Moisture Adsorption and Hygroexpansion of Paraffin Wax Emulsion-treated Southern Pine (*Pinus* spp.)

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Moisture adsorption and hygroexpansion behaviors of southern pine (*Pinus* spp.) treated with 0.5, 1, and 2% concentrations of paraffin wax emulsion were investigated. The specimens, 4 mm along the grain and 20 mm in radial and tangential directions, were exposed to seven different relative humidity conditions of 11, 22, 33, 45, 60, 75, and 92% for adsorption at 30 °C, which was controlled by a self-designed temperature conditioning chamber. Weights and transverse dimensions of the specimens were measured at certain time intervals during the adsorption processes. Results showed that paraffin wax emulsion treatments could reduce both equilibrium moisture content and adsorption rate. Additionally, paraffin wax emulsion treatments also improved dimensional stability, as indicated by estimation of the humidity expansion coefficient (*Y*) as well as moisture expansion coefficient (*X*).

Keywords: Wood; Paraffin wax emulsion; Adsorption; Hygroexpansion

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INTRODUCTION

Wood is a hygroscopic and porous material. The sorption behavior, shrinkage, and swelling, which restrict its wide usage, are very important properties and quality characteristics. Because of changes in atmospheric relative humidity (RH) and temperature, wood is constantly subjected to moisture adsorption and desorption (Ma and Zhao 2012), and concurrent dimensional changes also take place. Therefore, it is very important to investigate moisture changes and hygroexpansion of wood as well as their relationship with each other.

Static sorption research in the available literature has put a lot of effort into two main aspects, namely the hygroscopic capability and sorption rate (Ma *et al*. 2009). The hygroscopic capability can be defined by the amount of water that wood can adsorb under various environmental conditions. It is characterized by isotherm studies relating the equilibrium moisture content (EMC) to different relative humidities at constant temperature. Since the 1920s, researchers have proposed numerous moisture sorption theories to describe the isotherm mathematically. Thereby, Simpson (1973) evaluated 10 sorption models for wood-water systems on the basis of how well they could functionally relate data for the EMC and RH and found that the Hailwood-Horrobin model was the most accurate of those tested. On the other hand, investigations on the sorption rate of wood have been frequently conducted based on Fick’s Second Law (Stamm 1959; Droin-Josserand *et al*. 1988; Liu *et al.* 2001). However, the relation between moisture sorption and time cannot be easily determined because of the complex solution of the Fickian differential equation. As a result, some studies preferred describing sorption kinetics from the point of view other than Fickian behavior (Christensen and Kelsey 1959; Kelly and Hart 1969; Skaar *et al*. 1970; Nakano 1994a,b; Zhang *et al*. 2007; Ma *et al.* 2010).

Wood used outdoors is prone to physical, chemical, and biological degradation. The susceptibility to fungal decay of outdoor wooden constructions greatly depends on moisture content (MC). Therefore, efforts have been undertaken to reduce MC and keep it below the critical value of approximately 20%. Modification by chemical, thermal, surface or impregnation treatments can be helpful (Hill 2006). Until recently, these shortcomings have been addressed by impregnating wood with appropriate hydrophobes (Stamm 1964; Kumar 1994). Consequently, wood treated with water repellent or biocide not only improved the decay resistance of wood in above-ground applications but also promoted the weathering and dimensional stability of exposed lumber. Water repellent agents, such as linseed oil, waxes, silane, methyl methacrylate, polyethylene glycol (PEG), aqueous solutions of phenol-formaldehyde resin-forming compounds, styrene or methyl methacrylate, and neutral or mildly alkaline are diverse and abundant. Historically in China, water repellent was made from tung oil and bitumen (Wu and Zhang 1999). These substances are applied as solutions in a light organic solvent and usually by immersion or vacuum impregnation. Water repellents applied to wood commercially have been almost exclusively of the simple non-chemically bonded type, mainly based on paraffin waxes (Rowell and Banks 1985).

