

Electron-Beam Curing of Acrylate/Nanoparticle Impregnated Wood Products

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This study investigated the feasibility of using an electron beam (EB) process to cure chemically impregnated wood products. Maple wood planks were impregnated with the low-viscosity resins 1,6 hexanediol dimethacrylate (HDDA) and trimethylolpropane trimethacrylate (TMPTA). The addition of nanoparticles into the formulation was also studied. The impregnated wood was then cured by EB irradiation. The EB curing method utilizes highly energetic electrons at a controlled energy level to polymerize and cross-link the polymeric materials. The thermal analysis results of differential scanning calorimetry (DSC) confirmed that the curing of chemically impregnated wood by electron beam radiation was validated. Polymerization exotherms were observed for the neat acrylate resin and formulations of acrylate/nanoparticles impregnated maple samples. No polymerization exothermal peaks were observed for both EB-cured impregnated maple and control maple samples, confirming that EB irradiation can serve as an efficient curing method to polymerize acrylate-impregnated wood products. The surface hardness of the EB-cured impregnated maple wood was improved up to 200%.

Keywords: Wood modification; Electron beam curing; Cure behaviour; Thermal analysis; Surface hardness properties

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INTRODUCTION

Wood modification *via* chemical impregnation to improve specific wood properties for high quality wood products has been around for decades (Siau *et al.* 1965; Moore *et al.* 1983; Rowell 1983; 1991; Schneider 1994; 2000; Singh *et al.* 1999). Applications of this technology have been limited because of high cost resulting from a large volume of chemical consumption, a low efficiency impregnation process, and a slow curing process. Radiation such as atomic particles, neutrons, photons, gamma rays, X-rays has been used to cure wood-densification products (Siau *et al.* 1965; Burmester 1967). However, the safety, environment of radiation curing, and the cost of transportation of the various wood products to and from the radiation site would be prohibitive (Meyer 1965).

The authors' previous work investigated the feasibility of developing a fast surface impregnation process, which could significantly decrease chemical consumption while maintaining the same or even better properties of the final surface densified wood products (Cai *et al.* 2010; 2011). However, a fast curing method is needed to integrate the fast impregnation, curing and finishing process, which can strategically turn the whole wood modification process to an industrially viable and profitable process. Traditional thermal curing of impregnated wood needs long cure times ranging from 30 min to hours, and it requires high energy consumption (Cai *et al.* 2007a,b; 2010). Thermal cure methods also

produce volatile toxic by-products, create residual stresses in the materials, and require expensive tooling capable of withstanding high autoclave temperatures.

The EB curing method utilizes highly energetic electrons at a controlled energy level to polymerize and cross-link polymeric materials. The process utilizes ionizing irradiation in the form of accelerated electrons that interact with matter by transferring energy to the electrons orbiting the atomic nuclei of the target materials. These electrons may then be either released from atoms, yielding positively charged ions and free electrons, or moved to a higher-energy atomic orbital, yielding an excited atom or molecule (free radical) (Wilson 1974). These ions, electrons, and excited species are the precursors of any chemical changes observed in an irradiated material. At this point, crosslinking occurs in many materials.

Electron beam radiation as an alternative to chemical initiation in polymerization is a well-known technique (Wadhwa and Walsh 1982; Bly 1988; Janke *et al.* 1996; Crivello *et al.* 1996; Madani and Badawy 2004; Pusch and Van Herk 2005; Minibiolo and Yasenchak 2007). Allaway *et al.* (1983) successfully applied EB to cure multi-functional acrylate monomers to form a foaming polymer. Tran and Weiss (2000) reported a method of making an electron beam polymerized emulsion-based acrylate pressure sensitive adhesive. In this patent, a one-step process using electron beam radiation to polymerize pressure sensitive adhesives from acrylate emulsions was disclosed. Both patents showed that acrylate resin can be polymerized under exposure to EB radiation. Harper *et al.* (2006) described a method using electron beam processing to cure wood-polyethylene-acrylic composites. The same group (Griffith *et al.* 2006) presented results on electron-beam cured resin system for wood composites.

The goal of this research was to evaluate the feasibility of developing an alternative fast curing process *via* electron beam (EB) irradiation to polymerize chemically impregnated wood products to replace the conventional slow thermal cure process. Through the combination of previously developed high-efficient surface densification processes (Cai and Blanchet 2010a) and the instant online curable EB technique, a cost-effective industrially viable surface densification online curing process could be developed. Success with this approach could strategically turn the conventional discontinuous curing process into a continuous industrial viable curing process to polymerize chemical densification of wood products. This development will potentially benefit wood industrial such as wood flooring, siding, windows/doors, cabinets, and furniture.

