

Water Vapor Adsorption and Volumetric Swelling of Melt-Impregnated Wood–Polymer Composites

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Received 7 June 2005; accepted 26 December 2005

DOI 10.1002/app.24092

Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Wood–plastic composites were prepared through impregnation of solid wood with polyethylene. A resolution IV screening design of 16 runs for seven factors at two levels was adopted. The seven factors tested were ratio of maleated polyethylene in formulations, ratio of polyethylene of different molecular weights, four process factors (vacuum, pressure, time, and temperature), and wood species (red maple and aspen). Moisture adsorption content and volumetric changes as a function of time were investigated. This study also examined the effects of impregnation parameters and impregnants on water vapor adsorption and dimensional stability. The process parameters (pressure and temperature), polymer impregnants (polyethylene of different molecular

weights), and wood species contributed significantly to the equilibrium moisture content (EMC), whereas the moisture adsorption rate was mainly affected by the polymer impregnants (polyethylene of different molecular weights). The EMC was inversely proportional to polymer retention. However, none of the variables significantly contributed to volumetric swelling; the volumetric swelling rate was mainly affected by wood species, the molecular weight of the polyethylene, and impregnation vacuum. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 2668–2676, 2006

Key words: adsorption; composites; cooperative effects; polyethylene; swelling

INTRODUCTION

Wood is a porous and hydrophilic material. Heat treatment of wood permanently alters its physical and chemical properties.^{1–4} Heat treatment of wood darkens its color, reduces shrinkage and swelling, and lowers equilibrium moisture content in the same conditions. Very high temperature improves resistance to rot and also reduces susceptibility to fungal decay, but reduces its strength.^{5,6} The formation of wood–polymer composites (WPCs) by impregnating wood with vinyl monomers followed by *in situ* polymerization has received considerable attention in the last several decades.^{7–11} These WPCs generally exhibit enhanced strength properties and hardness, while displaying relatively poor dimensional stability in water because the monomers are mostly confined to the lumen. High-temperature melt impregnation by thermoplastic resins, on the other hand, is expected not only to enhance the mechanical properties, but also to improve dimensional stability and moisture adsorption behavior because of the degradation of celluloses

and hemicellulose and the reduced number of hydrogen bond sites.

In the first part of this study, resolution IV fractional factorial design was applied as the screening design for melt impregnation.¹² The variables investigated were: (1) chemicals used to treat wood, including maleated polyethylene and polyethylene of different molecular weights; (2) wood species; and (3) process factors (vacuum, pressure, temperature, and time). Polymer retention (PR) and hardness also were investigated.

WPCs prepared by impregnation have been shown to have reduced moisture adsorption and volumetric swelling rates.^{7–11} However, no studies have examined the time required for these kinds of WPCs to reach equilibrium in moisture content and volumetric swelling. The present study focused on moisture adsorption behavior and dimensional stability and determining the significance of the effects of the selected variables on these properties. Other goals were to determine the magnitude and direction of the most significant variables and to develop an appropriate strategy for future experimentation.

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Contract grant sponsor: Natural Sciences and Engineering Research Council of Canada (NSERC).

EXPERIMENTAL

Materials

Wood samples were chosen from defect-free boards of aspen and red maple supplied by a local wood

TABLE I
Raw Materials

Commercial name	Name	Contents
Epolene C-18	Maleated polyethylene	Acid number: 1.5–2.5 mg KOH; softening point: 98°C–106°C; viscosity: 2400–6000 CPS at 150°C
Epolene C-13	Polyethylene	M_w 76,000 g/mol, melt index (190°C) 200 g at 10 min with 2.16 kg, density 0.913 g/cm ³
Epolene C-15	Polyethylene	M_w 17,000 g/mol, melt index (190°C) 4200 g at 10 min with 2.16 kg, density 0.906 g/cm ³
B-215	Antioxidant	Mixture of 67% Irganos 168 and 33% Irganox 1010

company in Fredericton, New Brunswick, Canada. End-matched samples with dimensions of 55 × 40 × 6–7 mm (longitudinal × tangential × radial) were cut from lumber pieces in a pattern alternating treated with control samples. Maleated polyethylene C-18 and polyethylenes Epolene C-13 and Epolene C-15 were supplied by Eastman Chemical Canada Inc. (Mississauga/Ontario, Canada). Irganox B215 was kindly supplied by Ciba-Geigy Canada Ltd. (Mississauga, Ontario, Canada). The characteristics of these polymers are shown in Table I.

Preparation of wood-polymer composites by melt impregnation

The impregnation process scheme used is shown in Figure 1. A premixture of the polymer mixture with 0.1% antioxidant (B215) was transferred to the impregnation vessel and heated to the target temperature. The wood samples were oven-dried to a constant weight at 105°C for 24 h. Samples were then put in the oven at the required temperature for 20 min and transferred to the impregnation vessel. Impregnation conditions (parameters and impregnant formulations) are listed in Tables II and III. After impregnation, the samples were removed from the impregnation vessel, and excessive polymers were wiped off the sample surface. The control polyethylene samples (C-13, C-15, C-13 with 3.5% C-18, and C-15 with 3.5% C-18) were also cast to size 55 × 40 × 7 mm. Ten replicates were used for each treatment. For more detail, refer to Zhang et al.¹²

Water vapor adsorption and volumetric swelling

After treatment, the WPC and control specimens were oven dried at 105°C for 24 h. They were placed in a desiccator to cool to 21°C for the determination of weight and dimensions (length, width, and thickness). Then, all the samples were placed in a conditioning chamber at 65% relative humidity and a temperature of 21°C for 4 months. The weights and dimensions (length, width, and thickness) were repeatedly measured in the conditioning chamber during the 4 months.

