

SURFACE AND INTERFACIAL CHARACTERIZATION OF WOOD-PVC COMPOSITES: THERMAL AND DYNAMIC MECHANICAL PROPERTIES¹

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ABSTRACT

Maleation significantly influenced thermal and dynamic mechanical properties of resultant wood-PVC composites. Experimental results indicated that storage modulus (E') and complex modulus (E^*) increased with increase of maleated polypropylene (MAPP) retention at low MAPP retention for both E-43 and G-3015, but leveled off or decreased at high MAPP retention. However, $\tan\delta$ was independent of MAPP retention. Interfacial bonding strength was related to these moduli at low MAPP retention levels, but the correlation between interfacial bonding strength and moduli was not so significant at high MAPP retention. Compared with wood, PVC, and untreated composites, maleated wood-PVC composites had significant shifts in most DMA, TGA, and DSC spectra due to chemical coupling by MAPP at the interface.

Keywords: Dynamic mechanical properties, maleation, thermal analysis, wood-PVC composites.

INTRODUCTION

Chemical coupling agents usually act as a bridge to link polar wood fiber and non-polar thermoplastics. This helps transfer the stresses between wood and thermoplastics, thus improving the interfacial bonding strength in wood-polymer composites (WPC) (Woodhams et al. 1984; Dalvåg et al. 1985). The coupling forms include covalent bonds, secondary bonding (such as hydrogen bonding and van der Waals'

forces), polymer molecular entanglement, and mechanical interlocking. Although the coupling action in wood and polymer composites is complicated, the primary forms of covalent bonds for coupling agents are esterification, etherification, carbamation, and carbon-carbon bonding (Lu et al. 2000). Coupling agents (such as maleated polypropylene) create a new structure at the interface, which influences morphology, crystallization, rheology, mechanical, thermal, and other properties of wood-polymer composites (Rowell 1991; Kolosick et al. 1992; Quillin et al. 1993; Collier et al. 1996).

Thermal analysis has been extensively applied to investigate the thermal behavior of various materials as a function of temperature

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(Hatakeyama and Quinn 1994). A number of papers (Oksman and Lindberg 1995; Simonsen and Rials 1996) have reported researches on thermal properties of WPCs. Crystallization and morphology in WPCs have been investigated with many thermal methods by a number of research workers (Felix and Gatenholm 1994; Ying et al. 1999). Weight or volume ratios of wood fiber greatly influenced glass transition temperatures (T_g) and storage moduli (E') of the resultant composites (Gatenholm et al. 1993). Effects of fiber content and dimensional size and interphase modified with MAPP on dynamic mechanical behavior of wood flour/kenaf fiber-polypropylene composites have been reported (Sanadi et al. 1999). However, it is not clear how the interphase influences the thermal behavior of resultant wood-polymer composites and whether there is any relationship between coupling agent performance and thermal properties.

As a continuation of our earlier paper on the influence of maleation on graft polymerization, wettability, and interfacial adhesion in wood-PVC composites (Lu et al. 2002), thermal properties of wood-PVC composites with chemical coupling were investigated in this work. The objectives of this study were to investigate thermal characteristics of maleated wood-PVC composites and the relationship between thermal properties and coupling agent performance in resultant composites.

EXPERIMENTAL

Materials

Yellow poplar veneer (610 mm \times 610 mm \times 0.91 mm) was obtained by Columbia Forest Products Co., VT. Wood veneer was cut into 50.8 mm by 25.4 mm in size. Moisture content of all wood specimens was between 5% and 6%. Clean and rigid polyvinyl chloride sheets (508 mm \times 1,270 mm \times 0.0762 mm) with a density of 1,390 kg/m³ were purchased from Curbell Plastics Co., AZ. The glass transition and melting temperatures of the PVC sheets are 81°C and 175°C, respectively. They have a tensile strength

of 55 MPa and a tensile modulus of 2,800 MPa (Delassus and Whiteman 1999). Before manufacture of wood-PVC composites, PVC sheets were cut into a dimension of 25.4 mm by 12.7 mm for shear testing and 50.8 mm by 25.4 mm for DMA testing, respectively.

Two maleated polypropylenes (MAPPs), Epolene E-43 and Epolene G-3015, were used as coupling agents. E-43 has an average weight molar mass (M_w) of 9,100, and its acid number is between 40 and 55. G-3015 has a high molecular weight of 47,000 g/mol, but has a low acid number (between 12 and 18). E-43 contains more maleic anhydride groups than G-3015. Benzoyl peroxide (BPO) was used as an initiator, and toluene was used as solvent for both MAPPs.

