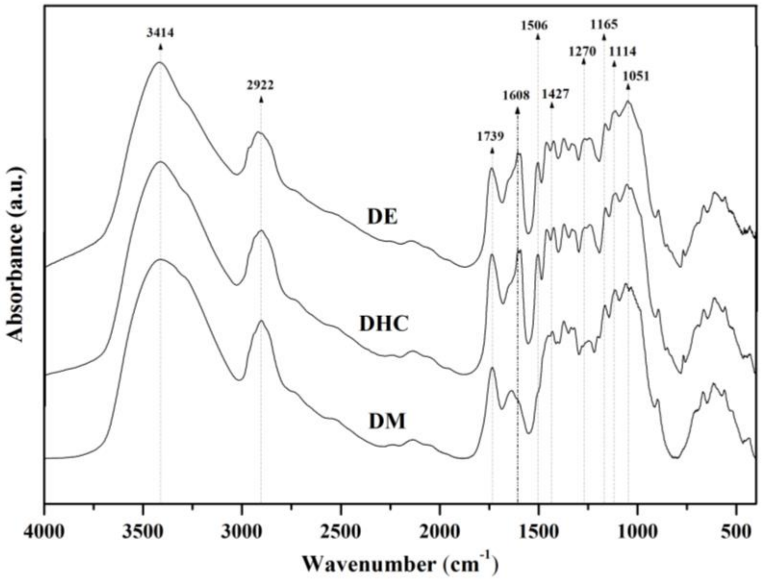

Fig. 1. FTIR spectra for different chemical component-removed samples

As shown in Fig. 1, compared with the DE, the intensity of the peaks at 1608, 1506, and 1427 cm^{-1}, attributed to benzene ring vibration, and the peak at 1270 cm^{-1}, corresponding to Ar-O stretching for the DHC, increases, suggesting an increase in the relative amount of lignin within the DHC caused by hemicellulose removal.

For the DM, the peak at 1506 cm^{-1} disappeared, and the intensity of the peaks at 1608, 1427, and 1270 cm^{-1} clearly decreased. Meanwhile, the peak of carbonyl stretching at 1739 cm^{-1} receded, which proved that both lignin and hemicellulose were degraded. Alternatively, comparing the DM with the DHC, the intensity of the peak at 1739 cm^{-1} of the DM was weaker, indicating that the hemicellulose loss of the DM was greater than that of the DHC.

XRD Analysis

XRD patterns for the treated samples are shown in Fig. 2. The positions of the diffraction peaks for crystal plane (002) and (040) at 2θ = 22.5° and 35° are used to characterize crystalline structure in cellulose (Liu *et al.* 2014). These positions of the DHC and DM did not change compared with the DE, suggesting that chemical treatment did not influence the main crystalline structure of the samples. What's more, the crystallinity of DE, DHC and DM was determined as 36.4%, 41.3%, and 48.5%, respectively, indicating the amorphous region was destroyed after hemicellulose-removal treatment and decreased after washing with distilled water, leading to an increase in the relative amount of crystallization. In addition, the DM showed higher crystallinity because hemicellulose extraction in the DM was greater, which can also prove that hemicellulose of the samples was removed without breaking the crystalline structure in cellulose.