The specimen's water vapor adsorption (M), also known as moisture adsorption content, was calculated as follows:

$$\% M_t = \frac{W_t - W_0}{W_0} \times 100 \quad (1)$$

where W_t is the weight of the sample after t days of storage in the conditioning chamber, and W_0 is the weight of the specimen after oven drying.

The volumetric swelling of the specimen (ΔV) was calculated as follows:

$$\% \Delta V = \frac{V_t - V_0}{V_0} \times 100 \quad (2)$$

where V_t is the volume of the sample after t days of storage in the conditioning chamber, and V_0 is the volume of the specimen after oven drying.

Data analysis

Analysis of covariance was used in this study to adjust the mean response for each run to eliminate the variability of end-matched untreated wood species samples. The adjusted response was used for further analyses.

The effect of the variable (x) on the response (E_x) was calculated as the difference between the resulting averages of the variable at the (+) and (–) levels as:

$$E_x = \frac{\sum Y(+)}{n} - \frac{\sum Y(-)}{n} \quad (3)$$

where $\sum Y(+)$ and $\sum Y(-)$ are the sums of the responses when x is at its highest (+1) and its lowest (–1) levels, respectively, and n is the number of times x is at the (+) or (–) level.

To determine the significance of the influences of various variables, a half-normal probability plot of effects was applied. First, the effects were ranked. From the

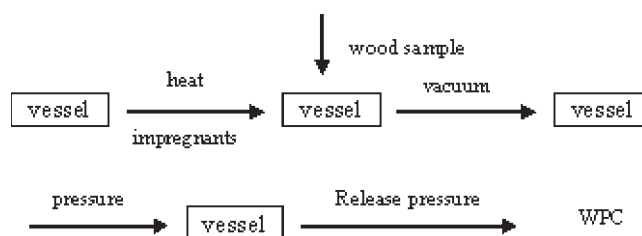


Figure 1 Scheme of impregnation procedure.

TABLE II
Levels of All Factors

Code	Variable	Code value	
		-1	+1
A	Epolene C-18/(Epolene C-15 + Epolene C-13) (wt %)	0.5	3.5
B	Epolene C-13/(Epolene C-15 + Epolene C-13) (wt %)	0	100
C	Time for maintaining vacuum of 30 mm Hg (min)	0	30
D	Pressure (psi)	0	100
E	Time for maintaining pressure (min)	30	90
F	Vessel temperature (°C)	140	165
G	Wood species	aspen	red maple

A, B, C, D, E, F, and G are the codes for the impregnation variables.

rank, the z value was calculated on the assumption that the estimates came from a normal distribution with a common mean. The half-normal plot of effects was plotted with absolute z values on the y axis and effects on the x axis. All the effects that lay along the line were negligible, whereas effects far from the straight line were significant. A multinomial linear model of variables with large effects with coded levels (-1 or +1) was used for predicting each response. Finally, a normal probability plot of the residual between the response and the prediction with the above model was adopted to check if all the points on the plot were reasonably close to a straight line, which would determine if the output regression model was reasonable and the assumptions of the analysis were justified.

RESULTS AND DISCUSSION

Water vapor adsorption

Water vapor adsorption of different treatments versus time

It was found that water vapor adsorption as a function of time followed the equation

TABLE III
Screening Design for Melt Impregnation

Run	A	B	C	D	E	F	G
1	1	-1	-1	-1	1	1	1
2	-1	-1	-1	1	-1	1	-1
3	1	1	1	1	1	1	-1
4	1	1	-1	1	-1	1	1
5	-1	1	1	-1	-1	1	1
6	1	1	1	-1	1	-1	1
7	1	-1	1	-1	-1	1	-1
8	1	-1	-1	1	1	-1	-1
9	1	-1	1	1	-1	-1	1
10	1	1	-1	-1	-1	-1	-1
11	-1	1	-1	-1	1	1	-1
12	-1	1	-1	1	1	-1	1
13	-1	-1	1	1	1	1	1
14	-1	1	1	1	-1	-1	-1
15	-1	-1	1	-1	1	-1	-1
16	-1	-1	-1	-1	-1	-1	1

A, B, C, D, E, F, and G are the variables in the screening design, shown in Table II.

$$M(t) = \alpha_M [1 - \exp(-\beta_M t)] \quad (4)$$

where α_M is the equilibrium moisture content (EMC, wt %), $M(t)$ is the moisture content at time t (wt %), t is the time (days), and β_M is a constant fitted parameter.