Soxhlet extraction

Soxhlet extraction was conducted on all wood specimens according to the ASTM standard D1105-96 (ASTM 1998) to reduce the influence of extractives on chemical coupling. The wood samples were first extracted with a 120-ml mixing solution of toluene and ethyl alcohol for 4 h. They sequentially underwent the second extraction with 120 ml ethyl alcohol for 4 h. The extracted wood specimens were finally oven-dried at 70°C for 24 h to reach a constant weight. The oven-dried weight of each sample was measured.

Coupling treatments

The procedure of coupling treatment for wood specimens was described in the literature (Lu et al. 2002). Wood specimens were dipped in coupling solution (a mixture of MAPP, BPO, and toluene) at 100°C for 5 min under continuous stirring with a magnetic stirrer. MAPP concentration levels in the coupling solution were designed to be 0, 12.5, 25, 50, and 75 g/L. The weight ratio between BPO and MAPP was 0.5. The treated specimens were removed from the solution and cooled to room temperature. All treated specimens were finally oven-dried at 70°C for 24 h to reach a constant weight. Retention of coupling agent on treated wood speci-

mens was calculated following the procedure outlined by Lu et al. (2002).

Manufacture of wood-PVC composites

In order to create a wood-PVC laminate, a piece of PVC sheet was inserted between two MAPP-treated wood specimens. The assembly was temporarily fixed with two pieces of narrow Scotch tape on each side. The assembly was then hot-pressed with a small-scale press under a pressure of 0.276 MPa for a shear specimen and under a pressure of 0.552 MPa for a DMA specimen. The pressing cycle for the wood-PVC assembly consisted of 3-min heating at 178°C and 1-min cooling under pressure. At the end of the heating period, the press platens were cooled with tap water to 70°C. The laminate was allowed to cool to room temperature.

Shear strength measurement

Shear tests were conducted with an INSTRON machine (Model 1125) according to the ASTM standard D3165 (ASTM 1993). Two mechanical tensile grips were used to clamp the sample to the loading frame. The span between the two clamps was 50.8 mm. Each sample was tested to failure at a loading speed of 2.54 mm/min. Shear strength (Pa) was calculated as a ratio of the maximum failure load (N) to the bonding area (m²).

Thermal analysis

Dynamic mechanical analysis (DMA).—A DMA system (Seiko DMS 110) was used to conduct dynamic mechanical analysis for maleated wood-PVC composites. The specimen size was 50 mm by 12 mm. The DMA testing procedure consisted of three cycles: first heating, first cooling, and second heating (Table 1).

A test specimen was subjected to sinusoidal stress under a three-point bending mode. The span between the load and each supporting point was 20 mm. The oscillating frequencies of the load acting on the specimens were 0.01 Hz, 0.1 Hz, 1 Hz, 10 Hz, and 100 Hz. Testing tempera-

TABLE 1. DMA test cycles for wood, PVC, and wood-PVC composites.

Specimen	Test mode	Test cycle	Temperature (°C)		Rate (°C/min)
			Start	Stop	
Wood	Bending	First heating	20	220	0.50
		First cooling	220	30	0.25
		Second heating	30	220	0.50
PVC	Bending	First heating	20	100	0.50
		First cooling	100	30	0.25
		Second heating	30	100	0.50
Wood-PVC composites	Bending	First heating	20	150	0.50
		First cooling	150	30	0.25
		Second heating	30	150	0.50

tures changed according to test materials. Starting from room temperature, the maximum heating temperature was 220°C for wood, 100°C for PVC, and 150°C for wood-PVC composites. The heating rate was 0.5°C/min, while the cooling rate was 0.25°C/min.

Thermogravimetric analysis (TGA).—A modulated thermogravimetric analyzer (TA Instruments TGA2950) was used to characterize the decomposition and thermal stability of maleated wood-PVC composites. A specimen was first placed into a Seiko aluminum sample pan on the platinum basket in the furnace, and then heated from room temperature to 600°C. The heating rate was 5°C/min. During testing, the heating unit was flushed under a continuous nitrogen flow at a pressure of 8 KPa. To separate possible overlapping reactions during measurements, derivative thermogravimetric (DTG) analysis was also conducted to measure the mass change of a specimen with respect to temperature (dm/dT) using the same TGA system.