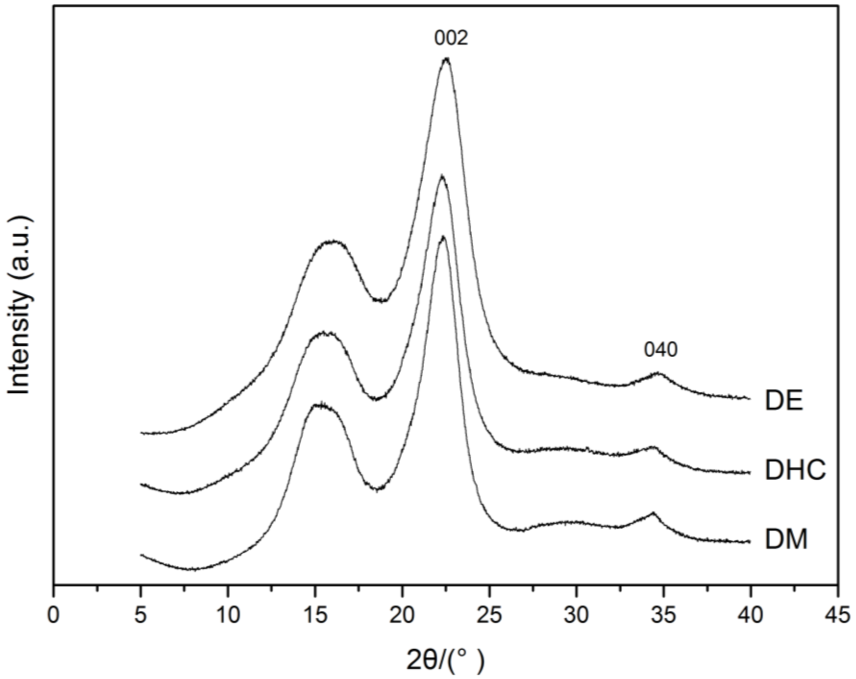

Fig. 2. XRD patterns for different chemical component-removed samples

Moisture Changes

Figure 3 takes sorption processes at 75% RH as an example to present moisture desorption and adsorption kinetics for the samples. Moisture changed rapidly for all four groups at the initial stage during sorption, wherein the rate gradually decreased until a state of equilibrium was reached. Among the four groups, the extractives removed samples exhibited the highest moisture content, followed by the untreated samples, hemicellulose removed samples, and matrix removed samples. As mentioned above, extractives would block of water passage to a certain extent, which can slightly restrain the moisture sorption of wood, while hemicellulose removal can greatly reduce wood hygroscopicity because of a dramatic loss in the number of exposed hydroxyl groups. Both lignin and hemicellulose exist in the wood cell wall in an amorphous state and are linked to each other by complex chemical bonds (Ralph *et al*. 1995); hence, delignification can greatly weaken resistance to hemicellulose removal. As a result, hemicellulose loss in the DM was greater than lignin loss (as shown in the section "Chemical Treatment"). Compared with the control, the hygroscopicity of the DM decreased because the hemicellulose loss of the DM exceeded lignin loss, although lignin is a hydrophobic heteropolymer and delignifying was supposed to lower water resistance. In addition, the DM showed less moisture than that of the DHC because hemicellulose extraction in the DM was greater.

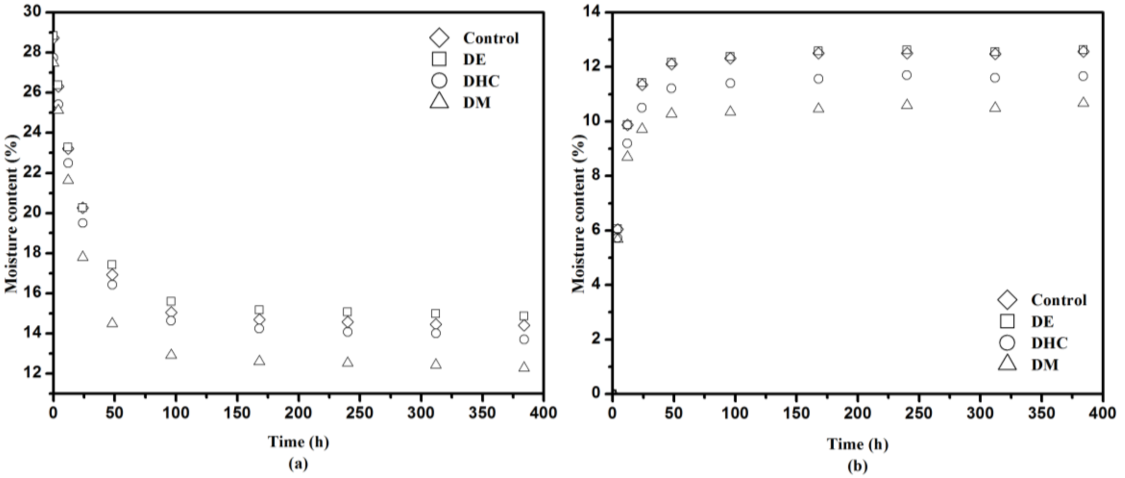

Fig. 3. Moisture (a) desorption and (b) adsorption processes under 75% RH at 25 °C for various samples

Table 2. Equilibrium Moisture Content (%) at Various Relative Humidities

	Desorption				Adsorption		
	11%	45%	75%		11%	45%	75%
Control	3.53(0.096)	7.70(0.147)	14.54(0.101)		2.22(0.095)	6.29(0.136)	12.40(0.011)
DE	3.72(0.239)	7.91(0.031)	14.87(0.111)		2.41(0.220)	6.47(0.068)	12.62(0.219)
DHC	3.28(0.159)	7.29(0.124)	13.74(0.184)		2.21(0.155)	6.11(0.119)	11.74(0.086)
DM	3.12(0.152)	6.66(0.050)	12.28(0.200)		2.20(0.131)	5.75(0.060)	10.67(0.049)

Data provided as the average value (standard deviation) from three replicates

Sorption Isotherm

Table 2 summarizes the equilibrium moisture contents of the samples at various relative humidities. Using these data, the main parameters given in Eqs. 5 and 6 can be calculated. The values for adsorption are listed in Table 3.