Eq. (4) describes $M(t)$ asymptotically approaching the EMC. The parameters α_M and β_M for different treatments and control samples listed in Table IV were obtained with the SAS nonlinear regression program. Eq. (4) was found to fit well for the treated and untreated samples. Table IV illustrates that runs 5 and 10 gave the highest EMC (~ 9 wt %), and runs 3 and 13 gave the lowest EMC (~ 5 wt %), which indicates that melt-impregnation treatments affect EMC. The moisture behavior of runs 3, 5, 10, and 13 and the controls are presented in Figure 2. Treatments retarded the time it took to reach the EMC compared with the untreated wood samples. Some treatments (such as run 3) took more than 3 months to reach the EMC and others took less. The β_M is a parameter that reflects

TABLE IV
Estimated Parameters of M for Different Treatments

Runs	Parameters in eq. (4)		Parameters in eq. (8)	
	α_M	β_M	α_V	β_V
1	6.48 (0.12)	0.068 (0.004)	3.61 (0.17)	0.061 (0.009)
2	6.25 (0.13)	0.060 (0.004)	4.51 (0.15)	0.077 (0.010)
3	5.55 (0.21)	0.030 (0.002)	4.00 (0.26)	0.032 (0.005)
4	7.53 (0.16)	0.041 (0.002)	3.81 (0.24)	0.035 (0.005)
5	8.79 (0.14)	0.049 (0.002)	4.44 (0.23)	0.037 (0.005)
6	8.45 (0.14)	0.054 (0.002)	4.24 (0.21)	0.044 (0.006)
7	6.02 (0.11)	0.078 (0.006)	3.94 (0.15)	0.078 (0.012)
8	5.90 (0.11)	0.085 (0.006)	4.21 (0.15)	0.078 (0.011)
9	6.84 (0.14)	0.053 (0.003)	3.88 (0.21)	0.042 (0.006)
10	9.11 (0.15)	0.047 (0.002)	4.25 (0.21)	0.044 (0.006)
11	6.14 (0.16)	0.042 (0.003)	3.54 (0.21)	0.043 (0.007)
12	7.27 (0.15)	0.047 (0.003)	3.80 (0.21)	0.043 (0.006)
13	5.01 (0.14)	0.052 (0.004)	2.54 (0.20)	0.047 (0.010)
14	5.84 (0.14)	0.049 (0.003)	3.39 (0.19)	0.049 (0.008)
15	6.31 (0.12)	0.069 (0.005)	3.05 (0.16)	0.064 (0.012)
16	7.75 (0.12)	0.063 (0.003)	3.91 (0.18)	0.053 (0.008)

Data in parentheses are standard deviations.

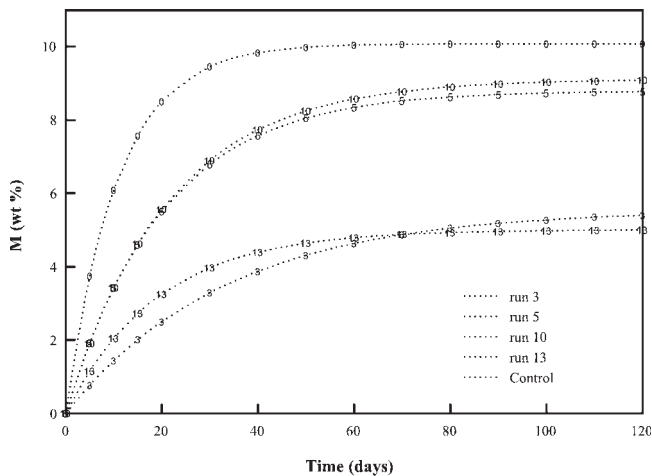


Figure 2 EMC as a function of time for different treatments.

the rate at which the samples absorb moisture. A smaller β_M implies a longer time to reach the EMC. Table IV clearly indicates that the treatments affected time required to reach the EMC because β_M varied with different treatments.

Day and Nelson¹³ developed a model describing the relationship between relative humidity (RH) and the EMC. Later, Simpson^{14,15} simplified Hailwood and Horrobin's model for the EMC of wood, which is a function of the RH and temperature. Chemical modification of solid wood retards the rate of vapor and liquid water adsorption, meaning the wood needs more time to reach the EMC.¹⁰ Gjerdrum¹⁶ found that adsorption of moisture by solid wood was affected by several factors: temperature, initial moisture content, time, and RH. Results from the present study are consistent with those obtained by Gjerdrum¹⁶.

Effects of variables on equilibrium moisture content

The effects of treatment factors on equilibrium moisture content (EMC) are described by eq. [3] and plotted in Figure 3. Figure 3 shows that the effects of different factors on the EMC varied in the following order: impregnation pressure (*D*) > polyethylene (*B*) > wood species (*G*) > impregnation temperature (*F*) > impregnation vacuum (*E*) > maleated polyethylene (*A*). It was found that the effects of impregnation pressure, polyethylene, wood species, and impregnation temperature were significant at the 0.05 level with half-normal plot analysis. The linear regression expression [eq. (5)] of the EMC against variables *D*, *B*, *G*, and *F* with an R^2 value of 0.77 is:

$$EMC(\%) = 6.79 + 0.67B - 0.77D - 0.50F + 0.60G \quad (5)$$

where *B*, *D*, *F*, and *G* are as defined in Table II, and their values are within the range bounded by the min-

imum (coded -1) and the maximum (coded +1). Diagnostic checks were applied to the Studentized residuals of the prediction with eq. (5) and EMC (α_M) in Table IV validated eq. (5). It can be seen that a higher impregnation pressure and temperature would reduce the EMC, whereas increasing the molecular weight of polyethylene and switching wood from aspen to red maple would increase the EMC.

