

Wood Plastic Composites by Melt Impregnation: Polymer Retention and Hardness

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ABSTRACT: Wood plastic composites were prepared through impregnation of solid wood with polyethylene. The effects of impregnation parameters on polymer retention and hardness were investigated. A screening strategy of 16-run resolution IV design for seven factors at two levels was adopted. The seven factors were: ratio of maleated polyethylene in formulations, ratio of polyethylenes with different molecular weights, four process factors (vacuum, pressure, time, and temperature), and wood species (red maple and aspen). Polymer retention (PR) and Brinell hardness (H_B) were investigated and discussed on the basis of the impregnation parameters. The present work showed that process parameters (pressure and temperature), polymer

impregnants (different molecular weight polyethylenes), and wood species contributed significantly to PR and H_B . Increasing pressure and temperature resulted in a higher PR and H_B , whereas increasing the molecular weight of polyethylene and switching wood species from aspen to red maple gave a lower PR and H_B . This study was aimed at understanding how impregnation parameters affect the final properties of wood plastic composites and developing an optimal fabrication process for wood plastic composites. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 1672–1680, 2006

Key words: wood plastic composite; impregnation; screening design; hardness; polymer retention

INTRODUCTION

The shortage of high quality hardwoods has driven researchers and wood product manufacturers to seek alternatives, lower cost resources for value-added applications. To reach this goal, proper technologies are needed to improve specific wood quality attributes (e.g., dimensional stability, durability, mechanical properties, and hardness) to meet end-use requirements. One approach is to combine wood with polymeric materials to create a new composite. There are two categories of wood plastic composites (WPC). One is prepared by impregnating solid wood with a monomer or prepolymer and then *in situ* polymerization.^{1–5} The other is plastics reinforced with wood fiber or particles. Although the former produces stronger products than wood, the conversion rate of polymerization hardly reaches 100%, and the residue monomers or prepolymers tend to leach from the product and have a negative impact on the environment. Plastics reinforced with wood fiber have poor dimensional stability even though they are less expen-

sive. These technological issues limit the acceptance of WPC by consumers.

On the other hand, plastic waste disposal has been recognized worldwide as an environmental problem. Recycled plastics are readily available almost everywhere. If one can develop new technologies for the cost-effective utilization of waste plastics with solid wood, it could solve the aforementioned problems of lack of quality wood and plastic waste disposal.

Singh et al.⁶ and Siau et al.⁷ found that impregnability of chemicals into wood differs significantly, depending on the type of chemical and species of wood. Some chemicals can fill the empty lumens in wood, whereas others may be able to penetrate into the cell walls or react with the wood material. Costanza and Miyara⁸ simulated the wood impregnation with vacuum and pressure procedure, and validated that the impregnation process had a significant impact on chemical loading. Perng^{9,10} found that the permeability of an impregnant in wood is related to its viscosity, and is dependent on wood species. However, up to now, no study has been undertaken to systematically quantify the permeability of melt thermoplastics into major commercial woods in eastern Canada. Such a study will help to assess the feasibility of developing high-performance WPC by impregnating solid wood with thermoplastics.

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TABLE I
Materials Used

Commercial name	Name	Contents
Epolene C-18	Maleated polyethylene	Acid number: 1.5–2.5 mg-KOH, softening point: 98–106°C; viscosity: 2400–6000 CPS at 150°C
Epolene C-13	Polyethylene	MW 76,000 g mol ⁻¹ , melt index (190°C) 200 g at 10 min with 2.16 kg, density 0.913 g cm ⁻³
Epolene C-15	Polyethylene	MW 17,000 g mol ⁻¹ , melt index (190°C) 4200 g at 10 min with 2.16 kg, density 0.906 g cm ⁻³

Impregnation of wood with melted chemicals at high temperature presents a number of technical challenges. Generally speaking, thermoplastics are processed at high temperatures, such as polystyrene at around 200°C, polyethylene terephthalate at around 260°C, and polypropylene at around 200°C. Cellulose is slowly broken down through gradual degradation, decomposition and charring on heating at temperatures up to 200°C. Above 200°C, cellulose undergoes rapid decomposition.¹¹ To minimize thermal degradation of wood, impregnation with plastics should ideally be carried out below 200°C.

The heat treatment of wood alters its physical and chemical properties permanently. It reduces shrinkage and swelling, and lowers the equilibrium moisture content of the wood.^{12–15} With high temperature melt impregnation of solid wood by thermoplastic resins, it is expected to not only enhance the mechanical properties but also improve dimensional stability. To evaluate the suitability of any potential impregnant for producing WPC, it is necessary to quantify the permeability behavior of the polymers into woods and understand how the various quality attributes and performances of WPC are affected by the impregnation process parameters, impregnant and wood species.