Paraffin has been gradually recognized because of its advantages, such as abundant source, cost-effectiveness for improving durability, and environmentally benign properties. In China, the three major kinds of paraffin used in artificial boards are based on solid paraffin, paraffin wax emulsion, and a new type that contains paraffin such as tall oil paraffin emulsion, tea saponin paraffin emulsion, and fatty acid ammonium paraffin emulsion (Liu *et al*. 2006). Also, water repellent agents known as the M-system, developed by the Chinese Academy of Forestry, performed satisfactorily in preserving solid wood and artificial boards (OuYang 1990). Besides, various companies have already commercialized water repellent systems for application such as CCA-treated decking, and formulations for ground contact applications may be available in the future (Chen *et al.* 2009; An 2013). Other researchers (Wang *et al*. 2014) have also examined the combination of water repellent and a metal chelator to protect wood against decay and mold fungi. The question is whether the same will be true for protection against moisture adsorption.

Therefore, in the present study, the influence of paraffin wax emulsion at economical feasible concentrations on solid wood was investigated under laboratory conditions. This work was conducted to observe the effect of paraffin wax emulsion (PWE) on moisture changes and transverse dimensional swelling of wood during adsorption processes.

EXPERIMENTAL

Materials

The test material was southern pine (*Pinus* spp.), free of visual defects with dimensions of 4 mm along the grain and 20 mm in both tangential and radial directions.

The specimens were divided into four similar groups according to weight; these included a control and treatments with 0.5, 1, and 2% PWE.

Paraffin wax emulsion was prepared in the laboratory, and the solid content was about 40% (Wang *et al*. 2014). The emulsion was diluted to concentrations of 0.5, 1, and 2%, recorded as 0.5% PWE, 1% PWE, and 2% PWE, respectively.

Methods

Wood treatments

Wood samples were treated *via* a full-cell process. Specifically, test samples were submerged with treatment liquids and placed in a vacuum at -0.1 MPa for 30 min. Subsequently, the pressure was increased to 0.5 MPa and maintained for 1 h. Finally, the pressure was relieved and the samples were taken out.

The weight percent gains (WPGs) were 0.7, 1.5, and 2.5% for 0.5, 1, and 2% WPE treated wood, respectively, based on the calculation from Eq. 1,

$$WPG_s = \frac{m_t - m_u}{m_u} * 100\,(\%) \tag{1}$$

where *m* is average oven-dry mass of the specimen before (u) and after (t) the treatment.

Moisture adsorption

The specimens were oven-dried at 103 ± 2 °C, after which their oven-dry weights and tangential and radial dimensions were simultaneously measured. They were then subjected to separate moisture adsorption processes in seven RH conditions maintained inside the self-designed temperature conditioning chambers (Zhao *et al*. 2013) by saturated salt solutions. Temperature inside the chamber was kept at 30 ± 1 °C throughout the experiment.

At this temperature, RH conditions of 11, 22, 33, 45, 60, 75, and 92% were obtained by using saturated salt solutions of lithium chloride, potassium acetate, magnesium chloride, potassium carbonate, sodium bromide, sodium chloride, and potassium nitrate, respectively (Macromolecule Academy 1958).

During adsorption, specimens were taken out of the chamber, weighed, and then measured for transverse dimensions at certain time intervals, at which moisture content and swelling ratio were calculated based on oven-dry conditions. The weight measurement (± 0.1 mg) was conducted with the weighing bottles capped to prevent the specimens from exchanging moisture with the surrounding environment, and dimensional determination was conducted using a digital vernier caliper. The adsorption experiment used five replicates, and average values of dimensions and weights of specimens were taken as the final results.

Moisture-repellent effectiveness (MRE)

An evaluation method was applied from a previous study (Rowell and Banks 1985), in which the comparison of treated with untreated material was based on the change (in dimension or moisture absorbed) upon exposure to moisture for a defined time. Hygroscopic improvement is expressed as moisture-repellent effectiveness (MRE) (Eq. 2),

$$MRE = \frac{D_c - D_t}{D_c} \times 100\,(\%) \qquad (2)$$

where D_c is the swelling (or moisture adsorption) of the control during exposure in moisture for *t* min, and D_t the swelling (or moisture adsorption) of the treated specimen, also for *t* min.

Scanning electron microscopy (SEM)

The surface of tangential section morphologies of untreated and treated wood were observed by using SEM (Cambridge S-360). Samples were processed into slices 1 to 2 mm thick and 8 mm square, and double sided tape was used to fix them. The samples were sputter-coated with gold and vacuum-treated prior to observation.