Polyethylene is a nonpolar polymer and does not adsorb moisture. Even when polyethylene was blended with 3.5 wt % maleated polyethylene, the moisture adsorption from oven-dry condition to 21°C and 65% RH was still negligible (0.11 wt % for blend of 96.5 wt % polyethylene C-13 and 3.5 wt % maleated polyethylene C-18, 0.11 wt % for blends of 96.5 wt % polyethylene C-15 and 3.5 wt % maleated polyethylene C-18) compared to untreated wood (~ 10 wt %). Impregnation of wood with polyethylene lowered the adsorption of moisture for several reasons: (1) hydrophobic polyethylene occupied parts of the vessels and lumens to make the treated wood less hydrophilic, and (2) polyethylene could have blocked some hydrophilic groups (hydroxyl group, ester groups, ether group) on the wood-polymer interface to make these groups harder for water vapor to reach. Increasing impregnation pressure substantially increased the PR,¹² which resulted in the wood being less hydrophilic and decreased the EMC [Fig. 4(a)].

Higher-molecular-weight polyethylene has higher viscosity, resulting in lower PR. This led to a higher EMC for the impregnation with higher-molecular-weight polyethylene, as shown in Figure 4(b). For similar molecular structure impregnants, their impregnation permeability in wood was characterized by viscosity as described by Perng.^{17,18}

At high temperatures, the lignin and hemicellulose components changed and became more hydrophobic because of the destruction of hydrogen bond sites in these constituents. Impregnation at a higher tempera-

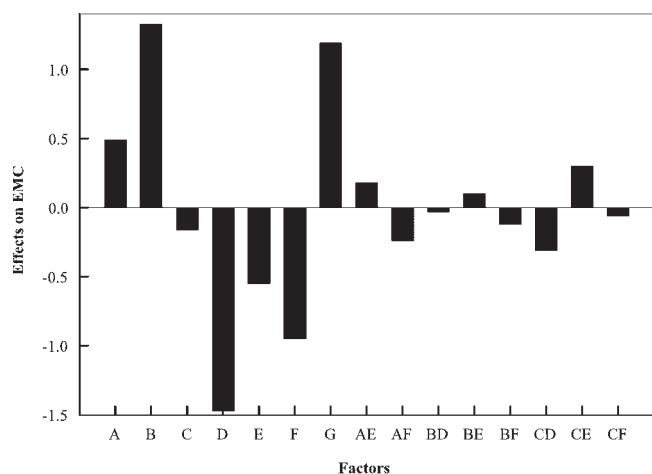


Figure 3 Effects of variables on EMC.