Differential scanning calorimetry (DSC).—A modulated DSC analyzer (TA Instruments DSC Q100) was used to determine the thermal complex transitions of maleated wood-PVC composites. A specimen pressed into an aluminum sample pan was placed into the heating chamber for DSC. For comparisons of wood, PVC, and resultant composites, the maximum temperature was controlled at 150°C for all composite specimens. The heating rate was 5°C/min. During measurements, the heating chamber was flushed with a continuous nitrogen flow at a pressure of

8 KPa. Each specimen was measured twice. For separating possible overlapping brands, a derivative DSC (DDSC) was applied to help analyze DSC spectra.

In order to remove the blocking effect of wood veneer on DSC spectra, a wood-PVC composite sample was delaminated with a sharp knife into a PVC film and two pieces of wood veneer to help investigate characteristics of esterification in DSC spectra. The maximum testing temperature for PVC-coupling agent interphases was less than 220°C, while the maximum testing temperature for wood-coupling agent interphases was controlled to be 400°C.

RESULTS AND DISCUSSION

Primary thermal and mechanical properties of wood-PVC composites are summarized in Table 2. Retention and graft rate of MAPP on wood after coupling treatment are listed in the literature (Lu et al. 2002).

Dynamic mechanical analysis

Wood-PVC composites presented different thermal behavior from wood and PVC. As shown in Table 2 and Fig. 1, the glass transition

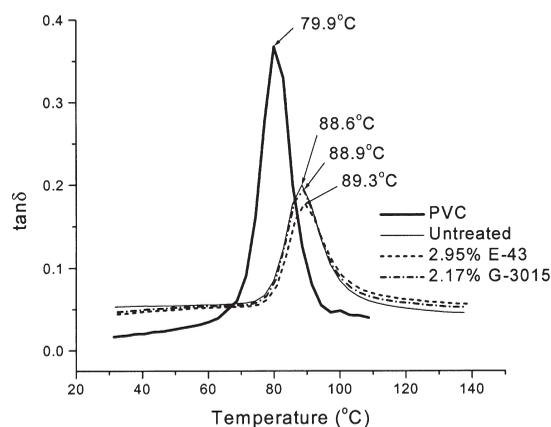


FIG. 1. Glass transitions of maleated wood-PVC composites in comparison with PVC in the second heating at a frequency of 1 Hz.

(T_g) of PVC was about 80°C, while T_g of yellow poplar was close to 150°C. From the second heating, T_g of maleated wood-PVC composites was around 89°C at the frequency of 1 Hz. Therefore, T_g of maleated wood-PVC composites was between those of wood and PVC.

Frequency of the oscillating load greatly influenced T_g of maleated wood-PVC composites (Fig. 2). From the second heating, the glass transition of wood-PVC composites with 6.83% E-

TABLE 2. Thermal and mechanical properties of wood-PVC composites.

Material ^a	Shear strength (MPa)	E' (GPa) ^b	E'' (GPa) ^b	tan δ ^b	T _m (III) (°C) ^c	T _g (II) (°C) ^{c,d}	ΔH at T _m (III) (J/g)	ΔH at T _g (II) (J/g)	TG at 600°C (%)
PVC	—	5.73	0.44	0.39	—	77	0	0	10.2
Wood veneer	—	10.43	0.41	0.05	—	151	0	0.003025	13.6
WPC with									
0% E-43	3.14	7.85	1.04	0.22	—	151	0	0.003025	16.8
2.95% E-43	2.90	7.96	0.97	0.22	153	141	0.08328	0.01684	17.9
4.12% E-43	3.03	9.45	1.23	0.24	152	140	0.2322	0.07899	18.2
6.83% E-43	3.32	9.16	1.15	0.23	146	133	0.2840	0.05358	16.5
7.41% E-43	3.71	—	—	—	147	134	0.3499	0.1009	—
0% G-3015	3.14	7.85	1.04	0.22	—	151	0	0.003025	16.8
2.17% G-3015	2.90	7.08	0.8	0.23	159	149	0.04766	0.01867	16.5
3.86% G-3015	2.94	8.98	1.16	0.24	157	148	0.07654	0.01561	15.4
6.35% G-3015	3.61	8.56	1.09	0.24	151	141	0.1204	0.1041	17.0
10.54% G-3015	3.85	—	—	—	152	141	0.4917	0.3300	—

^a Values in this column were retention of coupling agents.

^b All values were measured in first heating at 1 Hz.

^c The values were based on wood-MAPP interphases.

^d The value at the first and second row was glass transition of PVC and wood, respectively, and others were related to T_g(II).

