

Influence of Cure Conditions on the Properties of a One-Component Moisture-Cured Polyurethane Adhesive in the Context of Green Gluing of Wood

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ABSTRACT: A commercial one-component moisture-cured polyurethane-urea wood adhesive was investigated under different curing environments to simulate parameters during green gluing, that is, gluing of freshly sawn and undried timber. This process is an eco-efficient and waste eliminating process in which only the finished wood product properties have been tested; however, not the adhesive itself. Therefore, the effect of moisture and postcuring heat treatment on the adhesive properties such as cure, chemical, and physical characteristics, and adhesion to wood were studied. It was determined by rheometry that the water content was proportional to the time to

gel point, with moisture content of 2.6–5.6 wt % water, resulting in a higher initial storage modulus of the adhesive. Additionally, it was found that the strength of the wet glued bonds was significantly higher after the heat treatment, corresponding to the increase in ordered bidentate groups (Fourier transform infrared spectroscopy), higher storage modulus (rheometry), and higher T_g (dynamic mechanical thermal analysis). © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 000: 000–000, 2012

Key words: moisture curing adhesives; polyurethane adhesive; wood adhesive; green gluing; wet gluing

INTRODUCTION

One-component moisture-cured polyurethane-ureas (MCPUs),¹ made up of isocyanate-terminated prepolymers, were widely patented in the mid-1980s² and have become widespread in wood applications, both for nonstructural use and as certified structural adhesives.³ MCPUs wood adhesives for structural use are often based on prepolymerized methylene diphenyl diisocyanate (MDI) combined with polyols. The uncured adhesive contains free isocyanate groups and polyurethane groups. Curing of a one-component MCPUs adhesive occurs when the isocyanate is able to react with available water (in air and/or in contact with a substrate such as wood) to form carbamic acid. Carbamic acid dissociates into an amine that can form a urea bond after reacting further with an isocyanate group. Carbon dioxide, which is emitted during this reaction, can cause foaming of the ad-

hesive, risking the creation of cavities in the adhesive.⁴ Polyols used in MCPUs can also be terminated with different reactive groups to obtain the desired properties in the cured adhesive.^{5,6} Thus, in cured MCPUs adhesives, different interactions, mainly hydrogen bonding (H-bonding) and crosslinking are created between polyols, urethane, and urea groups.

In wood applications, MCPUs-containing polymeric MDI has been used for its ability to bond with the water and lignocellulose hydroxyl groups contained in wood, creating both a mechanical and a chemical bond that is tough, has high strength, and is water resistant.^{7,8} Further, the final mechanical properties, for example strength, and excess residual isocyanate content in the final bond, are dependent on the cure conditions such as temperature and relative humidity. These cure conditions can significantly affect the physical properties and the adhesion, owing to microscopic phase separation resulting in differences between intra- and intersegment interactions such as hydrogen bonding, mixing of soft (SS) and hard (HS) segments, and the degree of crystallinity of these segments.^{9–12}

Based on the conventional technology, timber is usually glued at a moisture content (MC) far below the fibre saturation point, preferably at a MC between 6 and 15 wt %.⁵ The chosen level of MC depends on the end use climate conditions of the

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product. For example, furniture components should be glued at 8 wt % MC and structural timber at 12–15% MC. During the last 20 years, new technology called green gluing has been developed, which involves gluing of undried (unseasoned) freshly sawn timber. Green timber has considerably higher MC, varying typically between 60 and 180 wt % of the dry wood weight.^{13–16} In processes involving green gluing with MCPU adhesives, timber is glued at room temperature and then subjected to kiln-drying at temperatures of up to 70°C. One of the main benefits of green gluing is that the fresh, undried, timber is not distorted in shape as a result of drying and consequently, is straight and flat when glued. This in turn means that the, for example, laminated products can be manufactured with less waste. Moreover, most wood pits remain open through the green gluing process, in contrast to wood that has been dried, facilitating the penetration and anchoring of the applied adhesive. MCPU adhesives are very attractive for this application because of the high water content in green wood. Few studies have been published investigating the properties of green glued products and the characteristics of the formed adhesive bonds.^{15,17–21}

One difficulty along the path to accepted, wide spread use of green gluing is that there is a lack of standards to certify adhesives for this application. Structural adhesives for wood are certified according to the traditional dry gluing processes. To develop a new type of certification, more understanding is required regarding the adhesive chemistry during the green gluing process, that is, a curing processes taking place in the presence of different quantities of water, and the effect of heat treatment after curing on the chemical, physical, and shear strength properties. Previous study has shown that green glued bonds can be even stronger in comparison to dried glue bonds when MCPU adhesive is used¹⁹ and heated,²² whereas a more in-depth understanding of the reasons for this is needed.