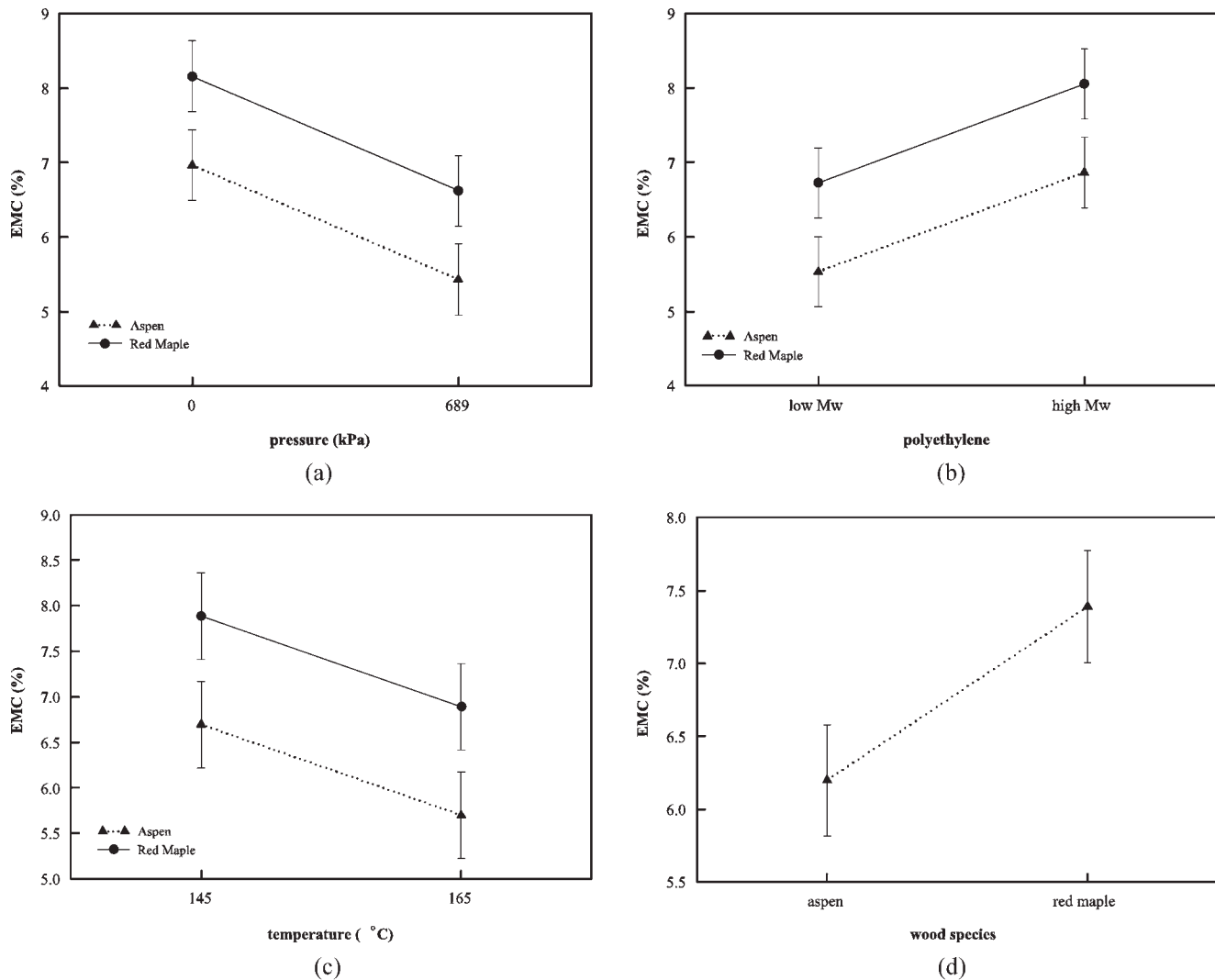


Figure 4 EMC as a function of (a) pressure; (b) polyethylene; (c) temperature; and (d) wood species (● red maple, ▲ aspen).

ture also resulted in a higher PR. The combination of these two factors resulted in a lower EMC for high-temperature impregnation [Fig. 4(c)]. Pétrissans et al.⁴ studied the wettability of thermally treated wood and found that heat-treated wood of all species tested (spruce, poplar, beech, and pine) became more hydrophobic and showed decreased wettability. Tjeerdsma et al.⁵ indicated that the hygroscopicity of thermally treated wood depended on the processing time and temperature of the treatment.

Aspen has a lower wood density ($\sim 460 \text{ kg/m}^3$) than red maple ($\sim 610 \text{ kg/m}^3$). Aspen and red maple are both hardwoods and therefore have vessels in their wood. Aspen has a higher void volume than red maple and thus has a greater PR for a given impregnation procedure. At the same impregnation conditions, PR for aspen (50 wt %) was higher than that for red maple (26 wt %). Thus, the treated aspen had a lower EMC than the treated red maple [Fig. 4(d)].

Relationship between PR and EMC

Figure 5 presents the EMC as a function of PR in this study. It was found that the EMC was strongly and negatively related to PR, with an R^2 value of 0.93, as shown in eq. (6).

$$\text{EMC}(\%) = 9.3706 - 0.0687\text{PR} \quad (6)$$

where PR is polymer retention (wt %) and EMC is the equilibrium moisture content (wt %).

Effects of treatment variables on β_M

Table IV and Figure 2 clearly show that the WPCs with different treatments required different periods to reach the EMC. The effects of different variables on β_M calculated by eq. (4) are presented in Figure 6. Polyethylene (B) had the largest effect on β_M , and only

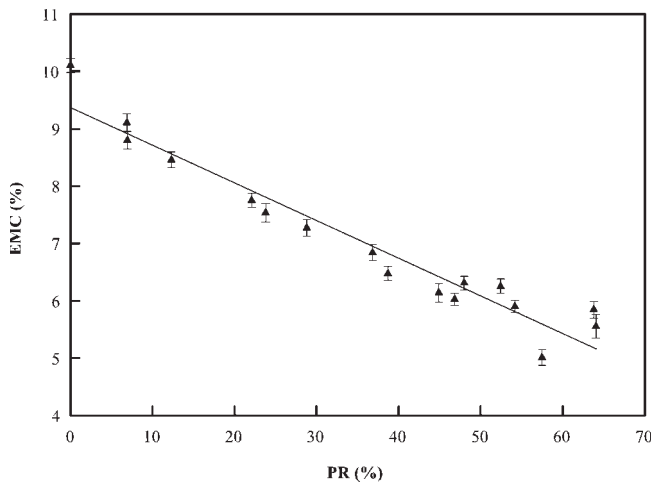


Figure 5 EMC as a function of PR.

polyethylene was determined to be significant at the 0.05 level with the half-normal plot analysis. The linear regression expression of β_M versus B had an R^2 value of 0.58:

$$\beta_M = 0.0055 - 0.0011B \quad (7)$$

where B is polyethylene, defined in Table II, and its values are within the range bounded by the minimum (coded -1) and the maximum (coded $+1$).

The small R^2 (0.58) suggests that β_M was affected by not only polyethylene (B), but also other variables such as maleated polyethylene (A), impregnation pressure (D), impregnation temperature (F), and several two-way interactions. However, polyethylene was the only variable that significantly affected the β_M at the 0.05 level. Nevertheless, there is a 76% probability that the change in β_M was accounted for by polyethylene.

Several previous studies on the moisture permeability of polyethylene¹⁹⁻²¹ found that density, film thick-

ness, crystalline orientation, crystalline content, and molecular weight affected the water vapor transmission rate. The present study has clearly shown that the molecular weight of the polyethylene affected the moisture adsorption rate. It was found that an increase in molecular weight reduced the value of β_M . (Fig. 7).