Early studies on the development of the solid wood polymer composite process were empirical in nature. The commonly used approach was to employ response surface methodology, though handling more than six variables was difficult. It is more efficient to have a screening experiment that will help to yield information about the influences of the variables so that the main variables can be reduced to three or four. A full factorial design can be used for screening. However, the number of experiments that must be performed increases rapidly with the number of variables. There are two common designs for fractional two-level designs: (1) standard “ 2^{k-p} ”s, where k refers to the number of factors and p to the fraction, and (2) Plackett-Burman (PB) design. Anderson and Whitcomb¹⁶ found that PB design could miss the main effects if interaction exists. Therefore, the resolution IV fractional factorial design was adopted in this study.

The main objectives of this study are to identify the most significant variables during the impregnation

process that influence the permeability of wood (polymer retention) and hardness, and to develop an appropriate strategy for future experimentation.

MATERIALS AND METHODS

Materials

Wood samples were cut from defect-free boards of aspen and red maple supplied by a local supplier in New Brunswick, Canada. End-matched samples with dimensions of 55 mm × 40 mm × 6–7 mm (longitudinal × tangential × radial) were obtained in an alternating pattern of treated and control samples. Maleated polyethylene C-18, polyethylene Epolene C-13, and Epolene C-15 (Table I) were supplied by Eastman Chemical Company.

Experimental design

Screening design refers to an experimental plan with the primary purpose of identifying which of the variables of interest have important effects. Screening designs are also called main effects designs. In this study, several variables were studied: (1) the chemicals used to treat wood: maleated polyethylene (Epolene C-18), and polyethylenes with different molecular weights (Epolene C-13 and Epolene C-15); (2) the wood species: aspen and red maple; and (3) the processing parameters: vacuum, pressure, temperature and time. On the basis of Whitecomb's¹⁷ recommendations, two ratios were used to represent the three mixture components in the process space, namely (Epolene C-18)/(Epolene C-13 + Epolene C-15) and (Epolene C-13)/(Epolene C-13 + Epolene C-15). Throughout this article, the low level for each variable is designated as -1 and the high level as $+1$. The combinations of these variables used, along with their levels, are shown in Table II. Resolution IV screening design for seven factors having 16 runs was adopted in this study (Table III).

Impregnation procedure

A selected polymer mixture was premixed inside an impregnation vessel at a set temperature. A 0.1% an-

TABLE II
Variables Used for Screening Impregnation Parameters on Hardness and Polymer Retention

Variable designation	Variable	Levels	
		-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 maintaining vacuum at 30 mm Hg (min)	0	30
D	Pressure (kPa)	0	689
E	Time maintaining pressure (min)	30	90
F	Vessel temperature (°C)	140	165
G	Wood species	Aspen	Red maple

tioxidant (B215), mixture of 67% IRGAFOS 168 and 33% IRGANOX 1010, supplied by Ciba-Geigy Canada, Mississauga, Ontario, was added to each mixture to prevent polyethylene oxidization during impregnation. The impregnation process is shown schematically in Figure 1. The aspen and red maple samples were oven-dried to a constant weight at 105°C for 24 h. After drying, the samples were placed in a laboratory oven with a sandwich structure steel lattice trellis and were preheated for 20 min at the same temperature as the experiment run before they were transferred to an impregnation vessel together with the steel lattice trellis. There was no contact between specimens, and a weight was applied on the steel lattice trellis so that no sample flotation occurred during impregnation. Then, a vacuum was drawn for the required time. Because high pressure nitrogen was not available at the laboratory, compressed air was used instead. For safety reasons, the pressure vessel was placed in a fume hood with an antiexplosive window. Subsequently, compressed air was applied to the system at the conditions shown in Table III. After impregnation, the

samples were taken out of the vessel and excess polymers were wiped off the sample surface. All data on weight and dimensions of wood samples were recorded before and after impregnation. A minimum of 10 specimens were used for each treatment.

The polymer retention (PR) of the treated specimens was calculated as follows:

$$\% \text{ PR} = \frac{D_{\text{WPC-dry}} - D_{\text{wood-dry}}}{D_{\text{wood-dry}}} \times 100 \quad (1)$$

where $D_{\text{WPC-dry}}$ and $D_{\text{wood-dry}}$ are oven-dry densities of WPC and wood, respectively.

Hardness tests

The hardness tests were performed on untreated and treated samples using a universal test machine. All specimens were stored in a conditioning chamber at 21°C and 65% relative humidity for 4 months to equilibrate their moisture contents. During the test, the

TABLE III
Design Matrix for and Response of Screening Impregnation Parameters on Polymer Retention and Hardness