EXPERIMENTAL

Materials

The engineered wood planks used in this research included maple (*Acer saccharum*), with a dimension of 72.4 cm × 8.9 cm × 0.3 cm. The 1,6 hexanediol dimethacrylate (HDDA) and trimethylolpropane trimethacrylate (TMPTA) were supplied by AkzoNobel (formally Chemcraft International, Warwick, Canada). The mixture of these two resins was utilized as the transport medium resins for nanoparticles. The Claytone® APA nanoparticles, used in this work were provided by Southern Clay Products, Inc. (USA). The Claytone® APA is a nanoclay that was surface modified with function group of $R_1R_2R_3R_4N^+Cl^-$. The specific gravity of this nanoclay is 1.70 with particle size around 8

μm where each particle contains 3000 platelets (Cai *et al.* 2010). The wood planks with a moisture content of 8% were impregnated with a formulation of HDDA/TMPTA (75/25) either containing 1% nanoparticles or neat monomers. The details of the impregnation process are described in previous works (Cai *et al.* 2007a; Cai and Blanchet 2010). The chemical retention (CR) of the impregnated maple wood was calculated based on their mass changes as follows,

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

where M_0 is the mass before treatment and M_1 the mass after impregnation.

Methods

EB curing of HDDTA/TMPTA/nanoparticle impregnated wood planks

The accelerated electrons can penetrate the curing substrate and the depth of the penetration depends on the dose of the EB generator (Epstein 2008). The acrylate monomers-impregnated wood planks were sent to EB Services Inc. for curing. All samples were irradiated to 100 kGy, using 4.5 MeV energy electrons. The accelerator used was a RDI Dynamitron (E-Beam Services; Lebanon, Ohio, US). The dose provided increments of 25 kGy per pass. Samples were processed from one side. The temperature of the samples was below 49 °C upon completion of the irradiation. The time between passes was about 7 min when running at full speed. The polymerization reaction of HDDA and TMPTA was completed under the accelerated electron radiation.

Validation characterization with DSC

All DSC measurements were made with a Mettler DSC 20 (Mettler-Toledo, LLC; Columbus, OH, USA) equipped with a Mettler TA400 thermal analysis system and STAR[®] software. The neat HDDA/TMPTA resins and the nanoclay/resin mixtures with 1% of thermal catalyst Vazo[®]67 (DuPont Canada; Mississauga, ON, Canada) were tested, with about 10 mg of each sample in a sample crucible. A high-pressure steel-sealed capsule that could withstand a vapour pressure up to 10 MPa was used to prevent the components of the samples from evaporating at high temperature. Dynamic scans were made from 25 °C to 200 °C at a heating rate of 5 °C/min. A minimum of 3 repetitions of DSC characterization was carried out for each group of samples. Both the neat resin and the resin with 1% nanoparticle-impregnated maple and oak wood were scanned with DSC by placing about 10 mg of each sample into an aluminum gold-sealed capsule. The EB-cured impregnated maple and oak wood samples and control pure wood samples were also scanned by placing about 10 mg of each sample into an aluminum capsule sealed with a gold film. The apparent activation energy of the curing reaction of the HDDA/TMPTA resin along or a mixed nanofiller/HDDA/TMPTA sample was calculated on the basis of the variation of the peak reaction temperature (T_{max}) as a function of the heating rate increase of the DSC scan, using Kissinger methods (1957). The integral of the exotherm peak was calculated and summarized.

Scanning electron microscopy and surface hardness measurements

The cross sections of the EB-cured HDDA/TMPTA-impregnated wood samples were characterized using a JEOL JSM-840A scanning electron microscope (Japan) operating at an accelerating voltage of 15 kV. The blocks of untreated maple and oak wood, and those treated with a chemical (or chemical/nanoparticles) were prepared with a

microtome by carefully cutting one of the end-grain faces to a depth of about 3 mm. All blocks were desiccated with phosphorus pentoxide for two weeks. A gold/palladium alloy was sputtered onto the prepared surfaces prior to the analysis.

The Brinell surface hardness of the acrylate-impregnated maple wood cured by EB radiation was measured according to the European Standard EN 1534 (2000), using an Alliance RT/50 system from MTS Systems Corp. (Minnesota, USA). The Brinell hardness was calculated as the slope of load vs. indentation within a 20% to 60% indentation range. Three points were tested for each sample and at least 9 samples were tested for each combination.

Impact resistance measurement and data analyses

The falling ball impact resistance test measures the ability of impregnated wood to resist impact of a free-falling large steel ball when dropped on the specimen face from a specific height. The test followed ASTM D 2794-93 (ASTM 1999). Because of electromagnets, the ball can be placed at different heights, low (76 mm), medium (229 mm), and high (457 mm), to evaluate impact resistance of impregnated wood boards. For each impact, indentation diameter and depth were examined and measured. The control maple wood samples and the EB-cured acrylate impregnated maple wood with different impregnation processes were tested. Three heights represent three levels of impact (low, medium, and high impact, from a height of 7.62 cm, 22.86 cm, and 45.72 cm, respectively). A minimum of 5 samples were tested. There were 6 testing points for each sample, thus 30 test data points were collected for each parameter. Both indentation depth and diameters were measured for three different impact strengths.