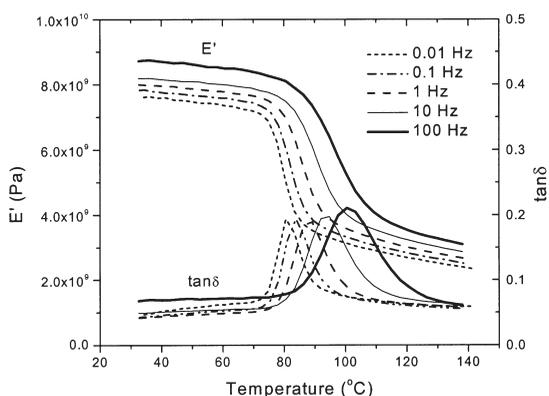


FIG. 2. Influence of frequency on storage modulus and $\tan\delta$ of wood-PVC composites with 6.83% E-43 at the second heating.

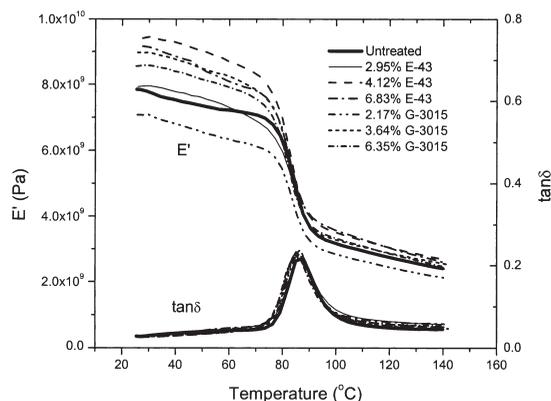


FIG. 3. Influence of maleation on storage modulus E' and phase angle $\tan\delta$ of wood-PVC composites in the first heating at a frequency of 1 Hz.

43 shifted about 20°C from 0.01 Hz to 100 Hz. Similar trends also occurred in wood-PVC composites with other MAPP retention levels in the same heating procedure and composites with all retention levels in other two heating procedures. Thus, the larger the frequency used, the higher the glass transition of maleated wood-PVC composites.

Storage modulus E' increased with increase of MAPP retention (Table 2). At low MAPP retention levels, E' of maleated wood-PVC composites was lower than, or close to, that of composites without maleation (Fig. 2). When MAPP retention levels were over 3%, E' was as high as 9 GPa. The modulus was lower than 9 GPa as the MAPP retention level was larger than 6% for both coupling agents. However, MAPP retention did not significantly influence the glass transition of wood-PVC composites. Composites treated with MAPP had almost the same glass transition temperature (Fig. 3). The value of $\tan\delta$ for wood-PVC composites was about half that of PVC and much larger than that of wood (Table 2). There was no significant difference in $\tan\delta$ for composites with and without maleation. Therefore, $\tan\delta$ was independent of MAPP retention and graft rate.

Interfacial bonding strength of wood-PVC composites increased with increase of storage modulus E' and complex modulus E^* at low MAPP retention levels and graft rate. However,

interfacial bonding strength was not so sensitive to these moduli at high retention levels and graft rate. This indicated that interfacial adhesion was more closely related to MAPP retention, graft rate, and the interfacial structure (Lu et al. 2002).

Thermogravimetric analysis

According to the TG spectra (Fig. 4a), PVC underwent two separate degradation steps under heating. PVC was stable at low temperatures. At about 250°C, its thermogravimetric percentage (TG%) drastically dropped to 50%. It gradually decreased before 400°C. There was another drastic drop at around 410°C. TG% of PVC decreased to about 15% and was stable until the temperature reached 600°C. Wood also presented two separate decomposition procedures. The first decomposition occurred at around 80°C. This was because of water evaporation from wood. The moisture content of wood accounted for about 5–6% based on weight. Wood started degrading at 250°C. The second procedure was slow and gradual. TG% of wood finally dropped to about 14% at 600°C (Fig. 4a).

Thermal decomposition behavior of wood-PVC composites was closer to that of wood (Fig. 4a). Since PVC accounted for a very small weight proportion (<5%) in composites, wood-PVC composites acted more like wood. Similar to wood, wood-PVC composites also had a grad-