Statistical Analysis

The significance of the differences between untreated and treated wood samples was evaluated by a computerized statistical program (OriginPro 8). The evaluation included analysis of variance (one way ANOVA) and subsequent Tukey tests at the 95% confidence level. Statistical evaluations were made on homogeneity groups, in which the groups of the same relative humidity and various PWE concentrations reflected statistical significance.

RESULTS AND DISCUSSION

Moisture Content and PWE Effectiveness

Equilibrium state: sorption isotherms

The EMC data for untreated and treated samples given in Fig. 1 exhibit the classical Type II sigmoidal sorption isotherm. With an increase in RH, there was a concurrent increase in EMC. There were differences between EMC values in control samples and treated wood samples, in which the treatment of three PWE concentrations resulted in a decrease in EMCs.

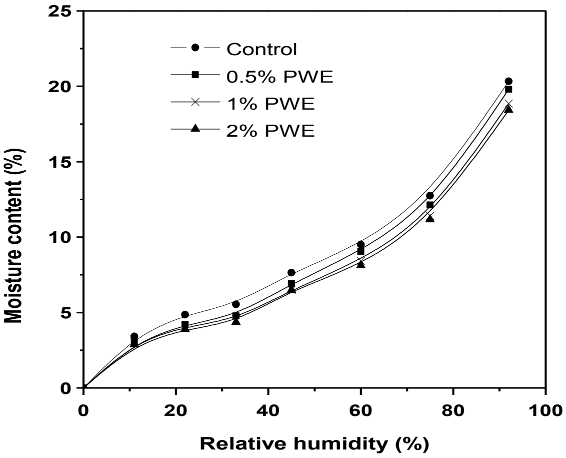

Fig. 1. Sorption isotherms of untreated and PWE treated wood at 30 °C

Moisture adsorption coefficient (Z)

According to Noack (1973), an analysis of the sorption and swelling of wood under conditions that exist during practical use showed that, instead of the maximum values of swelling, other physical characteristics are required for a distinct determination of the sorption and swelling behavior of wood in the region between 35% and 85% RH conditions. The mentioned RH range is similar to that in the practical applications. The slopes of sorption isotherms indicated the percentage change in moisture content. This is an indication of the hygroscopic sorption capabilities of the wood, namely the moisture adsorption coefficient Z. The moisture adsorption coefficient within the 45 to 75% RH range in the present study for untreated and the three treated wood groups were determined, and results are shown in Table 1.

Table 1. Moisture Expansion Coefficient (X), Humidity Expansion Coefficient (Y), and Moisture Adsorption Coefficient (Z) within 45 to 75% RH

Wood Group	**X_r**	**X_t**	**Y_t**	**Y_r**	**Z**
Control	0.2179	0.4043	0.0689	0.0365	0.1698
0.5 % PWE	0.2223	0.4140	0.0719	0.0387	0.1339
1 % PWE	0.2265	0.3776	0.0617	0.0370	0.1633
2% PWE	0.2328	0.4059	0.0685	0.0361	0.1560
	0.231[a]	0.246[a]	0.0680[b]	0.0390[b]	0.1800[b]

[a] Data of Chomcharn and Skaar (1983) in static condition for Basswood

[b] Data of Noack (1973) for 28 wood species

Table 1 suggests that the moisture adsorption coefficient of the control is similar to the mean values of 28 wood species from the study by Noack (1973). In addition, the Z values in PWE treated wood groups are lower than that in the control, which indicates the weakening in hygroscopic capacity of wood.

Moisture repellent effectiveness (MRE)

By applying Eq. 2 for PWE treated wood at the equilibrium state, the moisture repellent effectiveness is shown in Fig. 2. The results suggest that the MRE values decrease with an increase of RH. At a given RH condition, the optimal MRE belongs to 2% PWE treated wood.