The effect of different impregnation processes on impact resistance was analyzed with the Statistical Analysis System (SAS) software. The analysis of variance (ANOVA) of impact resistance of both indentation fracture diameters and depths at different impact strength on maple engineered wood planks was performed.

RESULTS AND DISCUSSION

EB Curing of HDDA/TMPTA/Nanoparticles Impregnated Wood Planks

The total dose received by the precursor emulsion, the acrylate resin impregnated into the wood, primarily affects the extent to which monomer is converted to a polymer and the extent to which the polymers are crosslinked. To provide enough penetration and polymerization of acrylate-impregnated wood planks, it is preferable that the dosage be greater than 25 kGy with 4 passes. A radiation polymerization step can be carried out in a broad range of temperatures, with the preferred range being from about 20 °C to 30 °C. In this range of ambient temperatures, the heating or cooling energy requirements of the process are minimized. The fully penetrated multifunctional acrylate monomer formulations or formulations mixed with nanoparticles-impregnated engineered wood planks are then passed under the electron beam so as to polymerize the acrylate monomers to a polymer composite structure.

Figure 1 shows the EB-cured HDDA/TMPTA impregnated maple planks with different impregnation times, where sample A is the control sample, B is acrylate monomers-impregnated maple planks with 10 min of vacuum process, C is acrylate monomers-impregnated maple planks with 5 min of vacuum, and D is acrylate monomers-impregnated maple planks with conventional 15 min of vacuum followed by a 15 min

pressure process. The EB-cured acrylate monomer formulation or formulations with nanoparticle-impregnated engineered wood planks exhibited smooth surfaces, and some polymerized acrylate polymers were evident on the side faces of the cured impregnated maple wood samples.



Fig. 1. EB cure impregnated sugar maple samples: (A) Control; (B) 10 min vacuum; (C) 5 min vacuum; and (D) 15 min vacuum/15 min pressure

The mass of the EB-cured impregnated wood samples was measured to calculate the density of the final surface modification wood products. The chemical retention of the acrylate-impregnated maple was in the range of 58% to 61 wt%, and the density of the EB-cured final products are presented in Fig. 2. The density of the EB-cured acrylate-impregnated maple planks and the control maple wood are also indicated in Fig. 2.

Curing Behavior of EB Polymerizing Acrylate/Nanoparticles

The validation analysis of polymerization of EB irradiation is very important to develop an online-applicable curing process to cure impregnated wood planks. To verify that the EB cure is an efficient polymerization method, further investigation *via* differential scanning calorimetric (DSC) characterization was used to validate the curing behaviour of EB-cured acrylate or acrylate/nanoparticle-impregnated wood samples. Neat HDDA/TMPTA acrylate multifunction monomers, and HDDA/TMPTA/nanoparticles plus 1% of thermal catalyst Vazo[®]67 were characterized by DSC from 25 °C to 200 °C, with a heating rate of 5 °C/min. A minimum of three experiments was repeated for each formulation. The DSC results are presented in Fig. 3. Large exothermal peaks were observed for both the neat acrylate monomers and acrylate/nanoparticles. The peak temperature of the neat acrylate monomer was 102.2 °C, and a slightly higher peak was observed with the addition of 1% nanoparticles into the acrylate monomer formulations, where a 105.2 °C peak temperature was observed (Table 1). The onset temperature of the reaction ranged from 98 °C to 101 °C for the neat acrylate monomers and those with nanoparticles.

The HDDA/TMPTA acrylate and HDDA/TMPTA/nanoparticles acrylate monomer impregnated maple wood were placed in an aluminum gold-sealed capsule. The scanning parameters used for the neat acrylate monomers or formulation plus nanoparticles were also used to characterize the impregnated maple wood. The DSC analysis results are presented in Fig. 4.

