

ACRYLATE WOOD DENSIFICATION: EFFECTS OF VACUUM TIME AND NANOPARTICLES ON CHEMICAL RETENTION, PENETRATION, AND RESIN DISTRIBUTION

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Abstract. The feasibility of preparing a surface-densified wood product by replacing the traditional time-consuming pressurization stage with only a short vacuum time was investigated. Sugar maple and red oak wood specimens were successfully impregnated with low-viscosity resins of 1,6 hexanediol dimethacrylate and trimethylolpropane trimethacrylate, with or without silicate nanoparticles, using vacuum times of 30 s to 10 min without pressurization. Chemical retention (CR) and vertical density profiles of the treated wood specimens were measured. The CRs obtained with the short vacuum impregnation process, even with a vacuum of 30 s or 60 s, proved comparable to those achieved by the traditional process of 30-min vacuum plus 30-min pressure. A 52-63 wt% CR was found for maple impregnated with neat resin, while the formulations containing nanoparticles achieved 44-55 wt% as the vacuum time was increased 30 s to 10 min. Oak yielded lower CR values. The vertical density profiles indicated better treatability for maple than oak. Examination of the resin and resin/nanoparticle penetration into the wood by scanning electron microscopy revealed successful wood impregnation with both nanoparticles and resin.

Keywords: Nanoparticles, surface densification, density profile, chemical retention, morphology.

INTRODUCTION

Wood modification by chemical impregnation to produce high value-added wood products with high-quality densification has existed for decades (Stamm 1964; Meyer 1965, 1981, 1982; Beall et al 1973, 1975; Moore et al 1983; Schneider 1994; Dale Ellis and O'Dell 1999; Mahmoud et al 2000; Gindl et al 2003a, 2003b; Schneider and Witt 2004; Siau 1984; Ayer et al 2003). Despite the performance attributes of the products, applications have been limited

because of costs of high chemical retention levels, slow curing methods, and the general low efficiency of the process. Conventional vacuum/pressure impregnation processes typically involve two steps, application of vacuum (a minimum of 15 min to several hours) to remove the air from the wood followed by pressure (a minimum 15 min to several hours) to force the chemicals into the wood structure. The chemical retention (CR) achievable in a conventional vacuum/pressure impregnation process is quite high, in some cases up to 80-200 wt%, which leads to extremely high costs (Beall et al 1973; Rowell 1991; Wright and Mathias 1993a, 1993; Breid 2002; Wan 2004).

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Because long impregnation schedules require batch processing, production efficiency is quite low. Shorter processing time combined with reduced CR such as that achievable through treatments focusing on wood surfaces, in which high density is needed, would yield significant savings and added value. It has been observed that the use of a “vacuum break” enhances chemical penetration into wood, and they are an essential element in the traditional impregnation process (Fuller et al 1997). The vacuum break is the end of the vacuum period when the impregnant is introduced and permitted to penetrate the wood. Fuller et al (1997) used hexanediol diacrylate (HDDA) or hexanediol dimethacrylate with a 15-min vacuum process plus a 5-40 min soak time for monomer to impregnate red oak veneers. The chemical retention of this vacuum process and soaking time was 30-80 wt%. Pressure steps maximize chemical penetration into wood void spaces, sometimes resulting in excessive CR levels. Elimination of the pressure step appears to offer potential for better control of penetration paths and chemical consumption.

Recent work by Cai et al (2007a, 2007b, 2008) has demonstrated that nanoparticles can penetrate the wood cell walls as part of a vacuum/pressure impregnation process, producing significantly improved mechanical/physical properties with additions of only 1% layered silicate nanoparticles in a low-viscosity resin as transport medium. A combination of nanoparticles and a melamine-urea-formaldehyde (MUF) resin significantly improved the surface hardness, abrasion resistance, and modulus of elasticity of the impregnated wood specimens while drastically enhancing their moisture resistance and dimensional stability.