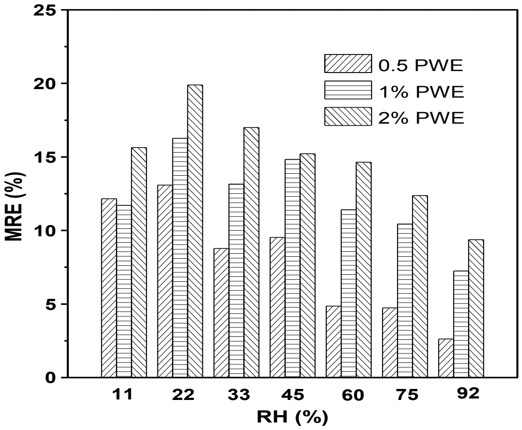

Fig. 2. Moisture repellent effectiveness of PWE treatments

Many factors influence the moisture adsorption properties of the treated wood, of which –OH accessibility is a significant component. Although, there are no chemical bonds formed between free-OH and PWE, the PWE present in the wood cell lumen and adhering to the wood cell wall also hinder moisture entry (Rowell and Banks 1985). Because the 2% PWE treatment produced the highest weight gain rate in the wood cell lumen compared with 1 or 0.5% PWE, the hindering ability of adsorbed moisture was more distinct.

The SEM analysis confirmed the appearance of PWE in wood after the treatment. After impregnation, the carrier solvent was allowed to evaporate, leaving the water-repellent substances deposited on the external and some internal surfaces of the treated wood. Figure 3b displays the envelope of cells evenly coated with a hydrophobic layer.

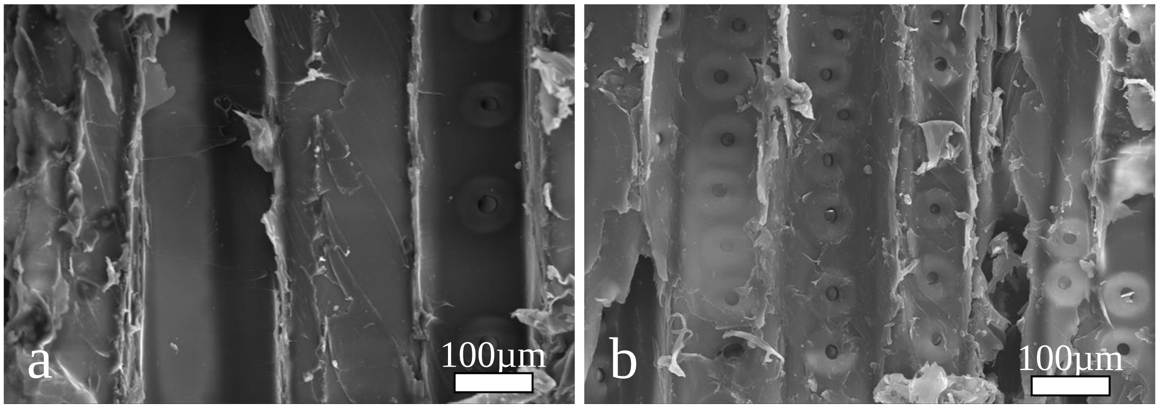

Fig. 3. SEM micrographs of (a) control and (b) 2% PWE treated wood

During adsorption processes

Along with adsorption, moisture increased slightly and finally reached equilibrium. The essential time to equilibrium state was 160 h for all groups.

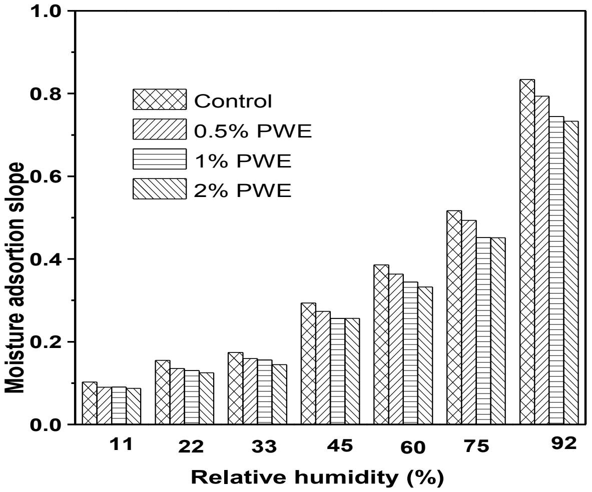

Fig. 4. The moisture adsorption slope in the first 24 h of untreated and treated wood at 30 °C

As shown in Fig. 5, moisture increased rapidly at the beginning of adsorption. The steep slope at the beginning stage indicated rapid adsorption. Because the samples in this stage were on the verge of oven-dry state, the surface and internal surface of specimens exhibited high energy. This phenomenon mainly took place in monomolecular adsorption. With increasing of adsorption rate, the desorption speed out-of-wood increased, so the net adsorption speed decreased, and this resulted in gradually reaching equilibrium state. The slope comparison of moisture adsorption at various RH in the first 24 h is given in Fig. 4 to evaluate the effect of PWE on the sorption rate of southern pine. It suggests that with an increase in relative humidity, the slope of moisture adsorption of wood samples increases. On the other hand, the slope values of PWE treated samples were less than that of control at any given RH condition, reconfirming the positive effect of PWE treatment.