The aim of the present study is to investigate the effect of a range of curing conditions (moisture and temperature) on the cure rate and resulting viscoelastic, chemical, and adhesive properties of a standard commercial MDI-based MCPU to better understand the mechanisms of green gluing. This is carried out by curing a commercial MCPU in four different relative humidities and subsequently postcuring them at elevated temperatures, similar to the process of green gluing performed in industry today, to determine the effect on the resulting chemistry, and mechanical properties. Finally, the adhesion was tested by measuring the green strength with a lap-shear test to evaluate the adhesive bond strength in wood joints and correlate this to the chemical, physical, and thermal properties as observed by rheometry,

Fourier transform infrared spectroscopy (FTIR), and dynamic mechanical thermal analysis (DMTA).

EXPERIMENTAL

Materials

The adhesive has the trade name Prefere 6000 and was supplied by Dynea AS. This one component polyurethane adhesive for wood has a NCO content of 14.5%.²³ This adhesive is certified for structural use (i.e., in load bearing wood structures) and is a traditional moisture curing isocyanate functional prepolymer based on a polyol and MDI. Beech veneer samples of dimension 2 × 20 × 117 mm were obtained from a local source. Beech wood has a relatively high density and stiffness, and therefore was chosen for this study to minimize premature wood failure as much as possible.

Instrumentation

Rheometry

A Nova Rheometer from Reologica Instruments AB was used with a plate diameter of 20 mm, a frequency of 1 Hz, a strain of 0.005, and a gap of 0.5 mm. The sample 0, 0.9, 2.6, 5.6, 6.1, and 10 wt % water content was mixed with a spatula in a vial and directly placed on the plate and measured at 25°C every 120 s for 24 h, resulting in the storage modulus (G') and loss modulus (G''). Measurement on the MCPU containing 4.1 wt % water was also carried out at 70°C for 4 h after the adhesive was cured at 20°C to determine the effect of these conditions on the storage modulus over time. The sample was heated after cure from 25 to 70°C, taking 314 s until a stable temperature of 70°C was reached. A measurement was taken every 30 s for 4 h at 70°C. The flow behavior of the adhesive during cure was measured at room temperature. Duplicates were taken of select samples to ensure reproducibility with error within $\pm 5\%$.

Fourier transform infrared spectroscopy

Infrared spectra in the Midregion (FTIR) were acquired using a PerkinElmer FT-IR-2000 in reflectance mode equipped with a TGS detector. An average of 16 scans were taken at 2 cm^{-1} resolution in the range of 4000–600 cm^{-1} . Samples were analyzed in duplicate using Spectrum version 3.02.01 and graphing software Origin 7.5. Measurements were taken of uncured and cured adhesives, with two different samples taken to ensure reproducibility.

Conversion was calculated by normalizing all spectra to the aromatic and aliphatic C=H stretch that occurs in the region 2950–2800 cm^{-1} that remains unchanged during moisture cure of the adhesive. For the determination of the free isocyanate content, the

band at 2270 cm^{-1} was analyzed. The area of the band was calculated and the ratio of area before and after curing with different treatments was calculated. Moreover, for cured films the region between 1620 and 1760 cm^{-1} was chosen to show different peaks representing carbonyl group in different environments.¹² Carbonyl groups associated with free urethane groups are observed at the band range of 1740 – 1730 cm^{-1} , those of the urethane (N–H) group hydrogen-bonded (H-bonded) to the ether (C–O–C) group in polyol (HS/SS interaction) are observed at 1730 – 1725 cm^{-1} , carbonyls of the urethane (N–H) group that are H-bonded to a urethane carbonyl group (C=O) (HS/HS interaction) are observed at 1715 – 1700 cm^{-1} , those carbonyls that are in free urea, or less ordered H-bonded urea is observed at 1690 – 1650 cm^{-1} (monodentate), and those carbonyl groups that make up high-ordered H-bonded urea can be seen in the range of 1650 – 1640 cm^{-1} (bidentate).¹² The postcured height of these peaks was compared after normalization to determine the amount of these groups within the films after the described heat and moisture treatments.

Dynamic mechanical thermal analysis

A Rheometric Scientific Mark IV DMTA was used with a tensile mode clamp to measure the viscoelastic properties of the MCPU films. The specimen shape was a rectangular strip with a length of 6 mm, a width 3 mm, and a thickness of 2 mm. The temperature scans were performed with a heating rate of $2^\circ\text{C}/\text{min}$ and the loading frequency was 1 Hz. The scans were carried out from -60 to 200°C . The measured modulus of the cured and post-treated samples does contain a small error owing to a microporous structure (cavities) that were present. However, the measurements were repeatable and the phase transitions can be quantitatively compared. Samples were measured in duplicate to ensure reproducibility.