The objective of this work was to investigate the feasibility of preparing a densified veneer for engineered floor surfaces. Specific objectives were to shorten the impregnation schedule by eliminating the pressure step and generally optimizing the schedule. This approach could permit changing the conventional discontinuous impregnation process into continuous industrial surface densification.

MATERIALS AND EXPERIMENTAL PROCEDURE

Wood Specimens

Veneer from sawn lumber of two wood species intended for the top surface of engineered wood flooring, sugar maple (SM) (*Acer saccharum*) and red oak (RO) (*Quercus rubra*), were obtained from Lauzon International Ltd (Québec). SM is diffuse-porous while RO is ring porous and both are important hardwood species on the North American market. Specimen size was 720-mm long \times 100-mm wide \times 3.2-mm thick. The specimens were cut for faces in the longitudinal-tangential plane. As shown in Fig 1, the specimen preparation cutting pattern was selected to minimize property variations and optimize statistical comparisons. They were numbered 1-1, 2-1, n-1, . . . , 15-1; 1-2, 2-2, n-2, . . . , 15-2; n-m, 1-9, 2-9, n-9, . . . , 15-9, where the maximum n is 15, the total specimen number for each treatment; and m represents different formulations/vacuum time treatment parameters. There were eight treatment combinations plus the control samples group for a maximum m of nine.

Chemicals and Formulations

Low-viscosity resins, HDDA and trimethylolpropane trimethacrylate (TMPTA), supplied

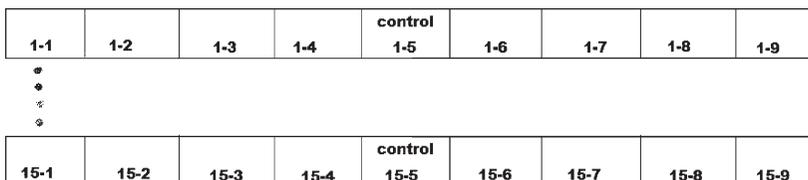


Figure 1. Specimen preparation for formulations involving different viscosities and micro/nanoparticles. Each treatment contains 15 specimens.

by AkzoNobel (formerly Chemcraft International, Warwick, Canada), were the carriers for nanoparticles. These are clear resins and safer to use than the methylmethacrylate (MMA) associated with traditional impregnation processes (Schneider 2001; Wan 2004). The viscosity of HDDA was 8 mPa·s at 25°C and that of TMPTA was 106 mPa·s. The nanoparticles were nanoclay (Claytone[®] APA; Southern Clay Products, Inc) at 1% loading blended with HDDA/TMPTA (75/25 by mass) resin in a high-speed mixer.

Impregnation Process

The veneer was oven-dried at 103°C for 24 h and the oven-dry mass recorded. Specimens were placed in an impregnation container, avoiding contact. The container was placed in a cylinder that was subjected to 3.3 kPa vacuum, maintained for 30 s, 60 s, 5 min, or 10 min, after which the vacuum line was closed. The chemicals were added to the container until the wood specimens were fully submerged, the vacuum released, and the container holding the specimens removed. The specimens were taken from the container, all residue was wiped off with paper towels, and the mass measured. Resin cure was achieved by electron beam (EB) radiation without catalyst. EB curing uses highly energetic electrons at a controlled energy level to polymerize and crosslink polymeric materials. The detailed EB curing process and EB curing behavior of the acrylate/nanofiller-impregnated wood will be discussed in a future paper.

Chemical Retention

Percentage CR was defined as the mass of the impregnated specimens (M_1) minus their oven-dry mass before impregnation (M_0) over M_0 multiplied by 100:

$$CR = (M_1 - M_0)/M_0 \times 100 \quad (1)$$

X-Ray Vertical Density Profiles

The untreated and treated specimens of SM and RO were cut to 50- × 50-mm subspecimens for the measurement of density profiles. The spec-

imens were conditioned at 65% RH and 20°C for at least 3 wk before testing. After conditioning, the dimensions and weight were recorded to determine densities. Density values were then converted into specific gravity at 12% MC using the following equation (Siau 1984):

$$\rho_{12} = \frac{\rho}{\left(1 + \frac{MC}{100}\right)} \times \rho_w \quad (2)$$

where ρ_{12} = wood specimen specific gravity at 12% MC, ρ = density, ρ_w = density at different thickness positions, and MC = moisture content.