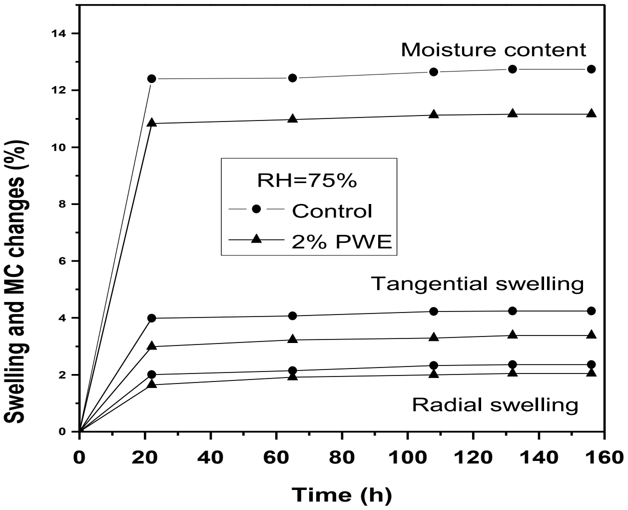

Fig. 5. Swelling and MC changes *versus* time

Dimensional Swelling and PWE Effectiveness

Dimensional swelling and transverse anisotropy

Table 2 lists lateral swelling as T/R ratios in various relative humidities at equilibrium state. These values increased when RH increased.

Table 2. Transverse Dimensional Swelling and Anisotropy at Seven Different Relative Humidities

RH (%)		Control	0.5% PWE	1% PWE	2% PWE
11	R	0.58 (0.13)	0.54 (0.11)	0.46 (0.06)	0.44 (0.09)
	T	0.72 (0.09)	0.67 (0.07)	0.55 (0.08)	0.51 (0.08)
	T/R	1.28 (0.16)	1.29 (0.38)	1.20 (0.19)	1.19 (0.30)
22	R	0.64 (0.05)	0.60 (0.15)	0.60 (0.11)	0.54 (0.08)
	T	0.94 (0.09)	0.74 (0.02)	0.74 (0.22)	0.67 (0.16)
	T/R	1.48 (0.27)	1.31 (0.39)	1.31 (0.51)	1.26 (0.37)
33	R	0.89 (0.23)	0.80 (0.16)	0.75 (0.17)	0.72 (0.07)
	T	1.49 (0.39)	1.09 (0.07)	0.92 (0.19)	0.89 (0.08)
	T/R	1.81 (0.78)	1.39 (0.25)	1.25 (0.25)	1.24 (0.07)
45	R	1.27 (0.16)	1.05 (0.11)	0.98 (0.04)	0.96 (0.17)
	T	2.18 (0.33)	1.51 (0.08)	1.43 (0.02)	1.32 (0.09)
	T/R	1.75 (0.40)	1.45 (0.17)	1.45 (0.04)	1.39 (0.19)
60	R	1.70 (0.04)	1.69 (0.09)	1.36 (0.14)	1.31 (0.06)
	T	2.90 (0.08)	2.63 (0.07)	2.15 (0.07)	1.98 (0.05)
	T/R	1.71 (0.05)	1.56 (0.09)	1.59 (0.19)	1.51 (0.08)
75	R	2.38 (0.08)	2.21 (0.03)	2.09 (0.05)	2.05 (0.36)
	T	4.24 (0.14)	3.67 (0.14)	3.28 (0.09)	3.22 (0.18)
	T/R	1.78 (0.07)	1.66 (0.05)	1.56 (0.07)	1.60 (0.22)
92	R	4.03 (0.55)	3.94 (0.27)	3.75 (0.37)	3.35 (0.36)
	T	7.45 (0.23)	6.90 (0.54)	6.17 (0.21)	5.70 (0.11)
	T/R	1.88 (0.33)	1.75 (0.12)	1.66 (0.17)	1.72 (0.20)