Exp. conditions	Design matrix							Polymer retention PR (wt %)	Hardness H_B (N/mm ²)
	A	B	C	D	E	F	G		
1	1	-1	-1	-1	1	1	1	39.04 (3.42)	22.98 (2.21)
2	-1	-1	-1	1	-1	1	-1	52.72 (3.63)	15.75 (0.98)
3	1	1	1	1	1	1	-1	63.90 (3.43)	16.71 (0.72)
4	1	1	-1	1	-1	1	1	23.63 (2.4)	20.87 (1.36)
5	-1	1	1	-1	-1	1	1	6.85 (2.19)	17.97 (1.56)
6	1	1	1	-1	1	-1	1	12.81 (2.93)	19.20 (1.35)
7	1	-1	1	-1	-1	1	-1	46.63 (4.15)	15.30 (0.81)
8	1	-1	-1	1	1	-1	-1	54.23 (3.13)	15.44 (0.78)
9	1	-1	1	1	-1	-1	1	36.56 (4.26)	21.91 (1.96)
10	1	1	-1	-1	-1	-1	-1	6.89 (1.05)	11.26 (0.84)
11	-1	1	-1	-1	1	1	-1	44.91 (2.36)	13.93 (1.20)
12	-1	1	-1	1	1	-1	1	28.20 (2.80)	21.41 (2.08)
13	-1	-1	1	1	1	1	1	57.99 (5.36)	25.38 (2.05)
14	-1	1	1	1	-1	-1	-1	64.06 (2.86)	15.65 (0.75)
15	-1	-1	1	-1	1	-1	-1	48.02 (4.3)	15.18 (0.86)
16	-1	-1	-1	-1	-1	-1	1	22.59 (2.76)	20.47 (2.06)

A, B, C, D, E, F, and G and their levels are defined in Table II. Values in parentheses indicate standard deviations.

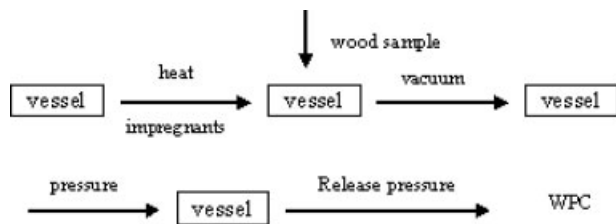


Figure 1 Impregnation scheme.

indenter (an 11.3-mm diameter steel ball), which was attached to the loading platen of the test machine, was lowered to the surface of the test specimen. A preload of 1–2 N was applied to stabilize the test specimen. The applied load was then increased at such a rate as to reach a target load of 1000 N in 15 s, and was maintained at this force for 25 s. The actual contact area under indentation was used to calculate the hardness of the specimen. The load-deformation data were collected at a sampling rate of 10 data points per second. Brinell hardness was calculated using eq. (2) shown below. At least 10 specimens were tested for each chemical formulation and untreated group.

$$H_B = \frac{F}{\pi D h} \quad (2)$$

where H_B is the Brinell hardness (N/mm²); F , the maximum applied force (N); D , the diameter of the steel ball (mm); and h , the indentation depth (mm).

Scanning electron microscopy

Scanning electron microscopy (SEM) was used to examine the impregnated and control wood samples. The samples were soaked in deionized water for several days. A fresh surface of each sample was then obtained by cutting with a microtome and the samples were allowed to dry. The surfaces of the samples were coated with palladium and gold, and then were examined with SEM under various magnifications.

Statistical analysis

Analysis of covariance was applied in this study to adjust the mean of response for each treatment to eliminate the influence of wood density or physical properties on the test results. The adjusted response was used for further analyses.

The effect (E) of a variable x on the response is calculated as the difference between the averages resulting from the (+) and (–) levels of the variable.¹⁸

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

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

To determine the significance of the influence of the various variables, the half-normal probability plot of effects was prepared. First, the effects were ranked. From the rank, the z -value was calculated based on the assumption that the estimates came from a normal distribution with a common mean. The half-normal plot of effects was obtained with the absolute variable z on the y -axis and the effect on the x -axis. The effects that lie along the line are negligible, whereas those located away from the line are significant. A multinomial linear model of factors with the large effects at the coded level (–1 or +1) was used for the prediction of each response. After that, a normal probability plot of the residual between response and the prediction with the abovementioned model was used to check if all the points on the plot lie reasonably close to a straight line, which determined if the output regression model was reasonable and if the assumptions of the analysis were satisfied.

RESULTS AND DISCUSSION

SEM analysis

In the cellular structure of both hardwood species treated in this study, there are vessels and lumen available for chemical filling. SEM observation (Fig. 2) shows considerable differences between untreated specimens of the two species. The diameters of vessels and lumen of aspen are bigger than for red maple. Aspen has more voids available than red maple does. If the impregnant mainly enters the cell wall, it should swell the wood and fill the vessels and lumen. According to Figures 2(c) and 2(d), the impregnation of polyethylene into red maple and aspen mainly occurred by filling the vessels and lumen. No evidence of cell wall penetration was detected.

Polymer retention

PR calculated with eq. (1) is also presented in Table III. Run numbers 3 and 14 gave the highest values (64%), whereas numbers 5 and 10 gave the lowest values (7%). This indicates that different combinations of factors had definite impacts on PR. The effects of the studied variables on PR, alone or in combination, were calculated using eq. (3). The results are summarized in Figure 3, which shows that the selected factors had different impacts on PR. The important effects, in decreasing order of influence, were wood species (G), impregnation pressure (D), ratio of impregnant molecular weight (B), impregnation temperature (F), and ratio of maleated polyethylene in the impregnant (A).