The vertical density profiles of both impregnated and unimpregnated specimens were determined using an X-ray density profilometer.

Microscopic Characterization of Resin Distribution in the Wood Structure

Resin distribution in the treated and untreated wood specimens and their morphology were characterized using a Jeol JSM-840A SEM. The blocks of untreated SM and RO and those treated with a chemical (or chemical/nanoparticles) were prepared with a microtome by carefully cutting an end-grain edge to a depth of about 3 mm. All blocks were desiccated over phosphorus pentoxide for 2 wk. A gold/palladium alloy was sputtered onto the prepared surfaces before SEM examination.

RESULTS AND DISCUSSION

Chemical Retention

CR at different vacuum times were measured and calculated by Eq 1 (Fig 2). For SM impregnated with neat formulations, a mean CR of 56.5 wt% was obtained. Weight gains varied with different vacuum times: 53.9, 62.5, 56.8, and 52.7 wt%, respectively, for 30 s, 60 s, 5 min, and 10 min, but vacuum time clearly had no major effect on CR. For SM impregnated with nanoparticle formulations, weight gains averaged 49.5 wt% with CR values of 51.7, 47.1, 54.9, and 44.4 wt% for 30-s, 60-s, 5-min, and 10-min vacuum times, respectively. It therefore appears

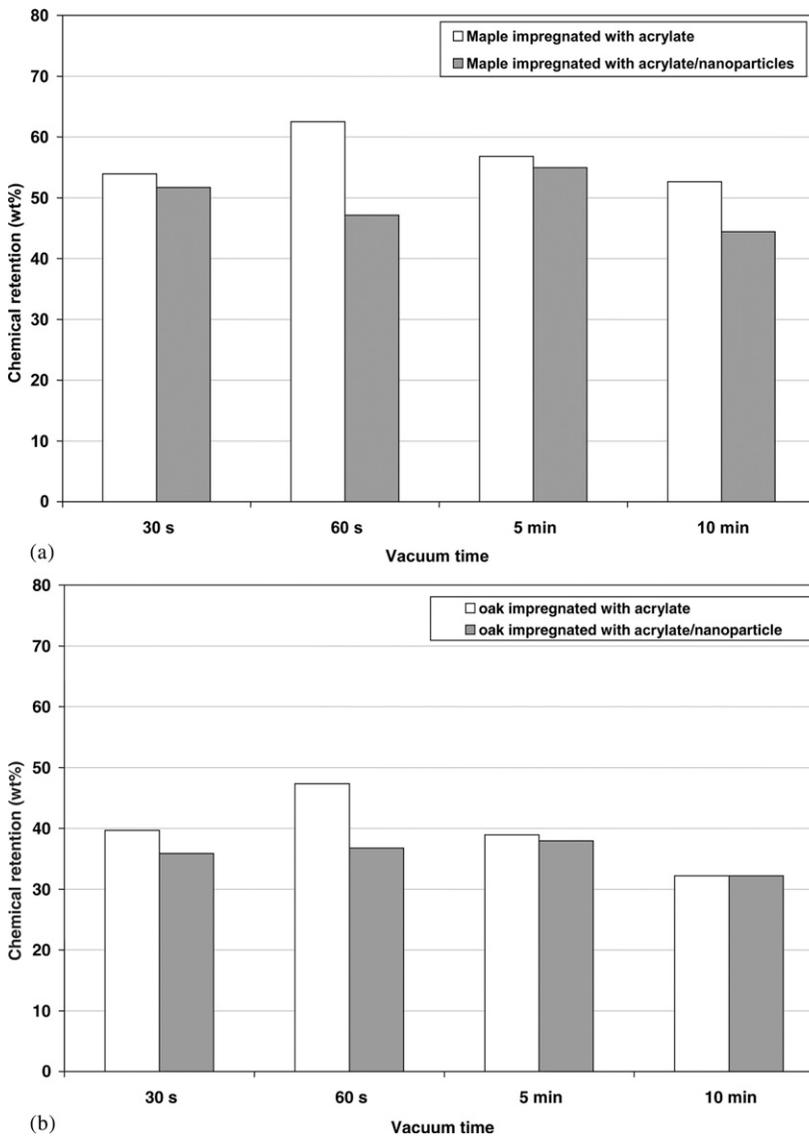


Figure 2. Chemical retention of wood specimens for different vacuum times and formulations containing nanoparticles (WN) or no nanoparticles (NN).