Data provided as the average of five specimens (Standard deviation)

For the treated wood, the lateral swelling was reduced, depending on the PWE degree in treated wood. Also, the lateral swelling in the control was greater than 0.5, 1, and 2% PWE treated wood at any given RH. The results indicate a positive effect of PWE in wood treatment, and here, the most obvious effect was in the case of the 2% PWE treated wood with the lowest swelling. The results also were confirmed by the statistical analysis in which standard deviation were evaluated. The dispersion of a set of data values were reduced in almost every instance of treated wood groups compared with the control group.

Table 2 also suggests that wood is anisotropic with respect to its transverse dimensional changes with various relative humidities corresponding to various moisture contents, as illustrated in Fig. 6. This is evidence that the T/R swelling ratio is not constant over the entire hygroscopic moisture range, in which the swelling measurement in a radial section is lower than in the tangential section. The results are in agreement with a previous study (Noak *et al*. 1973), indicating that in the region of fiber saturation, tangential dimensional changes of most wood species studied were relatively greater than radial changes compared with their behavior at lower moisture contents. The results also suggest a reduction in the T/R ratio of PWE treated wood.

Humidity expansion coefficient (Y)

As previously mentioned, the use condition (*i.e.*, environmental temperature and RH) of wood was in the range of 35 to 85% RH; Fig. 7 further displays maximum tangential swelling for 45, 60, 75, and 92% RH conditions for the control and the three treated wood groups, respectively. The slope, indicating the unit of dimensional swelling per change in unit of relative humidity, is one of the indexes to evaluate the dimensional stability of wood, namely the humidity expansion coefficient *Y*. Hence, *Y* values were determined for four wood groups in 45 to 75% RH and are shown in Table 1.

Similarly, the *Y* values in the control samples are close to the mean values of 28 wood species in a previous study (Noack *et al*. 1973), and these values for 1 and 2% PWE treated wood groups are lower compared with the control data, which indicates the improvement in dimensional stability of wood. The lowest *Y* belongs to 1% PWE of Y_t and 2% PWE of Y_r.

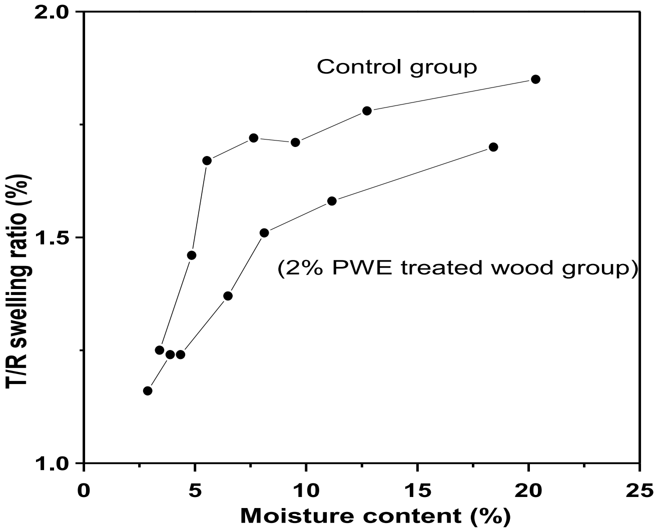

Fig. 6. T/R and MC relationship

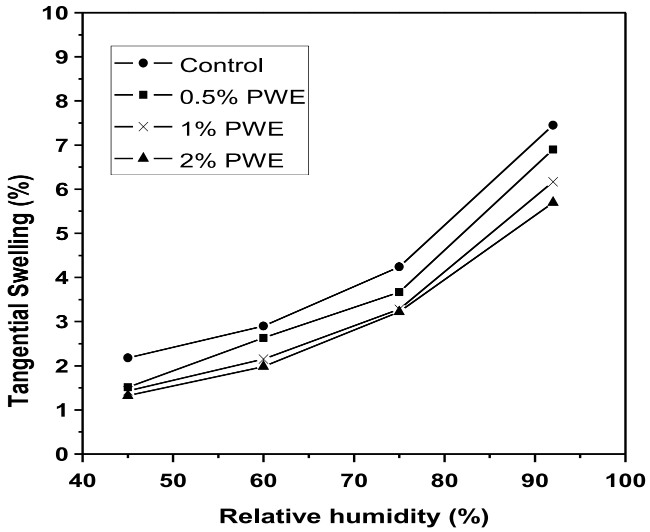

Fig. 7. Tangential swelling and RH relationship

The decreased swelling can be mainly attributed to the fact that hydrophobic substances after treatment with PWG resulted in smaller dimensional changes, thereby increasing the dimensional stability. Meanwhile, the coefficients of tangential swelling were greater than those of radial swelling for all specimens that reflect the anisotropy of wood.