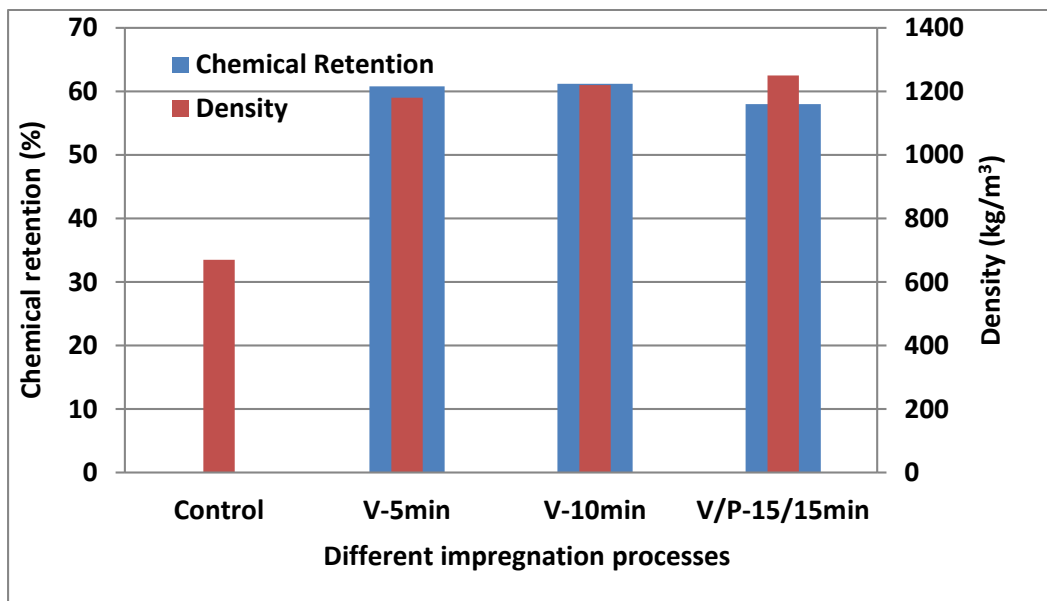


Fig. 2. Chemical retention and final density of HDDA/TMPTA-impregnated maple wood with different vacuum impregnation times

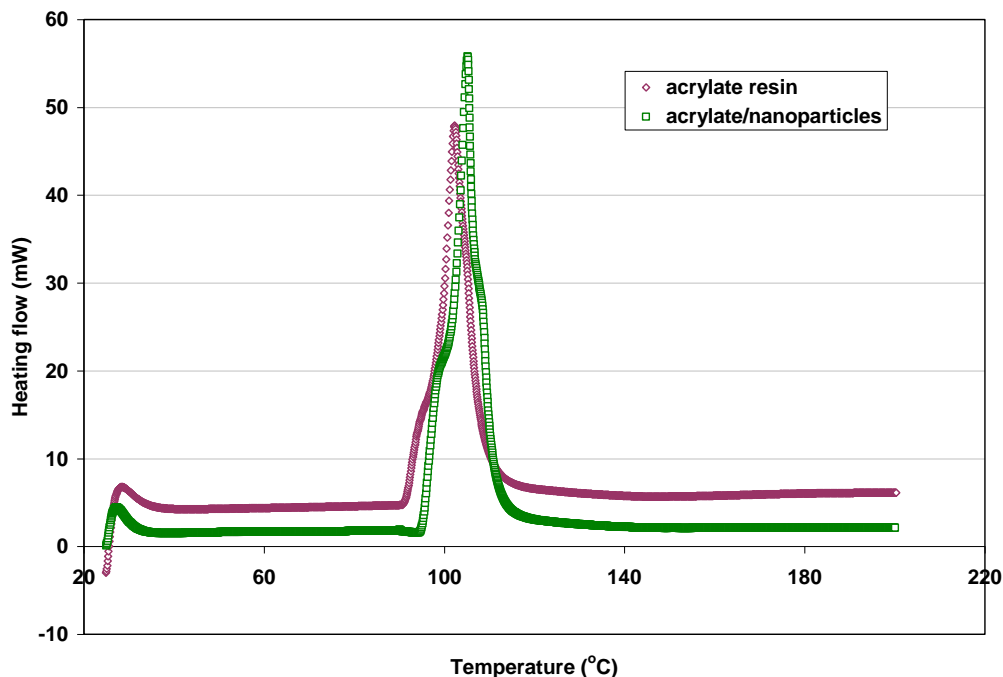


Fig. 3. DSC analysis showing the curing behaviour of HDDA/TMPTA (75/25 wt%)

Exothermic peaks were observed for both neat acrylate monomers and acrylate/nanoparticles. Similar to the neat acrylate resin and acrylate/nanoparticles, a lower peak temperature for neat acrylate impregnated maple wood was observed at 96 °C, while the acrylate/nanoparticles impregnated wood peak was observed at 104 °C (Table 1). The onset temperature of the reaction ranged from 85 °C to 102 °C, which indicated that the addition of nanoparticles shifted the polymerization onset temperature.