that, with the SM specimens, impregnation with nanoparticle formulations yielded lower CR values than neat formulations (averages of 49.5 and 56.5 wt%, respectively). This may be ascribed to the accumulation of nanoparticles in pores located near the surface as observed in our previous work (Cai et al 2007a, 2007b) and to the flow resistance caused by such nanoparticles, limiting resin penetration into the center of the specimens.

For the RO specimens impregnated with neat formulations (without nanoparticles), CR values averaged 39.5 wt%, much lower than for SM (56.5 wt%). With 30-s, 60-s, 5-min, and 10-min vacuum times, 39.7, 47.3, 38.9, and 32.3 wt% CR, respectively, were obtained. It was noted that the RO specimens had the highest CR with a vacuum time of 60 s, which implies that a vacuum of 30–60 s would be enough to achieve considerable weight gains. With formulations

containing nanoparticles, the RO specimens had an average 35.7; 35.9, 36.8, 38.0, and 32.2 wt% CR after 30-s, 60-s, 5-min, and 10-min vacuum times, respectively. The effect of vacuum time on the CR of specimens impregnated with nanoparticle/resin formulations was not clear; it appears that breaking of the vacuum is an important factor. As observed with the SM specimens, the CR of RO impregnated with formulations containing nanoparticles (35.7 wt%) was lower than for those impregnated with neat formulations (39.5 wt%).

A comparison between SM and RO shows that, under the same impregnation parameters (ie the same vacuum time and formulations), SM achieved higher CR values than RO. An analysis of variance performed on CR values (Table 1) indicated that the differences between SM and RO were not statistically significantly different ($\alpha = 0.01$) in terms of vacuum time or the use of nanoparticles. The introduction of nanoparticles did have a significant effect on CR at a lower level ($\alpha = 0.05$) for SM. The interaction effect of the two factors (vacuum time \times formula) on CR was not significantly different between the two species. In terms of wood species, CR were highly statistically different ($\alpha = 0.01$), assumed to be from different anatomical structures. Previous work on aspen, which is diffuse-porous and low in density, showed that impregnation with MUF/nanoparticles using a traditional vacuum/pressure method with 30-min vacuum plus 30-min pressure yielded an average CR of

67.7 wt% (Cai et al 2007a). For the high-density SM, a mean CR of 25-50 wt% was observed with MMA using a similar vacuum/pressure impregnation cycle (Wan 2004). A comparison with the CRs obtained in this study suggests that there is no major difference in chemical retention for the traditional long vacuum/pressure impregnation process and our shortened vacuum-only impregnation process, even with vacuum times less than 60 s, confirming the use of vacuum breaks.

X-Ray Vertical Density Profile

Figure 3 shows density profiles for the treated SM with different vacuum times and formulations (with and without nanoparticles). The average density of the untreated maple wood was 670 kg/m³ (Table 2), while the densities of all treated maple specimens, at different vacuum times with formulations with or without nanoparticles, were above 1000 kg/m³. With a 30-s vacuum, treatment with formulations without nanoparticles produced an average density of 1054 kg/m³ and an almost uniform density distribution. With formulations containing nanoparticles, a lower average density of 1007 kg/m³ was obtained with the density distribution decreasing from the surface to the center. Small peaks were observed on either side. The peak density was above 1100 kg/m³, while the lowest density, at the center, was about 986 kg/m³. Table 3 summarizes all peak positions in the treated wood with formulations containing