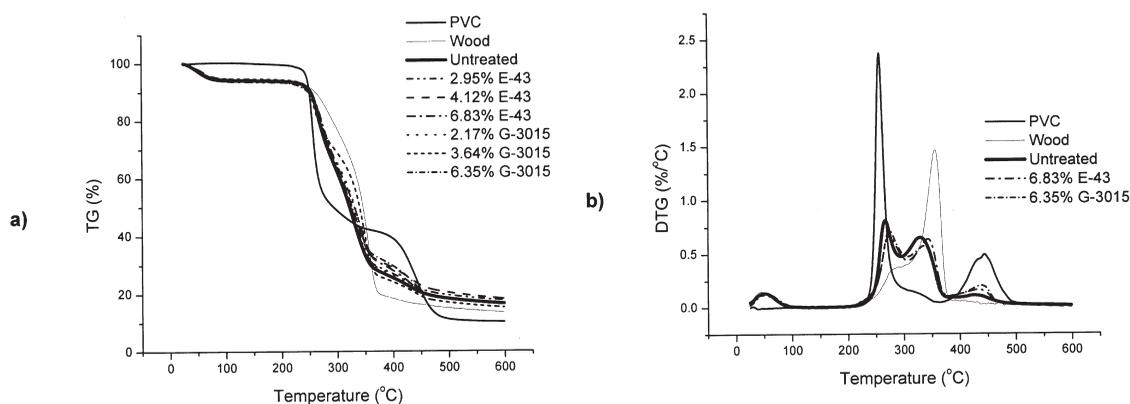


Fig. 4. Influence of maleation on decomposition of wood-PVC composites. a) TG and b) DTG.

ual decomposition procedure. The decomposition behavior of maleated wood-PVC composites was close to that without maleation at low temperatures (<250°C). However, TG curves of maleated composites deviated from those without maleation after temperatures were greater than 300°C. Particularly, composites with 3.64% G-3015 presented a distinguished deviation compared with G-3015-treated composites at other retention levels and composites with all E-43-treated composites.

According to the DTG spectra (Fig. 4b), PVC had a large and sharp DTG peak at 250°C and a small and broad peak at about 450°C. For wood, a small and broad peak occurred at 50°C, and a big and sharp peak at 350°C. The latter peak had a broad base, indicating that another peak at around 275°C overlapped with it. These four composite DTG peaks of wood and PVC were all presented in the DTG spectra of wood-PVC composites, but the second and the third peaks overlapped. The first and the third peaks were the characteristics of wood, while the second and the fourth were from PVC.

Like wood, wood-PVC composites had a small and broad DTG peak at around 50°C due to dehydration of wood. In the temperature range between 200°C and 400°C, the overlapping peaks of wood-PVC composites had a smaller DTG value (less than 1% per degree) than those of PVC and wood. Maleation made the overlapping DTG peaks shift right, compared with un-

treated composites (Fig. 4b). The second DTG peak of PVC at around 420°C was very weak in the DTG spectra of wood-PVC composites. According to Fig. 4, maleated wood-PVC composites had a smaller weight-degrading speed in the temperature range between 100°C and 600°C. This indicated that maleated wood-PVC composites had improved thermal stability compared with PVC and wood. However, there was no significant difference in thermal stability between maleated and unmaleated composites.

Differential scanning calorimetry

As shown in Fig. 5, the glass transition of PVC was 77°C, close to the result from DMA. For wood, a broad endothermic peak appeared at 80°C because of water evaporation from wood. For maleated composites, a similar peak also occurred in the DSC spectra. However, the spectra of E-43-treated composites had a significant shift to the right side compared with those of G-3015-treated composites. Because E-43 has more maleic anhydride (MA) groups in its molecular backbone than G-3015, many ungrafted or non-reacted MA groups may exist at the interface after manufacture (Lu et al. 2002). This shifting may be caused by these ungrafted or non-reacted MA groups at the interface. Therefore, the glass transition of wood-PVC composites cannot be easily detected due to the interference of water evaporation in wood and

the presence of single and double carboxylic acids from ungrafted MA groups at the interface.

On the DDSC spectra, E-43- and G-3015-treated composites presented similar thermal behavior (Fig. 6). Maleated composites had a small peak at T_m(IV) in the range between 50°C and 60°C. T_m(IV) was the melting temperature (T_m) of MA groups (CRC Press 2000). This indicated that there were ungrafted or free MA groups at the interface after coupling treatment. A weak band was presented at around 77°C in which T_g(I) was the glass transition (T_g) temperature of

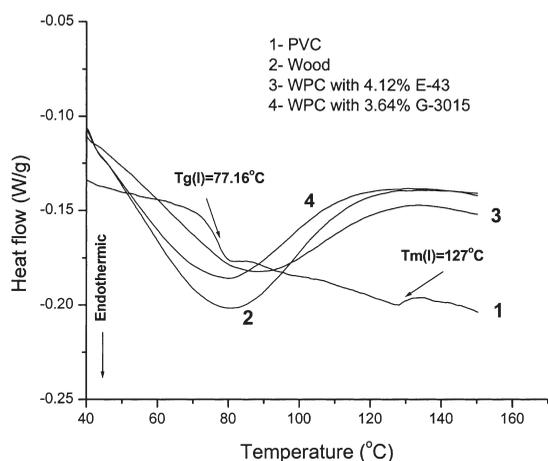


FIG. 5. Influence of wood and its moisture content on DSC spectra of wood-PVC composites.