Volumetric swelling behavior

Volumetric swelling of different treatments versus time

It was found that volumetric swelling as a function of time followed the equation:

$$\Delta V_t = \alpha_V [1 - \exp(-\beta_V t)] \quad (8)$$

where ΔV_t is the volumetric swelling of the samples at time t (%), α_V is the equilibrium volumetric swelling (%), β_V is the swelling rate parameter (day^{-1}), and t is time (day).

Eq. (8) was found to fit the volumetric swelling data of different treatments well. The parameters α_V and β_V for different treatments were obtained with the SAS nonlinear regression program and are listed in Table IV. The values of α_V and β_V indicated different treatments had differing effects on these parameters.

Below the fiber saturation point, the rate of shrinkage of solid wood depended on the wood species and environmental conditions. For different wood species, there was a roughly linear relationship between shrinkage and moisture content.²² Meijier and Militz²³ found that the rate of shrinkage or swelling of treated wood was directly proportional to moisture adsorption and desorption and adopted an asymptotic regression model based on earlier studies.^{24,25} The results of our study are consistent with those of Meijier and Militz.²³

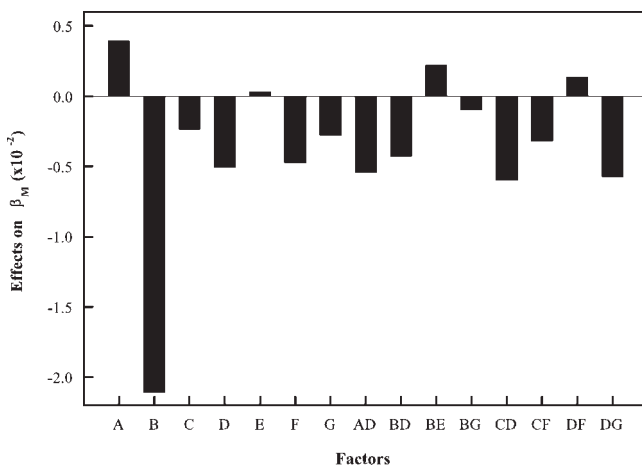


Figure 6 Effects of variables on β_M .

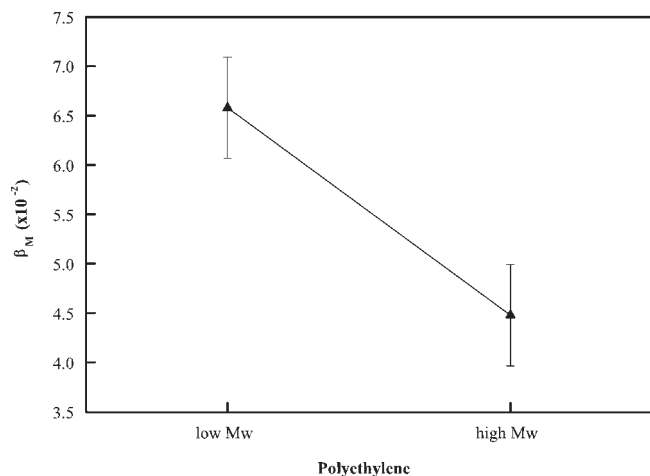


Figure 7 Effects of significant variables on β_M .

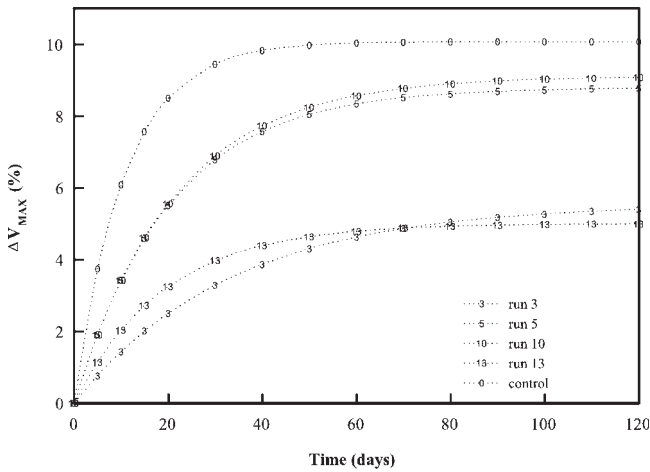


Figure 8 Volumetric swelling as a function of time.

The ΔV_t values of runs 3, 5, 10, and 13 and the control as a function of time are presented in Figure 8. Treatments retarded the time for composites to reach the equilibrium volumetric swelling compared with untreated wood samples. Some treatments (such as run 3) took more than 3 months to approach α_V , but others took less time. The lower the β_V values, the longer the composites would need to approach α_V .

Effects of treatment variables on α_V

The parameter in eq. (8) dictates the final volumetric swelling of a material from oven-dry conditions to 21°C and 65% RH. Figure 9 illustrates the effects of various factors on α_V and shows that impregnation time (*E*), the interaction of maleated polyethylene and impregnation time, the interaction of polyethylene and wood species, and the interaction of maleated polyethylene and impregnation vacuum had the highest effects on α_V . However, no variables significantly affected α_V at the 0.05 level as determined by the half-normal plot analysis.