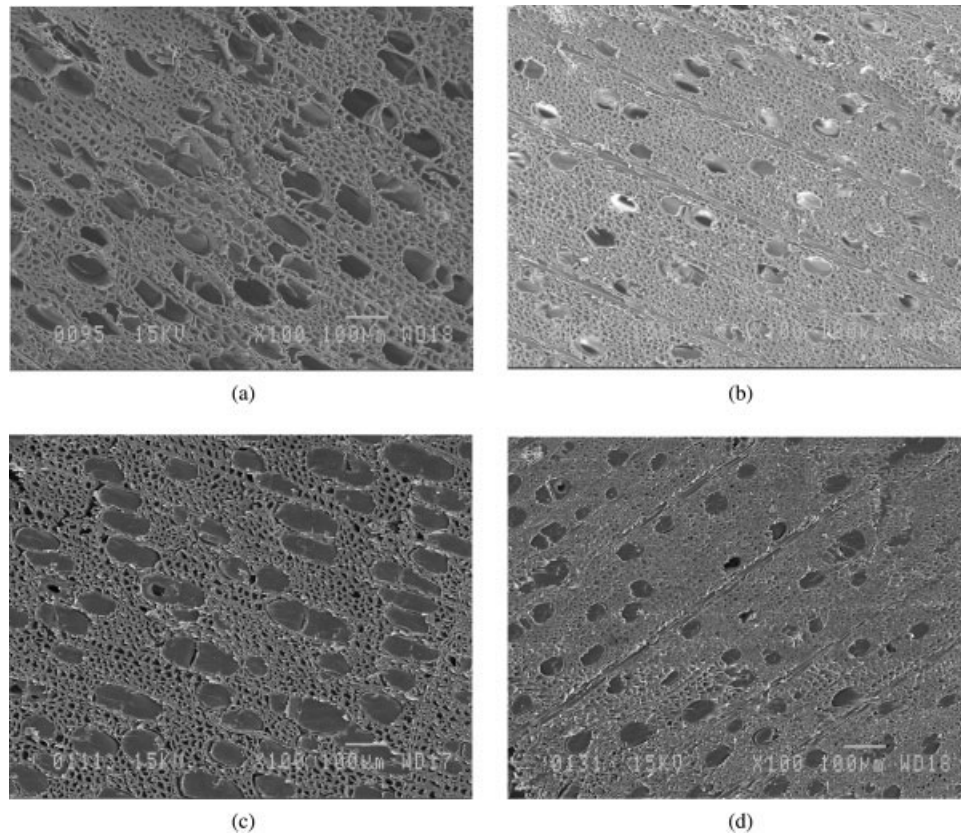


Figure 2 SEM micrographs of treated and untreated samples: (a) untreated aspen, (b) untreated red maple, (c) Run 8 in Table III, (d) Run 4 in Table III.

Wood species (G), impregnation pressure (D), ratio of impregnant molecular weight (B), and impregnation temperature (F) were found to be significant variables at the 0.05 probability level by the half-normal plot analysis. The linear regression relating these four significant variables to PR with a regression coefficient of 0.79 has been estimated as:

$$PR(\%) = 38.74 - 8.85B + 12.11D + 6.21F - 11.92G \quad (4)$$

where B, D, F, and G are defined in Table II, and their values are within the range bounded by the minimum (-1 code value) and maximum (+1 code value). A diagnostic check was applied to the residuals of the fit using eq. (4) and the actual PR, and the results supported the conclusion that wood species (G), impregnation pressure (D), ratio of impregnant molecular weight (B), and impregnation temperature (F) were the only significant variables for PR.

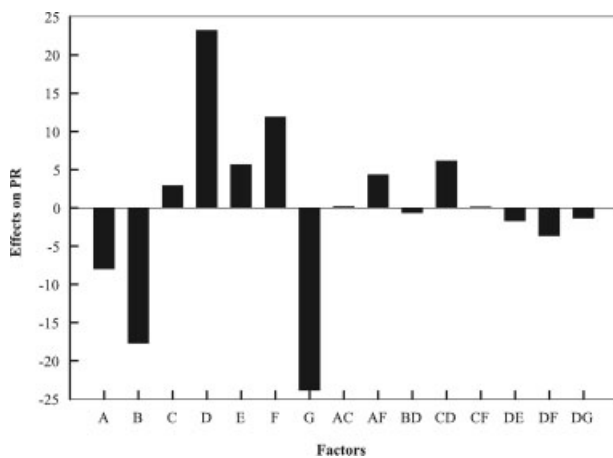


Figure 3 PR versus effects of factors (A-G are the variables defined in Table II).