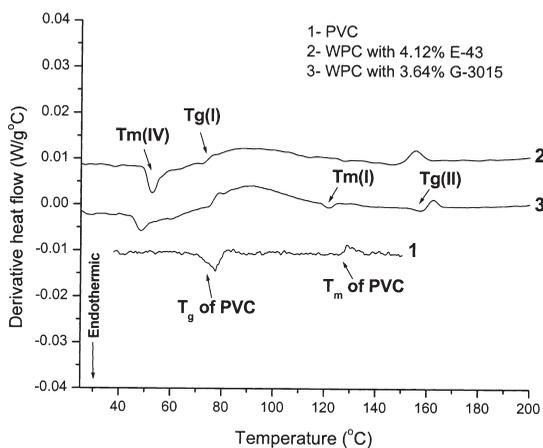


FIG. 6. DDSC spectra of maleated wood-PVC composites.

PVC. T_g(II) was presented at around 157°C for G-3015-treated composites. It was the glass transition of wood (i.e., T_g of lignin). For E-43-treated composites, T_g(II) was shifted left to around 150°C. T_m of PVC appeared as T_m(I) at around 125°C, but it shifted left to 123°C on the spectra of maleated composites. This denoted that maleation had significant influence on thermal behavior of resultant composites.

PVC film separated from composites presented the DSC spectra of PVC-coupling agent interphases (Fig. 7). T_g and T_m of PVC were 77°C and 125°C, respectively. For E-43, an endothermic peak appeared at around 70°C, perhaps because single and double carboxyl groups on the molecular chains of E-43 were transferred as MA groups by dehydration. All E-43-treated composites presented T_g of PVC at around 80°C. However, T_m of PVC shifted left, and the larger the MAPP retention, the larger the shifting (Fig. 7). Although E-43 had a strong band of melting temperature at around 160°C, composites with low E-43 retention did not present this feature. This peak was still weak even when E-43 retention levels were over 4%. It might indicate that there were few coupling reactions between MAPP and PVC.

DSC spectra of wood-coupling interphases with E-43 and G-3015 are presented in Fig. 8.

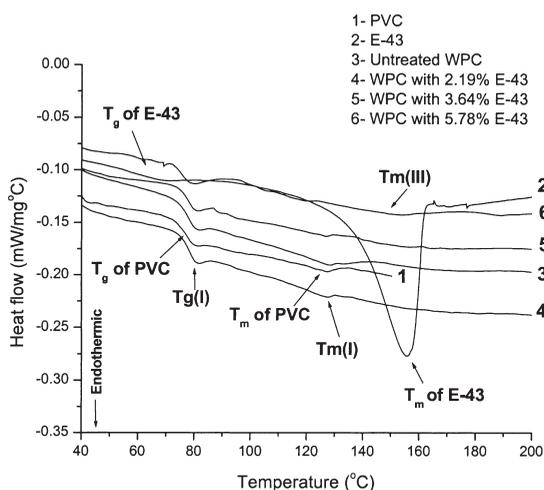


FIG. 7. DSC spectra of PVC-MAPP interphases.

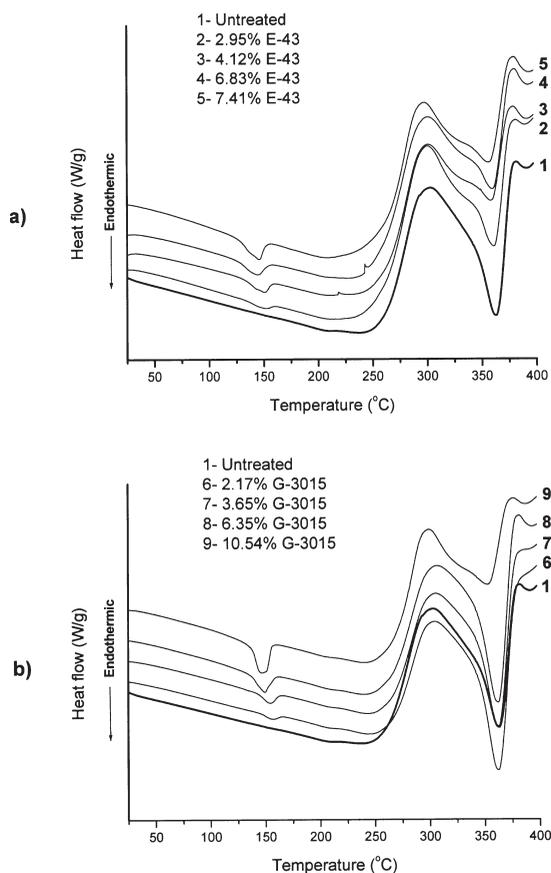


FIG. 8. DSC curves of wood-MAPP interphases. a) wood-E-43 interphases and b) wood-G-3015 interphases.