In Table 3, K_2 values of the samples ranged from approximately 0.8 to 0.9, similar to the values for solid wood (Skaar 1988). In addition, all the values of $18/W$ of wood flours under various chemical treatments fell in the range of 4% to 5%. In other words, the MC of samples should be between 4% and 5% when all the hydroxyls available are saturated by a monolayer of adsorbed water. This amount is also in accordance with previous research on solid wood, where a value of 5% was determined (Ma and Zhao 2012). A comparison of $18/W$ values between samples demonstrated that the DE was equal to the control, suggesting that the obvious moisture sorption of wood after the removal of extractives was due to the filling effect rather than an increase in hydroxyls. Alternatively, the $18/W$ values for both the DHC and DM were lower than the Control, suggesting a reduction of free hydroxyl groups after hemicellulose removal. Similarly, the hemicellulose loss of the DM was much greater than that in the DHC, and, accordingly, the DM showed a minimum $18/W$ value.

Table 3. Values for the Parameters in Hailwood-Horrobin Sorption Theory

	Control	DE	DHC	DM
K_1	6.13	7.23	6.53	7.65
K_2	0.86	0.87	0.85	0.83
$18/W$ (%)	4.69	4.69	4.59	4.29

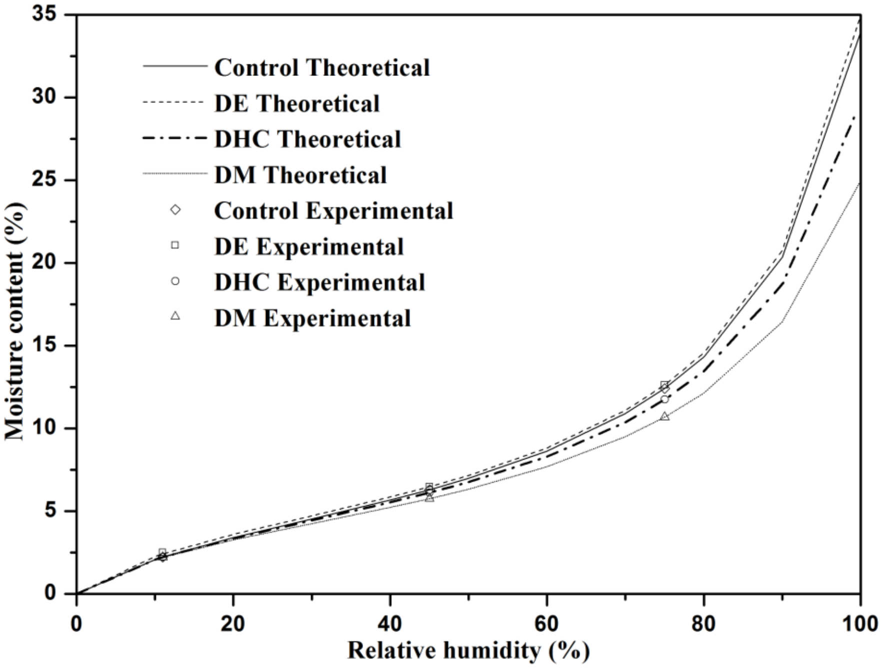

Fig. 4. Sorption isotherms calculated by H-H sorption theory and a comparison with experimental data (25 °C)

The sorption isotherms calculated using the equilibrium moisture content of adsorption according to H-H sorption theory are shown in Fig. 4. The theoretical curves are quite similar to the experimental results, which confirms the effectiveness of the data in Table 3. Furthermore, the final water absorption readings of the samples were in the following order: DE > Control > DHC > DM.

The hygroscopicity of wood decreased as hemicellulose removal increased, which agrees with the FTIR results, in which the hemicellulose loss of the DM was greater than that of the DHC. In addition, the contents of hydrated water (Fig. 5 (a)) and dissolved water (Fig. 5 (b)) in the samples were characterized by a trend in which the DE showed the highest MC, followed by the Control, DE, and DM.