Effect of treatment pressure on PR

PR as function of impregnation pressure is shown in Figure 4(a). The driving force for penetration is the difference between the pressure of the melt plastic surrounding the wood samples and that exerted by the residual air inside the wood material. At the same impregnation time and temperature, raising the external pressure resulted in an increase in the driving force pushing the melted polymer into the wood, which in turn gave a higher PR as illustrated in Figure 4(a). Costanza and Miyara⁸ simulated wood impreg-

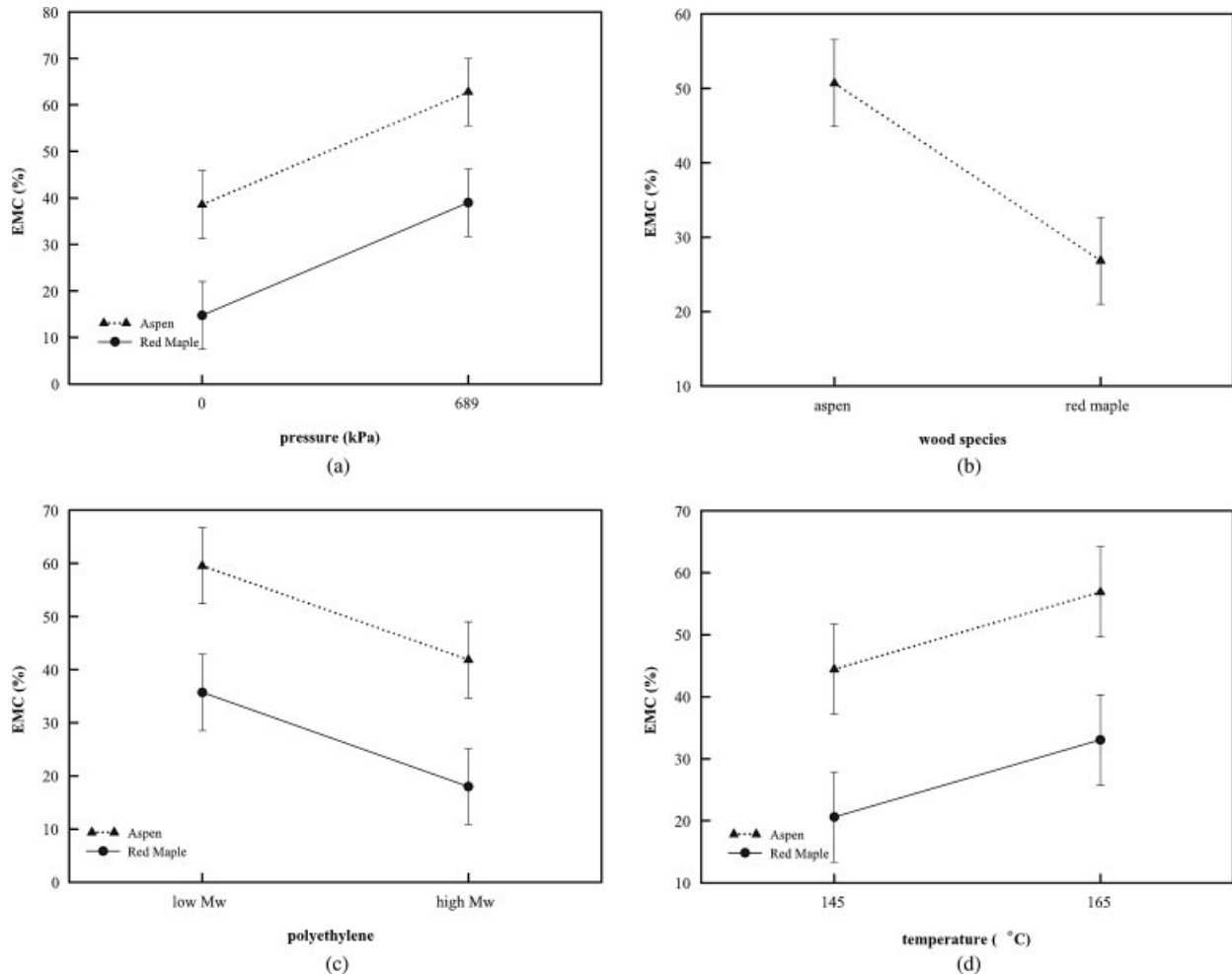


Figure 4 PR versus significant factors, (a) effect of impregnation pressure, (b) effect of wood species, (c) effect of polyethylene's molecular weight, and (d) effect of impregnation temperature.

nation with a vacuum and pressure procedure. They found that for a given period of time, higher external pressure led to higher PR in the wood. The result in the present study is consistent with this finding.

Effect of wood species on PR

Figure 4(b) clearly shows that PR is lower in red maple than in aspen. According to previous reports,^{8–10} the permeability of an impregnant is dependent on the wood cellular structure, such as the number of vessels per unit of cross-sectional area and vessel diameter, and the volume of the impregnant retention is proportional to the initial void volume of the untreated wood. In this study, the density of aspen was about 0.4 g/cm³ and that of red maple was about 0.6 g/cm³. The aspen had a higher void volume than the red maple, and from Figure 2, it is noted that the average diameters of its vessels and lumen were larger than those of red maple. All these factors made polymer penetration

into aspen easier than into red maple, leading to a higher PR value.