Table 1. DSC Characterization Results for Pure Acrylate Resin, Acrylate/Nanoparticles, Acrylate Impregnated Maple Wood, Acrylate/Nanoparticle Impregnated Maple Wood, EB Cure Impregnated Wood, and Wood Controls

Samples	Reaction energy (EA)	Integral energy of the peak (mJ)	Peak temperature (°C)	Onset temperature (°C)
Acrylate ¹	400.26	4036.15	102.24	98.2
acrylate /nano	349.07	4553.63	105.15	101.18
M-acrylate ²	454.62	1934.23	96.07	84.85
M-acrylate/nano	288.67	840.01	104.28	101.68
EB cure M ³	38.72	45.73	— ⁴	— ⁴
Maple control	94.39	44.11	— ⁴	— ⁴

¹ acrylate – HDDA/TMPTA (75/25)

² M-acrylate – HDDA/TMPTA (75/25) impregnated maple wood

³ EB cure M – EB cure impregnated maple wood

⁴ — No peak was observed (Fig. 5) for both control maple sample and EB cured impregnated maple samples

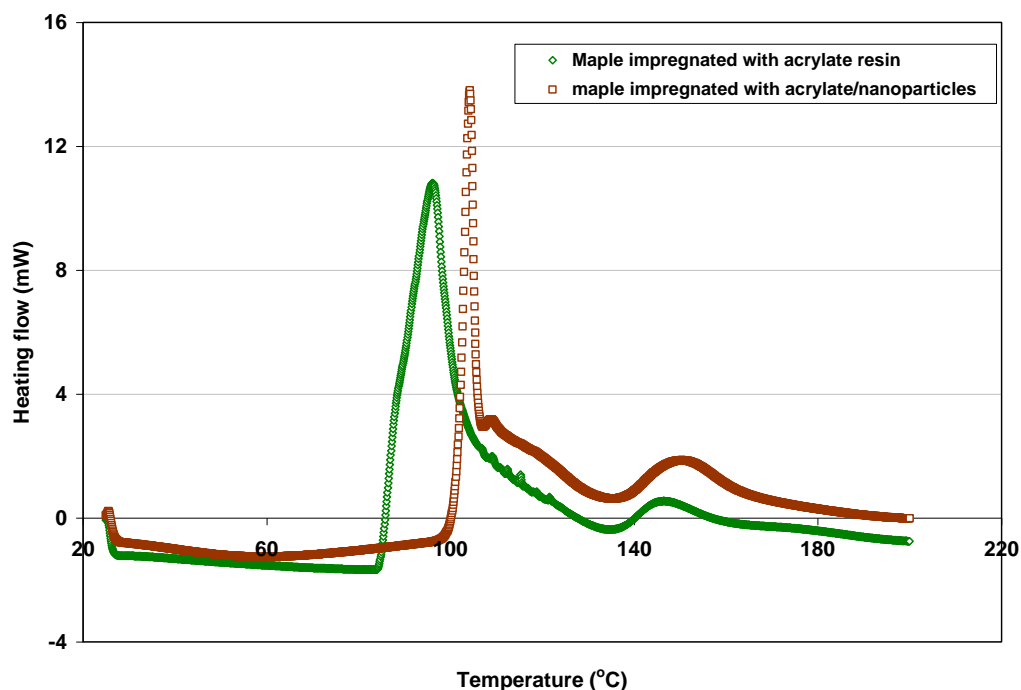


Fig. 4. DSC analysis showing the curing behaviour of maple engineered plank impregnated using acrylate monomer – HDDA/TMPTA (75/25 wt%), and acrylate monomer – HDDA/TMPTA (75/25 wt%) plus 1 wt% nanoparticles Claytone® APA

The EB-cured acrylate impregnated maple wood and control maple wood were characterized with DSC at the same test parameters as the uncured impregnated maple wood. The analysis results are presented in Fig. 5. No exothermic peaks were observed for both the neat EB-cured impregnated maple wood and the maple control wood. The DSC curves for the neat acrylate monomers, impregnated wood, EB-cured impregnated wood, and wood control samples are presented in Fig. 5. It can be observed that both the neat acrylate monomer and acrylate/nanoparticles showed big exothermic peaks as compared to the acrylate impregnated maple wood. The integral energy of the peaks (mJ) for the neat acrylate monomers, acrylate/nanoparticles, maple wood impregnated with acrylate monomers, maple wood impregnated with acrylate/nanoparticles, the EB-cured

impregnated maple wood, and the control maple wood are summarized in Table 1. The integral energy of the neat acrylate monomer peak was 4036.2 mJ, while the acrylate/nanoparticles was 4553.6 mJ, which was the highest peak as compared with all others. The integral energies were 1934.2 mJ and 840.0 mJ for the neat resin and acrylate/nanoparticles impregnated maple wood, respectively. For the EB-cured samples, the integral energy of the impregnated maple wood and that of the maple control sample showed, however, the lowest values of 45.7 mJ and 44.1 mJ, respectively, which are almost negligible as compared to those of the uncured neat resin and acrylate/nanoparticles impregnated wood samples. No exothermal peaks could be observed for both the EB-cured acrylate impregnated maple wood and the control maple wood samples, which implies that the resin curing was complete and no free monomer existed in the EB-cured acrylate impregnated wood. Further, the DSC curves of the EB-cured acrylate-impregnated maple wood and the control samples are almost overlapped, which confirms that the EB cure is an efficient curing method for acrylate and acrylate/nanoparticles impregnated engineered wood planks.