Table 1. ANOVA results for chemical retention (CR) of impregnated sugar maple and red oak.

| Source | Degree of freedom | Type III sum of squares | Mean square | F value | Pr > F |
|------------------------------------|-------------------|-------------------------|-------------|---------|----------------------|
| ANOVA for impregnated sugar maple | | | | | |
| Formula | 1 | 381.57 | 381.57 | 7.64 | 0.0108** |
| Vacuum time | 3 | 252.08 | 84.03 | 1.68 | 0.1972 |
| Formula \times vacuum time | 3 | 240.19 | 80.06 | 1.60 | 0.2146 |
| ANOVA for impregnated red oak | | | | | |
| Formula | 1 | 117.43 | 117.43 | 1.76 | 0.1975 |
| Vacuum time | 3 | 399.52 | 133.17 | 1.99 | 0.1420 |
| Formula \times vacuum time | 3 | 135.49 | 45.16 | 0.68 | 0.5754 |
| ANOVA for maple and oak comparison | | | | | |
| Species | 1 | 3794.56 | 3794.56 | 54.35 | <0.0001 ^a |

^a Significant difference at $\alpha = 0.05$.

ANOVA, analysis of variance.

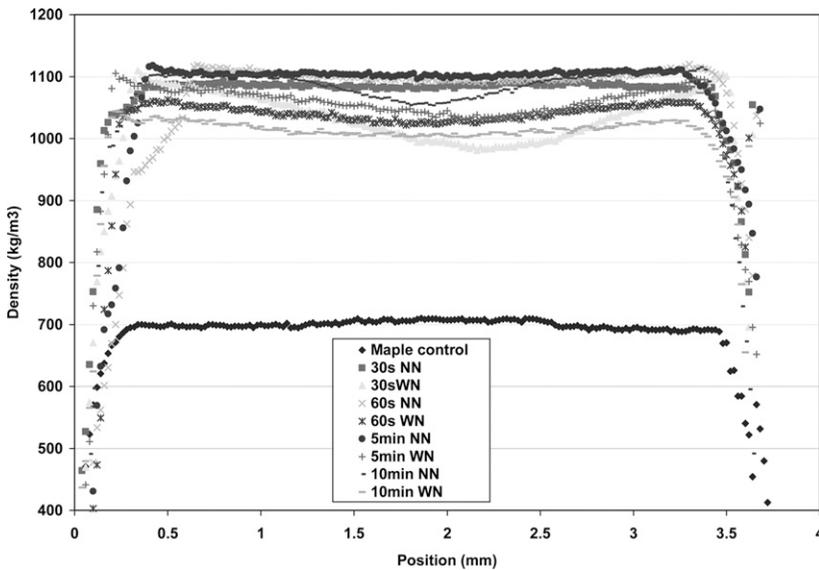


Figure 3. X-ray density profiles of maple specimens for different vacuum times and formulations containing nanoparticles (WN) or no nanoparticles (NN).

Table 2. Average density for sugar maple and red oak treated with different vacuum times and formulations (with and without nanoparticles).

| Treatment | Average density (kg/m ³) | | | |
|-------------------|--------------------------------------|--------------------|---------|--------------------|
| | Sugar maple | | Red oak | |
| | Average | Standard deviation | Average | Standard deviation |
| Control specimens | 670 | 89 | 663 | 85 |
| 30 s–NN | 1054 | 114 | 910 | 137 |
| 30 s–WN | 1007 | 127 | 881 | 108 |
| 60 s–NN | 1037 | 174 | 909 | 113 |
| 60 s–WN | 1001 | 151 | 916 | 127 |
| 5 min–NN | 1047 | 168 | 917 | 114 |
| 5 min–WN | 1024 | 136 | 933 | 86 |
| 10 min–NN | 1042 | 155 | 901 | 93 |
| 10 min–WN | 987 | 115 | 863 | 84 |

WN, with nanoparticles; NN, no nanoparticles.