Effect of polymer on PR

The relationship between the weight average molecular weight (MW_w) and zero shear rate viscosity (η_0) of thermoplastic linear polymers depends on the value of MW_w . When MW_w is less than the critical (MW_w)_C of entanglements, η_0 is proportional to the MW_w , while above (MW_w)_C, η_0 is proportional to MW_w to the power 3.4.¹⁹ An increase in linear polymer molecular weight results in a higher viscosity. According to Peng's studies,^{9,10} permeability of an impregnant is proportional to the inverse of its viscosity, and an impregnant with a higher viscosity has a lower permeability into wood. Therefore, higher molecular weight polyethylene would have a lower PR for a similar impregnation procedure. PR as a function of ratio of impregnant molecular weight (B) is shown in Figure 4(c).

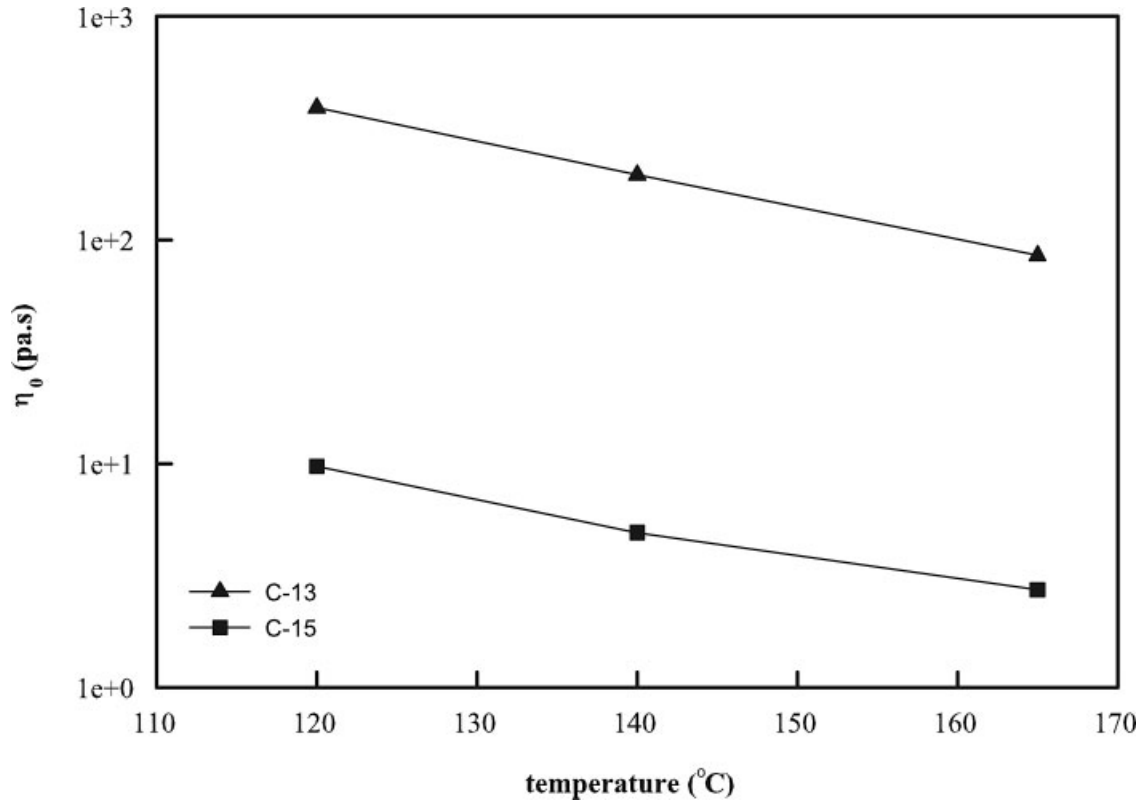


Figure 5 Viscosity as a function of temperature.

Effect of temperature on PR

An impregnant's permeability is related to its viscosity. Impregnants with low viscosity have higher permeability into wood.^{9,10} At a low shear rate, the viscosity of thermoplastic melt approaches η_0 . The viscosity of thermoplastics is strongly affected by temperature.¹⁹ Meissner²⁰ investigated the influence of temperature on the viscosity of a low-density polyethylene melt. η_0 decreases by two orders of magnitude as the temperature rises from 388 to 513 K. η_0 values of polyethylene C-13 and C-15 as functions of temperature are shown in Figure 5, which shows that increasing temperature lowers viscosity. Because viscosity decreases with increasing temperature, it is expected that higher permeability can be achieved with any increase in temperature. Figure 4(d) shows that PR is higher at a higher temperature than at a lower temperature, which is consistent with Perng's studies.^{9,10}

In summary, wood species (G), impregnant (B), and two processing parameters (pressure (D) and temperature (F)) are significant factors that affect PR. Equation (4) can be used to guide the optimization of processing conditions in the next stage of experimentation.