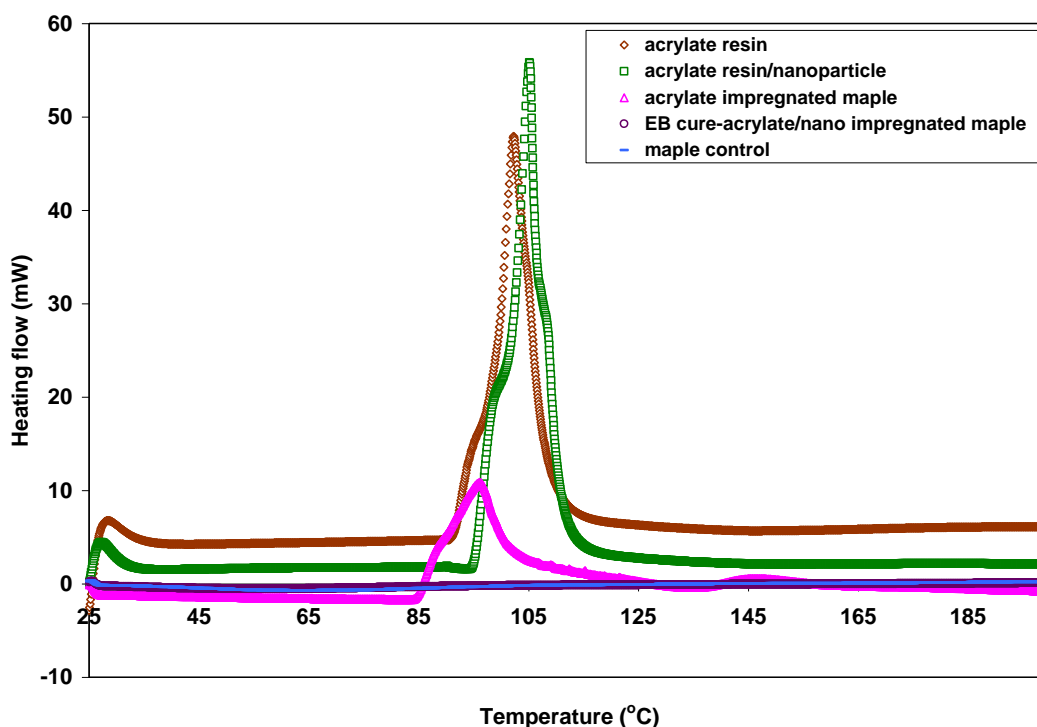


Fig. 5. Comparison of DSC characterization results of HDDA/TMPTA (75/25 wt%), HDDA/TMPTA (75/25 wt%) and 1 wt % nanoparticles, maple impregnated with HDDA/TMPTA, EB (electron beam)-cured impregnated maple sample, and pure maple sample without any treatment

SEM Characterization of EB-Cured Impregnated Maple Wood

The cross sections of the control maple wood samples, EB-cured neat acrylate monomer impregnated maple wood, and acrylate/nanoparticles impregnated maple wood samples were characterized by SEM observation to determine the morphology and location of the cured acrylate in the wood structure. The SEM pictures are presented in Fig. 6. Figure 6a is the control maple wood, Fig. 6b neat acrylate impregnated maple wood cured by EB, and Fig. 6c the acrylate/nanoparticle impregnated maple wood cured by EB irradiation. From Figs. 6b and 6c, it is observed that the void areas of the maple cross-section were

filled with cured acrylate polymers or cured acrylate/nanoparticle polymers, where the acrylate monomer was using as carrier medium for nanoparticles (Cai *et al.* 2010b) to form a three-dimensional network of the polymer/wood composite or nanocomposite within the wood.