On DSC curves, a coupling agent-wood interphase had four endothermic peaks and three exothermic peaks. The composite peaks at around 150°C were signals from T_g of wood and T_m of MAPP. The broad peaks at around 250°C were probably due to the pyrolyzation of hemicellulose and lignin (White and Diitenberger 2001). The small bands at around 320°C might be responsible for the chain scissor of MAPP under high temperatures, while the big and sharp peaks at around 360°C were due to depolymerization of cellulose. The first two exothermal peaks were mainly attributed to exothermic reaction of exposed char and volatiles with environmental oxygen. The third exothermic peak was due to the cleavage of carbon-carbon linkage be-

tween lignin structural units (White and Diitenberger 2001). Peak areas at 150°C and 350°C varied with MAPP retention, but those at 200°C and 250°C were almost independent of MAPP retention. The peak area at 150°C was proportional to MAPP retention, but that at 350°C decreased with increase of MAPP retention levels. For both E-43 and G-3015, the bands at about 150°C shifted left with increase of MAPP retention (Fig. 8).

According to DDSC spectra of wood-coupling interphases, E-43- and G-3015-treated composites had five endothermic bands in the temperature range between 25°C and 375°C (Fig. 9). The first band on DSC spectra was divided into two peaks on DDSC curves. By en-

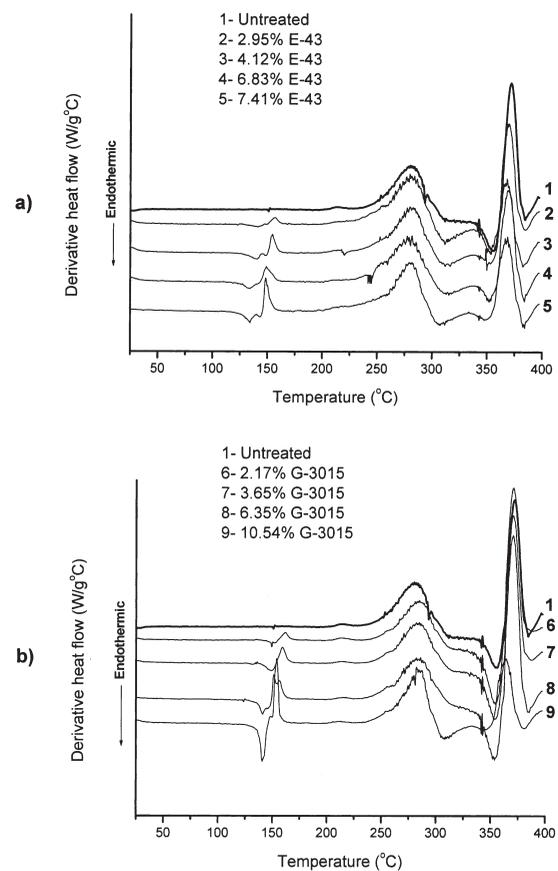


FIG. 9. DDSC spectra of wood-MAPP interphases. a) wood-E-43 interphases and b) wood-G-3015 interphases.

larging these DSC and DDSC spectra, overlapped regions at around 140°C–160°C were separated into two individual peaks (Fig. 10). T_g(II) at the first peak between 140°C and 150°C was T_g of wood, but T_m(III) at the second peak between 150°C and 160°C, T_m of MAPP. For both E-43 and G-3015, T_g(II) and T_m(III) had a left shift with increase of coupling agent retention. For example, T_g(II) decreased from 141°C to 134°C as E-43 retention increased from 2.95% to 7.41%. For G-3015-wood interphases, T_g(II) had a smaller decrease with increase of G-3015 retention (Table 2). Enthalpy (ΔH) values at T_g(II) and T_m(III) increased with increase of coupling agent retention. For wood-E-43 interphases, ΔH values at T_g(II) increased

from 0.003025 to 0.1009 J/g, and ΔH values at T_m(III) increased from 0 to 0.3449 J/g as E-43 retention increased from 0% to 7.41%. At T_g(II), wood-G-3015 interphases had higher enthalpy than wood-E-43 interphases at the same retention level (Table 2). However, wood-G-3015 interphases had lower enthalpy values at T_m(III) than wood-E-43 interphases at the same retention level. Therefore, the molecular structure of coupling agents influenced the thermal behavior of resultant composites.