Hardness

The hardness (H_B) resulting from different runs is presented in Table III. SEM micrographs in Figure 2

show that vessel and lumen filling occurs in the treated specimen. Previous studies have found that surface hardness is influenced more by lumen filling than by cell wall penetration of polymer.²¹ It can be seen that even though the hardness of an impregnant is lower than or similar to that of the control sample (untreated wood), they still can lead to improved hardness after treatment.

Effects of studied variables on H_B are shown in Figure 6, which reveals the following ranking of ef-

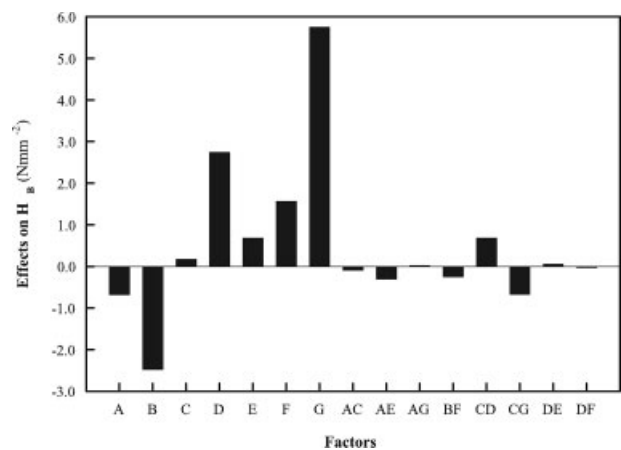


Figure 6 Effect on H_B versus effects of factors (A–G are the factors defined in Table II).

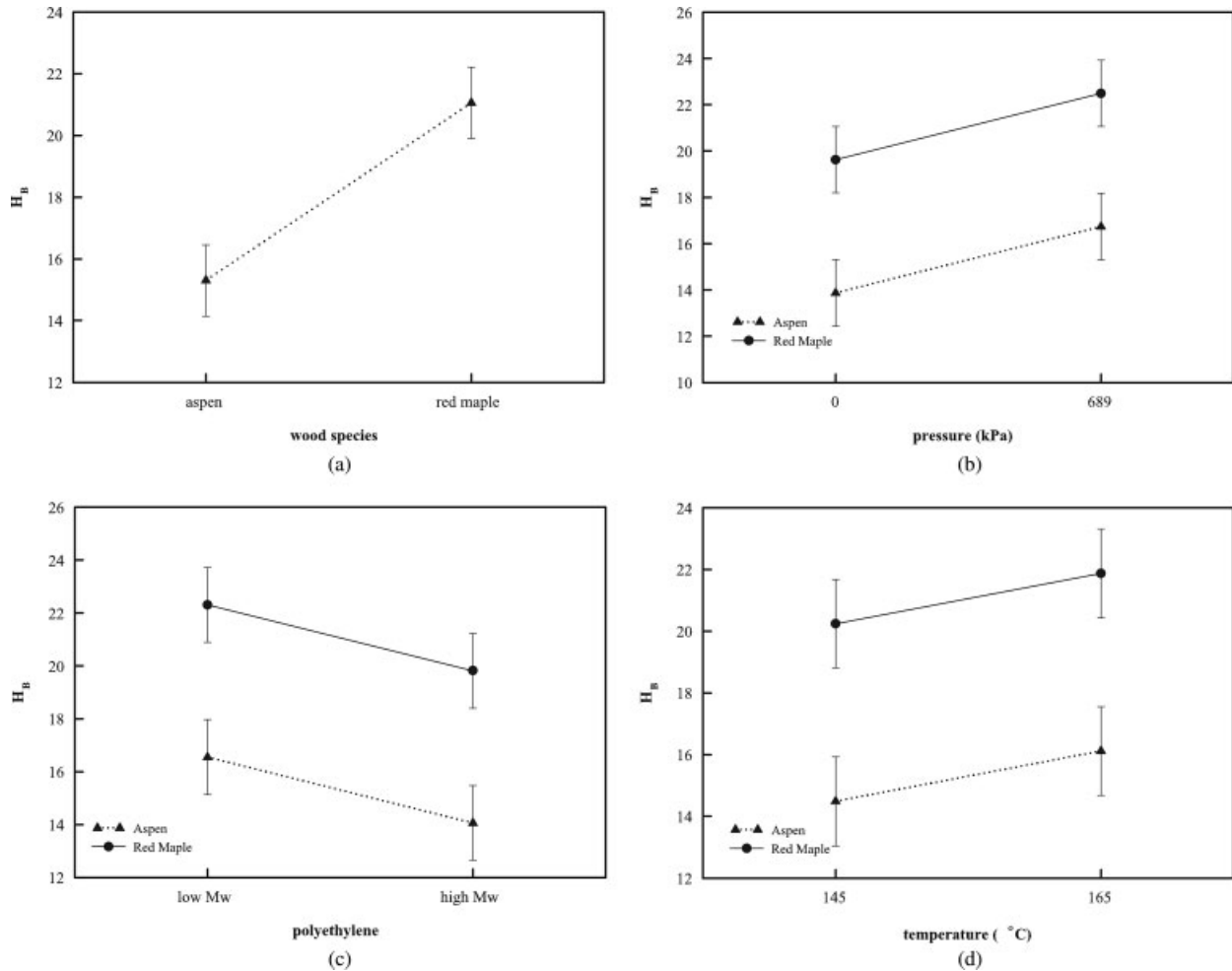


Figure 7 H_B versus significant factors, (a) effect of wood species, (b) effect of impregnation pressure, (c) effect of polyethylene's molecular weight, and (d) effect of impregnation temperature).