Dimensional changes against time

Figure 5 presents lateral swelling against time for untreated wood and 2% PWE treated wood specimens, using the adsorption in 75% RH as an example. The tendency of the dimensional changes was similar to moisture increase in the adsorption process. For both the tangential and radial directions, the upper curve shows that untreated specimens quickly took up moisture and swelled to a maximum extent, while the lower shows a plot of treated wood specimens. The reduced rate of moisture adsorption indicates increased repellency, while the reduced extent of swelling indicates greater stability. Thus, PWE treatment seems effective in improving both water repelling capacity and dimensional stability.

Lateral swelling and moisture content relationship

Figure 8 further reveals the lateral swelling and moisture linear fitted relationship in control wood group during adsorption. Moisture content increased from about 3 to 20%, and swelling concurrently increased progressively for both control and treated wood. It was found that the R^2 values were greater than 0.99, indicating a good relationship between radial as well as tangential swelling and moisture content of the fiber saturation region. This agrees with previous equilibrium state studies (Steven 1963), and PWE treated wood specimens also satisfy the above-mentioned linear relationship.

Moisture expansion coefficient (X)

Like the *Y* and *Z* coefficients, the slopes of the curve in Fig. 8 indicate a unit of dimensional swelling per change in unit of moisture content; this was one index to compare the dimensional stability of wood, namely moisture expansion coefficient *X*. Hence, the moisture expansion coefficients were determined for four wood groups in 45 to 75% RH and are shown in Table 1. The table suggests that the moisture X_r of radial direction specimens is similar to that of basswood in the Chomcharn and Skaar (1983) study. It was found that PWE treatments also improved wood stability.

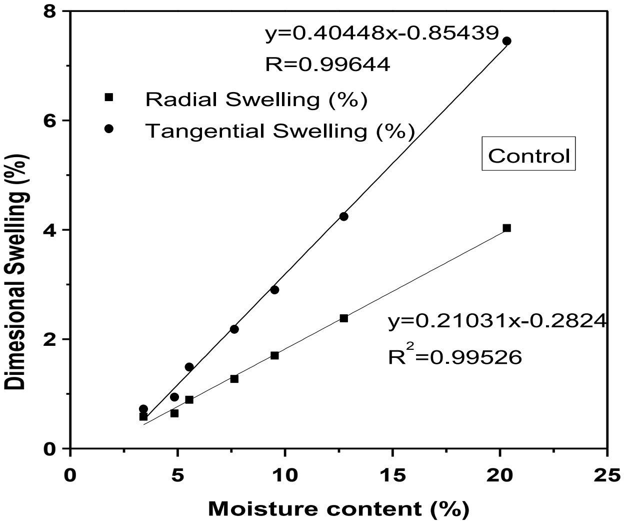

Fig. 8. Swelling and MC relationship

CONCLUSIONS

In this study, a self-designed temperature chamber was used to investigate the effect of paraffin wax emulsion (PWE) treatments on the moisture content and transverse swelling of southern pine exposed to several different relative humidities (RH_s) between 11 and 92% for adsorption at 30 °C. The results showed that:

1. The equilibrium moisture content (EMC) increased with increasing of RH and with a decrease in PWE concentration. The moisture adsorption coefficient (*Z*) exhibited its highest values in the case of control samples. The moisture repellent effectiveness varied from 3 to 20%, depending on RH values and degree treatments of PWE. The PWE treatments also decreased the moisture sorption rate of wood.
2. The dimensional swelling was anisotropic at every RH, and the lateral swelling decreased when PWE concentration increased. Additionally, the humidity expansion coefficients (*Y*) and moisture expansion coefficient (*X*) reconfirmed the role of PWE treatment in dimensional stability.

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