CONCLUSIONS

Maleation significantly influenced thermal and dynamic mechanical properties of wood-

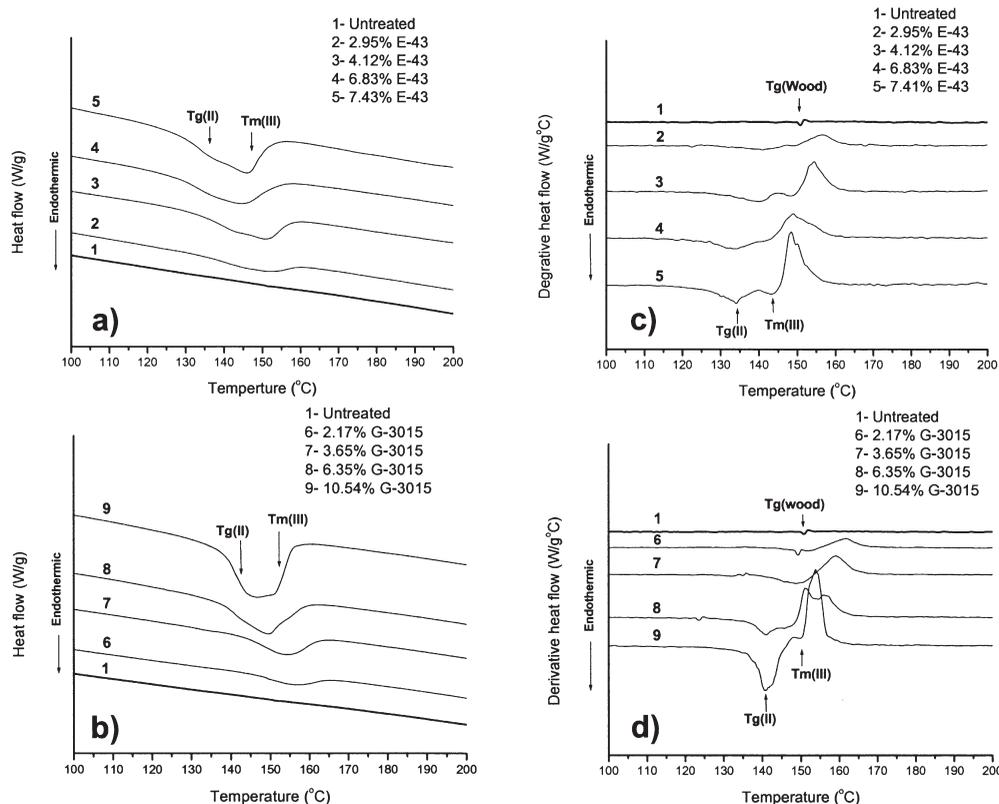


FIG. 10. DSC and DDSC curves of wood-MAPP interphases in a temperature range between 100°C and 200°C. a) and c) wood-E-43 interphases and b) and d) wood-G-3015 interphases.

PVC composites. Storage modulus E' and complex modulus E^* of maleated wood-PVC composites increased with increase of MAPP retention. There was a small decrease of E' and E^* at high retention levels. However, $\tan\delta$ was independent of MAPP retention. Interfacial bonding strength was related to these moduli at low MAPP retention levels. However, the correlation between the strength and moduli was not so significant at high MAPP retention. T_g of maleated composites was around 89°C, between T_g values of PVC and wood.

Maleated wood-PVC composites had a larger TG% at 600°C than PVC and wood. In the temperature range between 100°C and 600°C, maleated wood-PVC composites had lower DTG% compared with wood and PVC. Therefore, maleated wood-PVC composites had better thermal stability than wood and PVC under high temperatures. However, there was no significant difference in thermal stability between maleated and unmaleated composites

Compared with wood, PVC, and untreated wood-PVC composites, maleated wood-PVC composites had significant shifts in most DMA, TGA, and DSC spectra. On most DSC spectra, T_g of wood and T_m of MAPP for maleated wood-PVC composites had a left shift at coupling agent-wood interphases with increase of coupling agent retention compared with wood veneer. However, T_g of PVC was almost independent of coupling agent retention. This shift was mainly due to chemical coupling by MAPP at the interface. Therefore, maleated wood-PVC composites had thermal behavior different from wood, PVC, and untreated wood-PVC composites.

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