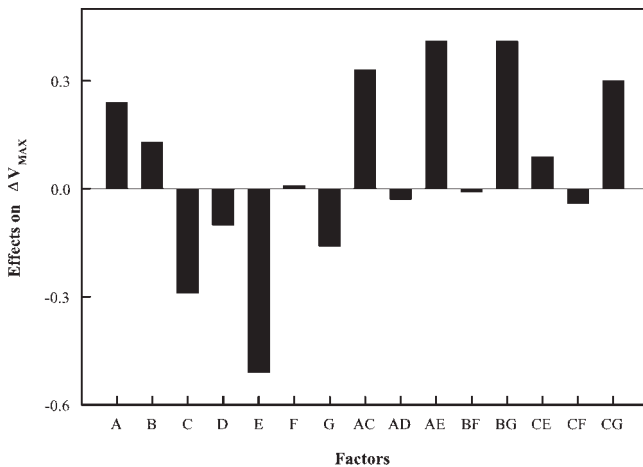


Figure 9 Effects of variables on ΔV_{max} .

This indicates that volumetric swelling was different from moisture adsorption and was the result of a combination of several variables and/or two-way interactions, such as impregnation parameters (time, vacuum), maleated polyethylene, and several two-way interactions of impregnation variables.

Effects of variables on β_V

The parameter β_V in eq. (8) is a measure of how fast a material reached the final volumetric swelling value. Figure 8 clearly shows that the time it took for treated woods to reach the α_V varied. The β_V was inversely proportional to the approach time of α_V (Table IV). The effects of different variables on β_V are presented in Figure 10. The order of the absolute values of the variables were: polyethylene (*B*) > wood species (*G*) > impregnation vacuum (*C*) > interaction of polyethylene (*B*) and wood species (*G*). It was found that the molecular weight of the polyethylene, wood species, and time under vacuum to 30 mmHg were the significant variables at the 0.05 level under the half-normal plot analysis. The linear regression expression of β_V versus the significant factors, with an R^2 value of 0.78, can be shown as

$$\beta_V = 0.051 - 0.011B - 0.0039C - 0.0068G \quad (9)$$

where *B*, *C*, and *G* are as defined in Table II and are within the range bounded by the minimum (coded -1) and the maximum (coded +1).

Below the moisture saturation point, absorbed moisture swells wood. As discussed earlier, the density, film thickness, crystalline orientation, crystalline content, and molecular weight of polyethylene film all affect the water vapor transmission rate.¹⁹⁻²¹ Lowering the moisture vapor transmission rate resulted in retardation of the moisture adsorption and then of the volumetric swelling of treated wood compared to

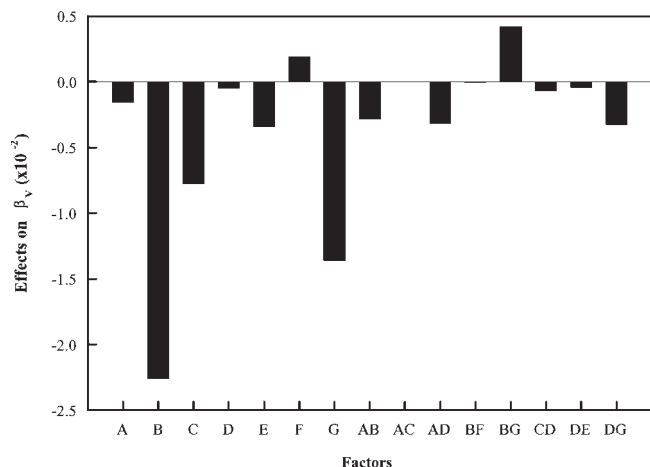


Figure 10 Effects of variables on β_V .