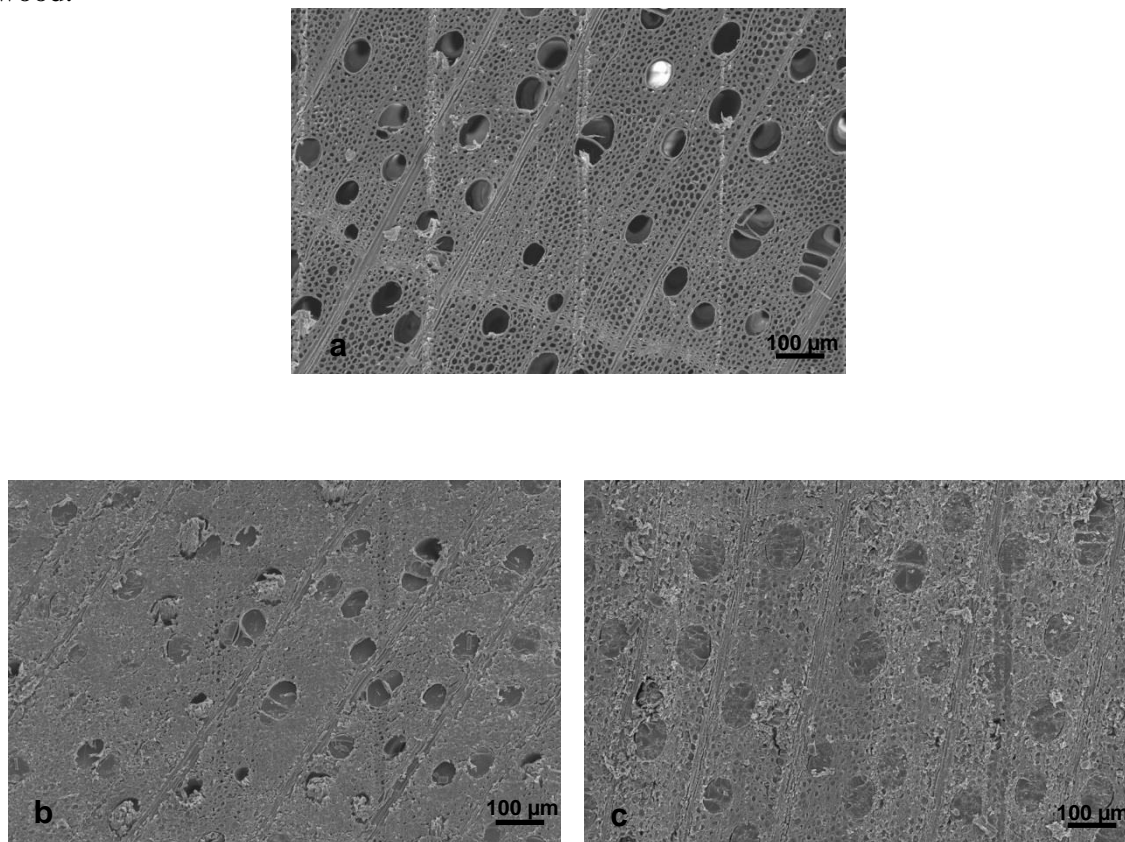


Fig. 6. Scanning electron micrographs of the cross-section of (a) maple controls, (b) EB polymerized maple wood impregnated with HDDA/TMPTA and 1% nanoparticles, and (c) EB polymerized maple wood impregnated with HDDA/TMPTA resin

Improvement of Brinell Surface Hardness and Impact Resistance

Surface hardness and impact resistance are two important properties for wood products, especially for some specific applications; for example, when used as wood flooring products. The improvement of surface hardness and impact resistance *via* a surface densification process are desirable, particularly for the wood products industry. Brinell surface hardness of the maple control samples and neat HDDA/TMPTA resin-impregnated maple wood cured by EB were measured according to standard EN1534 (2000) and the obtained results are presented in Fig. 7. For the control samples, the Brinell surface hardness was 5.2 MPa. The surface hardness was enhanced to 15.4 MPa for the EB-cured acrylate-impregnated maple wood with a 5 min vacuum process, which is a 197% improvement as compared to the control. The 10 min vacuum process and that of 15 min vacuum plus 15 min of pressure impregnation resulted in surface hardness of 14.8 MPa and 16.4 MPa, an improvement of 185% and 215%, respectively, after EB curing. It can be seen that the surface hardness of the EB-cured acrylate-impregnated maple wood was improved drastically.

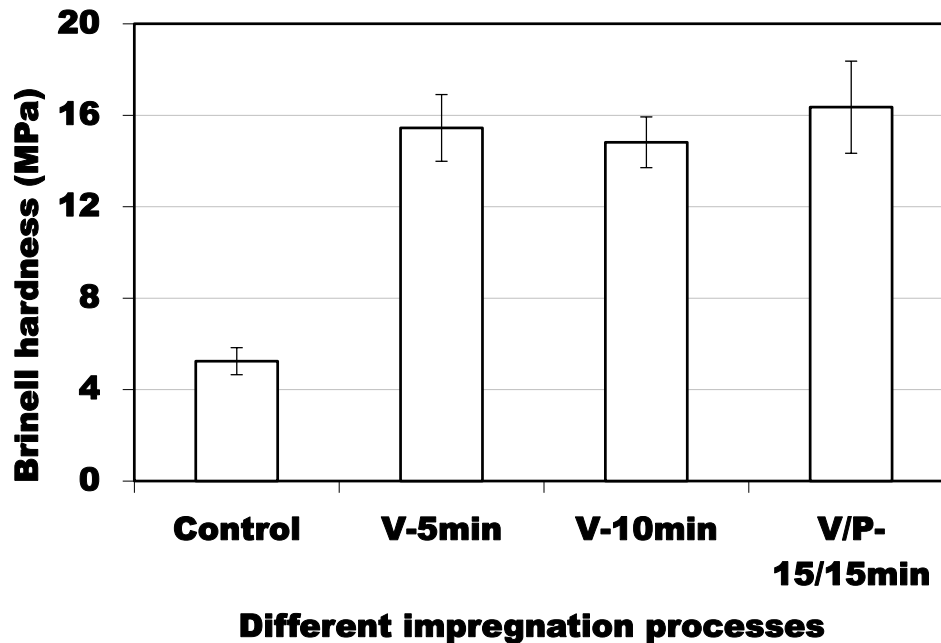


Fig. 7. Brinell surface hardness of the EB-cured HDDA/TMPTA-impregnated maple wood with different vacuum impregnation times