fects: wood species (G) > impregnation pressure (D) > ratio of impregnant molecular weight (B) > temperature (F) > impregnation time (E). Other variables that affected H_B were ratio of maleated polyethylene in the impregnant (A), the two-way interaction of vacuum and impregnation pressure ($C \times D$), and the two-way interaction of vacuum and wood species ($C \times G$). Again, wood species (G), impregnation pressure (D), ratio of impregnant molecular weight (B), and impregnation temperature (F) were found to be the most significant variables which affected H_B based on half-normal plot analysis. Linear regression of H_B as a function of these significant variables has been established ($R^2 = 0.95$) as follows:

$$H_B = 18.18 - 1.24B + 1.43D + 0.81F + 2.88G \quad (5)$$

where B, D, F, and G are defined in Table II, and their values are within the range bounded by the minimum (-1 code value) and maximum (+1 code value). A studentized residuals check has been used to validate the accuracy of eq. (5).

Effect of wood species on H_B

Figure 7 shows how H_B was affected by the four most significant variables. The average hardnesses of untreated aspen and red maple were 10.99 and 17.65 N/mm², respectively. Even though aspen had a higher PR (50%) than red maple at the same impregnation conditions, the higher polymer retention could not compensate for the difference in natural hardness of the two species. The average hardnesses of impregnants C-13 and C-15 were 10.40 and 8.62 N/mm², respectively. Figure 6 does show that wood species had the highest influence on hardness among all the tested variables. With similar impregnation processes, treated red maple samples had higher hardness than treated aspen [Fig. 7(a)] despite a lower PR.

Effect of treatment pressure on H_B

As discussed earlier, an increase in impregnation pressure resulted in a higher PR, and more impregnant occupying the vessels and lumen. Figure 7(b) shows that higher treatment pressure also produced a higher H_B .

Effect of polymer on H_B

The average hardnesses of C-13 and C-15 polyethylenes were 10.40 and 8.62 N/mm², respectively. If the specimens impregnated with C-13 and C-15 has the same PR, the specimen impregnated with C-13 should have a higher hardness than the one treated with C-15. Under the same impregnation process, polymer retention in wood for C-15 (59% in aspen and 35% in red maple) was higher than it was for C-13 (42% in aspen and 18% in red maple). Wood treated with the lower molecular weight polyethylene had a higher hardness than when impregnated with the higher molecular weight polymer, even though the hardness of C-13 is higher than that of C-15. Figure 7(c) clearly shows that wood treated with a lower molecular weight polyethylene had a higher hardness.

Effect of temperature on H_B

From earlier investigations,^{22–24} wood exposed to a high temperature undergoes a large reduction in hemicellulose content and then becomes more dimensionally stable. However, the strength and especially toughness is inevitably reduced. Depending on thermal treatment conditions, surface hardness of treated wood could be improved. The change in hardness as a function of temperature is shown in Figure 7(d). It depicts that high temperature treatments gave higher hardness than did the low temperature treatment counterpart. As discussed earlier, increased temperature decreased the viscosity of the impregnant (Fig. 5), and resulted in higher PR in the system [Fig. 4(d)]. As a consequence, high temperature treatments provided more hardness to treated specimens than the lower temperature treatments did. High temperature (165°C) treatments could also alter physical and chemical properties of wood.

Overall, among all the factors, wood species (G), polymer impregnant (B), and two process parameters (pressure (D) and temperature (F)) had significant effects on polymer retention (PR) and surface hardness (H_B).

CONCLUSIONS

The experimental design approach has identified the significant variables of the melt impregnation process for polymer retention (PR) and surface hardness (H_B). The present work showed that process parameters (pressure and temperature), polymer impregnant (different molecular weight polyethylenes), and wood species contributed significantly to PR and H_B . Increasing pressure and temperature resulted in higher PR and H_B , whereas increasing the molecular weight of the polyethylene impregnant and switching wood species from aspen to red maple gave a lower PR and H_B . There was no significant two-way interaction ef-

fect on PR and H_B . Continuation of the experimental work is essential and the following experimental design could be investigated in a future study.

1. A full factorial design with the few identified significant variables and additional wood species.
2. The use of two or three levels of important factors to determine the optimized process parameters for polymer retention and hardness.

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