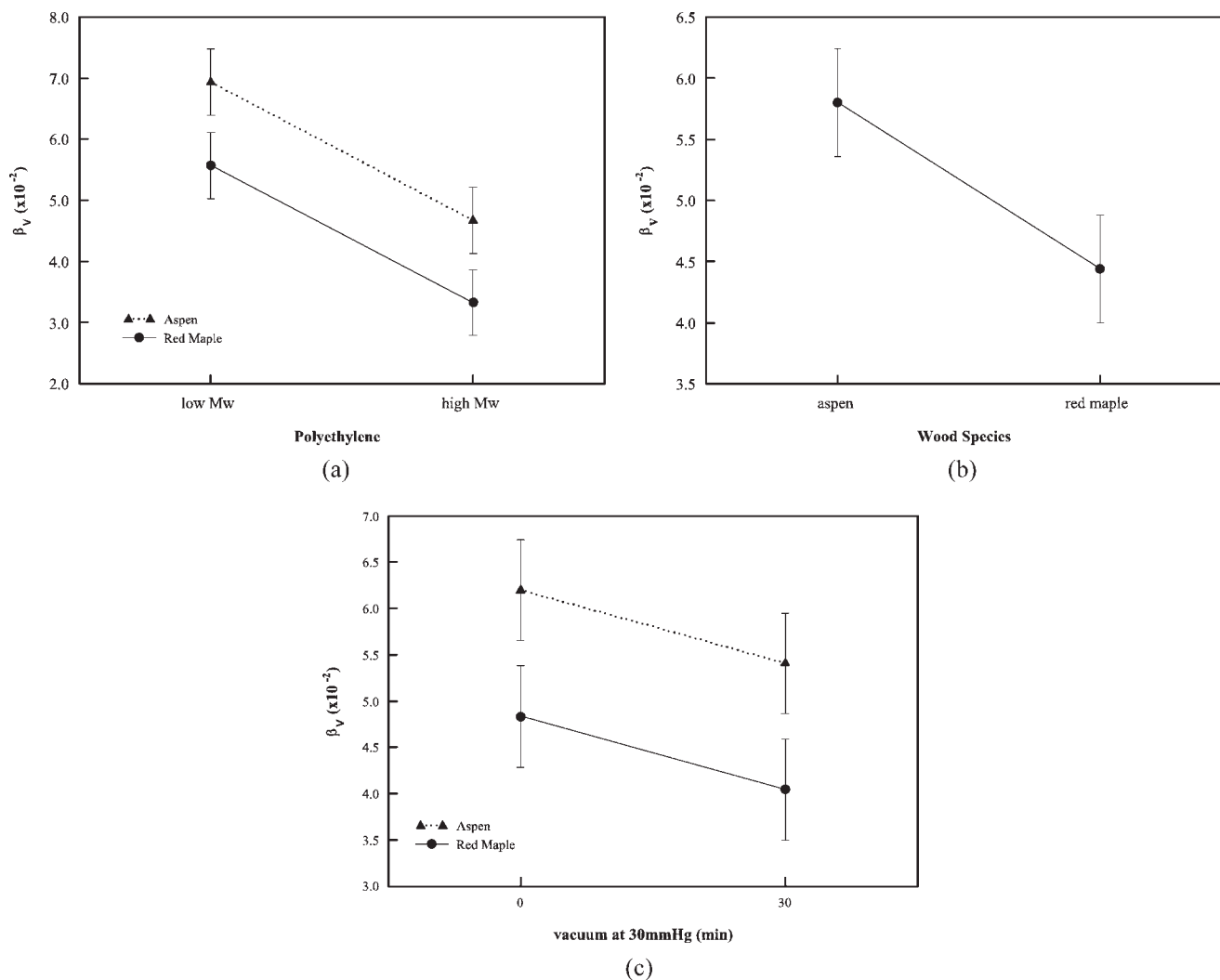


Figure 11 Effects of significant variables on β_V of (a) polyethylene; (b) wood species and (c) vacuum (● red maple, ▲ aspen).

untreated wood. Figure 7 shows that increase in the molecular weight of polyethylene (from 17,000 to 76,000 g/mol) reduced the moisture adsorption rate. Consequently, the volumetric swelling rate was also reduced, as shown in Figure 11(a).

High-temperature drying has been found to change the structure of wood and to degrade hemicellulose, and high-temperature treatment can markedly change the chemical constituents, particularly hemicellulose,²⁶ which could result in increased hydrophobicity of wood after treatment, and therefore the retarding of moisture adsorption. Aspen is generally composed of 53% cellulose, 31% hemicelluloses and 16% lignin,²⁷ but red maple is composed of 41% cellulose, 35% hemicelluloses, and 24% lignin.²⁷ Thus, red maple has a higher hemicellulose content than aspen, and the high-temperature treatment partly degraded the hemicellulose of woods. This may explain why red maple had a lower β_V than aspen [Fig. 11(b)].

Rizvi et al.²⁸ investigated the thermal performance of wood-flour in the temperature range from 100°C to

225°C and found that under 200°C, the thermal degradation of the main components of the wood (cellulose, hemicellulose, and lignin) was negligible and that the volatile emissions were from extractives consisting of a number of resins, waxes, and tannins. Therefore, melt impregnation at high vacuum and high temperature would accelerate the emission of volatiles from the wood and redistribute them in the wood, which would alter the moisture adsorption paths of wood and further affect the swelling rate of wood, as shown in Figure 11(c).

CONCLUSIONS

Water vapor adsorption (M) and volumetric swelling (ΔV) of wood-polymer composites and untreated woods as a function of time asymptotically reached the equilibrium state and could be well described by exponential equations [eqs. (4) and (8)]. The equilibrium moisture content (EMC), maximum volumetric swell-

ling (α_V), moisture adsorption rate (β_M), and volumetric swelling rate (β_V) were determined by the impregnants, wood species, and impregnation conditions.

An experimental design approach allowed us to determine the most significant variables of the melt impregnation process for EMC, α_V , β_M , and β_V . The process parameters (pressure and temperature), polymer impregnants (polyethylene of different molecular weights), and wood species affected the EMC significantly, whereas β_M was mainly affected by the polymer impregnants (polyethylene of different molecular weights). The EMC was inversely proportional to polymer retention. Although no variables influenced the volumetric swelling significantly, β_V was mainly affected by wood species, polyethylenes of different molecular weights, and impregnation vacuum.

Further studies are needed, and the following experimental designs should be considered in future studies:

1. A full factorial design comprising the identified significant variables;
2. The use of two or three levels of important factors in order to determine the optimized process parameters for EMC, β_M , α_V , and β_V .

The authors thank Dr. Hui Wan of Forintek Canada Corp. (FCC) for useful discussion about the project, Dr. Patrick J. Whitcomb of Stat-Ease, Inc. for assistance with the experiment design, and Ms. Sylvia Bayliss of Eastman Chemical Canada Inc. for supplying maleated polyethylene and polyethylene. The assistance of Ms. Francine Cote and Ms. Marie-Claude Giguere of FCC in the laboratory work is much appreciated.

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