The impact resistance properties were measured with a lab standard setup practiced at the FPInnovations eastern lab. Both indentation depth and diameters were measured from three different impact strengths. The obtained testing results are summarized in Table 2.

For the indentation depth, the depth of the treated samples was significantly lower than that of the untreated control wood at the different impact levels. The indentation depths were 0.187 mm, 0.332 mm, and 0.485 mm for the untreated control maple wood from the different falling heights of 7.62 cm, 22.86 cm, and 45.72 cm, respectively. The depth values for the EB-cured acrylate-impregnated maple wood with the 5 min vacuum process were 0.043 mm, 0.053 mm, and 0.080 mm for the steel ball falling from 7.62 cm, 22.86 cm, and 45.72 cm, respectively. Similar results were observed for the other impregnation processes. This indicates that the indentation depth of the EB-cured acrylate-impregnated maple wood was decreased significantly compared to the untreated maple samples (classification of a and b group means statistically significantly different). A similar tendency regarding the impact indentation diameters were also observed.

For the untreated maple wood samples, an indentation diameter of 5.07 mm, 6.65 mm, and 7.77 mm was seen for the test with a ball falling height of 7.62 cm, 22.86 cm, and 45.72 cm, respectively. These values were 3.48 mm, 4.06 mm, and 4.96 mm for the EB-cured acrylate-impregnated maple wood (with similar 5 min vacuum impregnation process), significantly lower than those of the untreated maple wood. Both indentation depth and diameters were decreased significantly for the EB-cured acrylate-impregnated maple wood with different impregnation processes, which indicated that the impact resistances of the EB-cured surface densification wood products were improved drastically.

Table 2. Impact Resistance of EB-Cured Impregnated Sugar Maple Samples

Impact resistance	Indentation depth (mm)			Indentation diameter (mm)		
	Light impact force (falling from 7.62 cm)	Medium impact force (falling from 22.86 cm)	High impact force (falling from 45.72 cm)	Light impact force (falling from 7.62 cm)	Medium impact force (falling from 22.86 cm)	High impact force (falling from 45.72 cm)
Control	0.187 a	0.332 a	0.485 a	5.07 a	6.65 a	7.77 a
V-5 min	0.043 b	0.053 b	0.080 b	3.48 b	4.06 a	4.96 b
V-10 min	0.040 b	0.057 b	0.084 b	3.48 b	4.43 b	5.31 b
V/P-15/15min	0.041 b	0.056 b	0.090 b	3.31 b	4.16 b	5.04 b

CONCLUSIONS

1. This work studied the feasibility of developing an online EB curing process to polymerize chemical surface-densified wood to replace the traditional slow thermal/pressurization curing process. Low viscosity HDDA/TMPTA-impregnated maple wood with a chemical retention of 62 to 64 wt% was cured by EB irradiation.
2. The EB-cured acrylate-impregnated maple wood, acrylate and acrylate/nanoparticle impregnated wood, neat acrylate resin, and acrylate/nanoparticles were characterized by DSC analysis to validate the polymerization reaction of the acrylate impregnated into the maple wood. Polymerization exothermal peaks were observed for the neat acrylate resin, formulation of acrylate/nanoparticles, and the acrylate/nanoparticles-impregnated maple samples. No polymerization exothermal peaks were observed for both the EB-cured impregnated maple and control maple samples. The DSC characterization curves of the EB-cured impregnated maple and control samples were almost overlapped when compared with the neat resin and impregnated maple samples, which confirmed that EB irradiation is an efficient curing method to polymerize acrylate-impregnated wood surface densification products.
3. The surface hardness and impact resistance properties of the EB-cured acrylate/nanoparticles-impregnated maple wood were drastically improved as compared to the untreated maple wood. The surface hardness was improved up to 200% of that of the untreated maple wood.
4. The cross section of the EB-cured acrylate/nanoparticles-impregnated maple wood was characterized by scanning electron microscopy, and it was confirmed that the EB-cured acrylate polymer was distributed into the voids and lumen of the maple structure.
5. The results of this work demonstrated that EB curing might be an efficient alternative instant curing process to cure chemically impregnated wood products.

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