nanoparticles. For SM, all peaks dropped off within 0.22–0.38 mm from the surface. This was because some nanoparticles accumulated in the pores near the surface, causing higher surface density. The higher nanoparticle concentration and resulting higher density in the top layer are a benefit for the resulting wood products. Higher surface density causes an increase in surface hardness, an especially desirable attribute in flooring. Some other properties such as abrasion resistance may also be enhanced (Cai et al

Table 3. Density peak position from surface (from left to right) according to the vertical density profiles of maple and oak wood specimens impregnated with nanoparticles/formulations.

| impregnation condition | Density peak position from the surface (mm) | |
|------------------------|---|---------|
| | Sugar maple | Red oak |
| 30 s–WN | 0.22 | — |
| 60 s–WN | 0.22 | 0.30 |
| 5 min–WN | 0.34 | 0.22 |
| 10 min–WN | 0.38 | 0.18 |

WN, with nanoparticles.

2007a). Because nanoparticles accumulating in the wood surfaces reduce flow and penetration of the chemicals into the core of the wood, a lower overall chemical retention can adequately densify the surfaces.

The average density of treated SM with 60-s vacuum time and formulations without nanoparticles was 1037 kg/m³ as compared with 1001 kg/m³ at the same vacuum time with formulations containing nanoparticles. Like with RO, neat resin formulations yielded a slightly higher density and more uniform density distribution than those combining nanoparticles and a resin. With a 5-min vacuum time, the average density of SM treated with a neat resin formulation was 1047 kg/m³ as

compared with 1024 kg/m^3 with formulations containing nanoparticles. For the resin/nanoparticle formulation, the peak density was 1105 kg/m^3 near both faces, the lowest density being 1034 kg/m^3 at the center. When a 10-min vacuum was used, density averaged 1042 kg/m^3 for the formulation without nanoparticles and 978 kg/m^3 with the formulation containing nanoparticles.

The average density of untreated RO was 663 kg/m^3 and density distribution was relatively even (Fig 4). With a 30-s vacuum time, the average density of RO treated without nanoparticles was 910 kg/m^3 , and the density distribution was even throughout (Table 2). With nanoparticle formulations, a lower density (881 kg/m^3) was observed. With a 60-s vacuum, the density averaged 909 kg/m^3 with a neat resin formulation and 915 kg/m^3 with a nanoparticle formulation. This was a slight difference, but density peaks of about 1034 kg/m^3 were observed near the surfaces as compared with 912 kg/m^3 at the center. A similar pattern occurred with a 5-min vacuum: 916 kg/m^3 for neat resin and 933 kg/m^3 for nanoparticle resin. As vacuum time increased to 10 min, an average density of 901 kg/m^3 was obtained with neat resin, but a lower

density (863 kg/m^3) was observed with the nanoparticle formulation.

From the average density results, all the treated RO exhibited higher densities than the untreated control specimens. The nanoclay filler generally showed little impact on mean density, but its effect on density profiles was obvious with density peaks occurring close to wood surfaces. Density distribution proved relatively even across wood sections when a neat formulation was used. The density peaks in treated RO were 0.18–0.30 mm from the surface (Table 3). With RO, some differences in density distribution were observed with different vacuum times.

Morphology of Impregnated Wood by Scanning Electron Microscopy

Figure 5 shows SEM images of SM before and after treatment with 60-s vacuum impregnation: (a) control, (b) treated with HDDA/TMPTA and 1% nanoparticles (47.1 wt% chemicals including nanoparticles), and (c) treated with HDDA/TMPTA (containing 62.5 wt% of chemicals) in a typical cross-section. Good penetration and adhesion of the HDDA/TMPTA resin can be