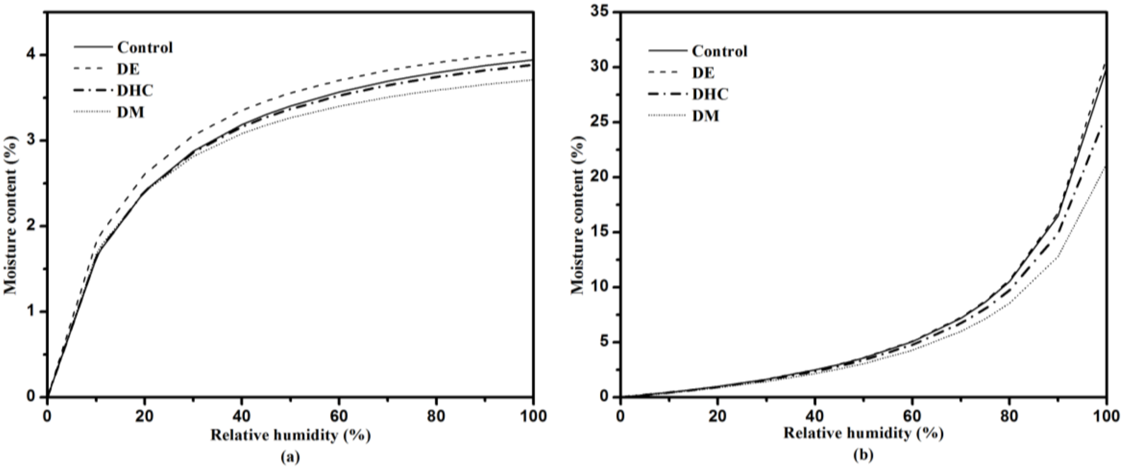

Fig. 5. Moisture content of samples for (a) hydrated water and (b) dissolved water

The slope of the sorption isotherm illustrates the variation in moisture content per unit RH change. This is defined as the moisture adsorption coefficient *Z*, which can be used as an index to evaluate wood hygroscopicity (Yang and Ma 2013). The coefficients within the RH range of 45% to 75% in this study are listed in Table 4. It can be found that the *Z* of the control was 0.20, a value that was close to the mean values for 28 wood species in a previous study (Noack *et al*. 1973). Additionally, the *Z* of the DHC and the DM were lower than that of the Control, indicating a drop in hygroscopicity for both the DHC and DM.

Table 4. Moisture Sorption Coefficient *Z* of the Samples

	Control	DE	DHC	DM
Z	0.20	0.21	0.19	0.16

Table 5 gives the sorption hysteresis ratio *A*/*D* of the samples at three relative humidities. The *A*/*D* of chemically treated wood flour was still in the same range as that of solid wood, *i.e.*, 0.75 to 0.9 (Skaar 1988). Moreover, with an increase in relative humidity, the *A*/*D* of the samples increased, indicating a reduction in sorption hysteresis. This was because an increase in RH promoted the softening of hemicellulose (Engelund *et al*. 2013), which weakened the difference in MC between adsorption and desorption, causing sorption hysteresis to decrease as a result. The hysteresis ratio of the DE changed slightly, but those of the DHC and DM increased. Again, this can be explained by the relaxation theory proposed by Engelund *et al*. (2013) which states that sorption hysteresis of wood is primarily affected by the softening of hemicellulose, so a reduction in sorption hysteresis would follow after hemicellulose removal.

Sorption Hysteresis

Table 5. A/D of the Samples at Several RHs

RH	11%	45%	75%
Control	0.630	0.817	0.853
DE	0.648	0.819	0.849
DHC	0.674	0.838	0.854
DM	0.707	0.864	0.869

CONCLUSIONS

The adsorption and desorption behaviors of chemical component-removed wood at a constant temperature and various humidities were investigated in this study. The main conclusions are as follows:

1. Moisture changed rapidly in all four groups at the initial stage during sorption, in which the rate gradually decreased until a state of equilibrium was reached.
2. Among the four groups, the extractives removed samples exhibited the highest moisture content and moisture sorption coefficient, followed by the untreated samples, hemicellulose removed samples, and matrix removed samples.
3. With an increase in RH, an increase in the hysteresis ratio *A*/*D* followed, indicating a reduction in sorption hysteresis, which was further decreased by hemicellulose extraction.

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