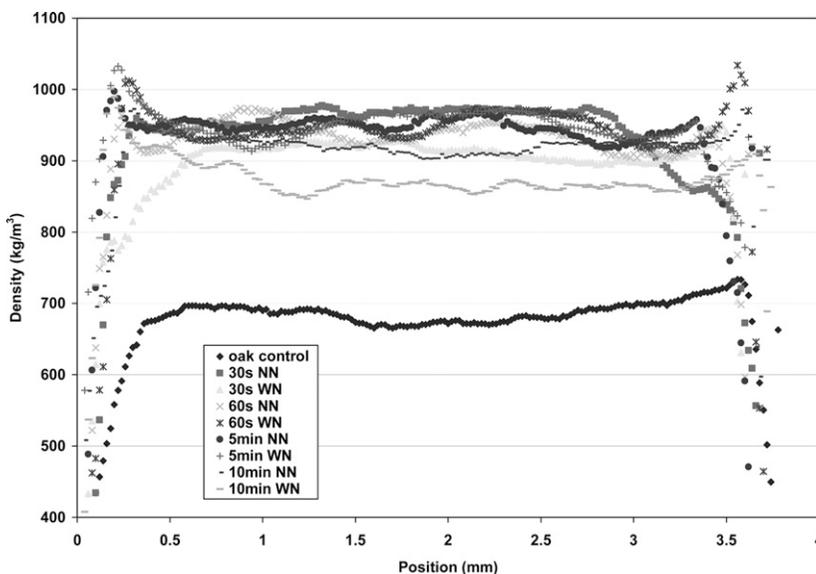


Figure 4. X-ray density profiles of oak specimens for different vacuum times and formulations containing nanoparticles (WN) or no nanoparticles (NN).

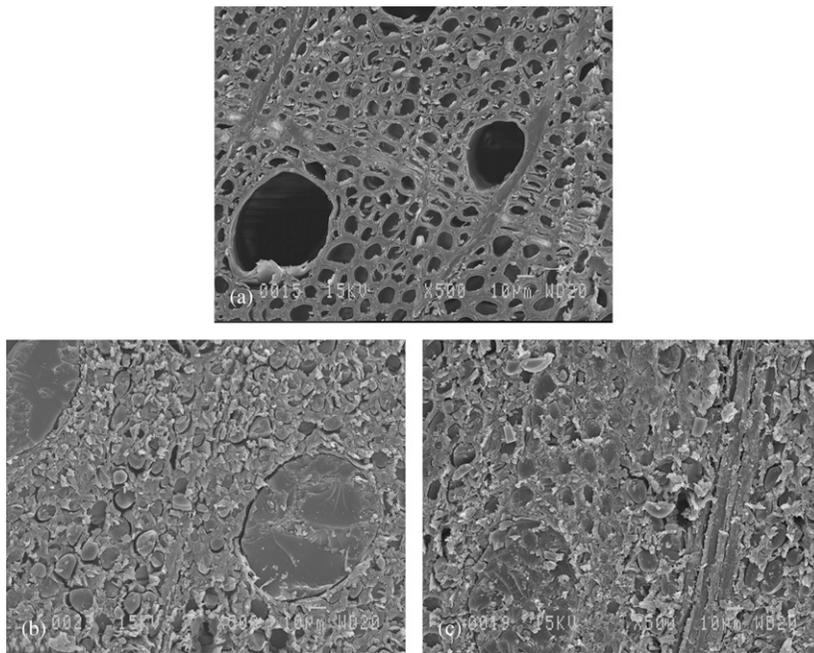


Figure 5. SEM photographs of maple wood specimens before and after treatment with 60-s vacuum impregnation time: (a) control specimen; (b) maple treated with hexanediol diacrylate/trimethylolpropane trimethacrylate (HDDA/TMPTA) and 1% nanoparticles (47.1 wt% chemicals with nanoparticles); (c) maple treated with HDDA/TMPTA (62.5 wt% chemicals).

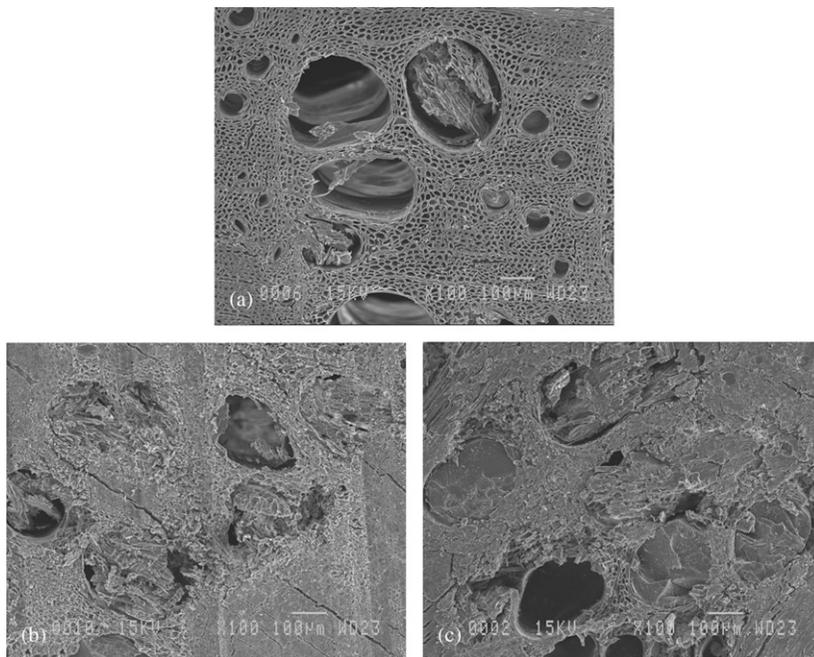


Figure 6. SEM photographs of oak wood specimens before and after treatment (with 60-s vacuum impregnation time): (a) oak control specimen; (b) oak treated with hexanediol diacrylate/trimethylolpropane trimethacrylate (HDDA/TMPTA) and 1% nanoparticles (36.8 wt% chemicals/nanoparticles); (c) oak treated with HDDA/TMPTA (47.3 wt% chemicals).

clearly observed in the treated SM. For the nanoparticles/HDDA/TMPTA-treated SM, the chemical loading was lower, and the impregnated materials appeared to be in both cell walls and lumens (Figs 5b-c). A similar cross-section morphology can be observed with the different impregnation parameters. The morphology of HDDA/TMPTA penetration into SM wood with the short vacuum times (30 s, 60 s, 5 min, and 10 min) was comparable to that obtained with the conventional impregnation process based on 15-45 min vacuum plus 15-45 min pressure (Cai et al 2007b, 2008). The weight gains achieved were highest with the 60-s vacuum time treatment involving HDDA/TMPTA formulations without nanoparticles (62.5 wt%).

Figure 6 shows morphologies for typical cross-sections of an untreated control RO (Fig 6a) and treated RO (Figs 6b-c) with a 60-s vacuum impregnation time. For the untreated RO, empty cell wall, pits, and parenchyma can easily be observed, while for the impregnated oak, the empty pores have been filled with resin or nanoparticle/resin. However, some empty lumens (mainly vessels) were still visible.

CONCLUSIONS

The feasibility of preparing surface-densified wood by eliminating the traditional long impregnation pressure step with only a short vacuum step was investigated. Specimens of two species (SM and RO) were successfully impregnated with chemical formulations based on low viscosity resins, ie HDDA and TMPTA, and clay nanoparticles, using vacuum times of 30 s, 60 s, 5 min, or 10 min without subsequent pressure. Weight gains and density increases obtained with these short vacuum schedules were comparable to those achieved with the traditional 30-min vacuum plus 30-min pressure impregnation process. With vacuum times of 30 s to 10 min, chemical retentions of 52.7-62.5 wt% were observed for SM impregnated with a neat formulation of HDDA/TMPTA, the corresponding retentions being 44.4-54.9 wt% for the formulations containing nanoparticles. Retentions were

lower with RO for the same formulations and vacuum times. The density profile indicated that SM has better treatability than RO.

The morphology and penetration of the resin with and without nanoparticles were observed and characterized by SEM. This indicated that both nanoparticles and resin successfully penetrated all the wood in both lumens and cell walls. Because of nanoparticle accumulations, density peaks were observed near wood surfaces at a depth of 0.2-0.4 mm for both SM and RO when impregnated with nanoparticle formulations. This is considered a benefit for wood products for such specific applications as flooring.

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