Shear lap tensile testing

The tensile shear tests were performed using a Lortzen & Wettre Tensile Tester (Model ALWETRON 50) using a load rate of $5\text{ mm}/\text{min}$. The fractured surfaces were examined in a light microscope to investigate whether the fracture path took place in the adhesive, the interface (i.e., between adhesive and wood) or the interphase (i.e., wood penetrated with adhesive) of the bond.

Procedures and methods

Preparation of adhesive films

Films of the polyurethane adhesive were cured for 3 days at 20°C in four different relative humidities 30,

TABLE I
Naming Scheme of the MCPU films

Naming scheme	RH (%)	Curing temperature, 72 h ($^\circ\text{C}$)	Postcuring temperature treatment, 72 h ($^\circ\text{C}$)
RH 30-T20	30	20	20
RH 30-T70	30	20	70
RH 30-T103	30	20	103
RH 65-T20	65	20	20
RH 65-T70	65	20	70
RH 65-T103	65	20	103
RH 98-T20	98	20	20
RH98-T70	98	20	70
RH98-T103	98	20	103
W-T20	Water	20	20
W-T70	Water	20	70
W-T103	Water	20	103
Uncured	–	–	–

65, and 98% RH, and in liquid water. The samples were then either left at room temperature or post-heat treated for 72 h at 70 or 103°C (to mimic the industrial kiln drying process of green-glued wood products). Finally, the effect of this heat treatment on the properties of the cured polyurethane adhesives was determined. The different curing environments for the adhesive films and the naming scheme used in this study are summarized in Table I.

Preparation of wood adhesive bonds

In gluing of the wood adhesive bonds, beech veneer samples, with tangential surfaces, of dimension $2 \times 20 \times 117\text{ mm}$ were used. Wood samples were conditioned at 65% RH and 20°C before gluing, resulting in 12% moisture content (MC) in the wood. Three different gluing and drying conditions were tested: (1) wood glued at 12% MC and cured at 20°C , resembling a conventionally dry glued bond, (2) gluing of wood that had been immersed in water during 24 h and then dried to the original weight at 20°C , resembling gluing of undried wood, and (3) gluing of wood that had been immersed in water during 24 h and then dried to the original weight during 15 min at 70°C , resembling the bond obtained in the industrial green gluing process. However, this latter heat treatment is typically shorter than the typical green gluing process which is important to consider when analyzing the results. A press time of 2 h was applied, with an adhesive spread rate of $200\text{ g}/\text{m}^2$ and a pressure of 0.8 MPa. The specimens were glued using an Automated Bonding Evaluation System,²⁴ which is typically used for testing the development of wood adhesive bond formation.²⁵ The tensile shear specimens prepared had an overlap of 5 mm and thus, the glued bond area was 100 mm^2 . A short adhesive bond length was chosen to obtain a uniform stress

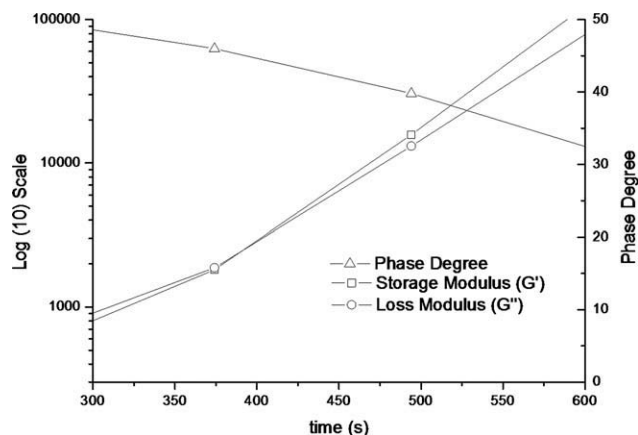


Figure 1 Storage modulus, loss modulus, and phase degree of the MCPU containing 5.6 wt % water at room temperature.

distribution. All types of specimens were produced in five replicates and were conditioned at 65% RH and 20°C for 2 weeks prior to testing to ensure a homogeneous MC within the specimens.

RESULTS AND DISCUSSION

The effect of water concentration on the adhesive cure rate (rheometry)

The state of moisture in wood gluing with freshly sawn undried wood surfaces is complex and the previous studies have attempted to describe the wood surface drying phenomena.²⁶ From these studies, it is apparent that the wood surface begins drying immediately after cutting and that diffusion of water occurs (up to a few millimeter) under the surface. This means that the MC on the surface that is available to the adhesive is dynamic and exists within a large range, from 0 to more than 100% MC. The levels of no added water (0 wt %) and intervals of added water up to 10 wt % to the MCPU, simulates this wide range of available moistures from dry (0 wt %) to MC above the wood fiber saturation point (10 wt %).

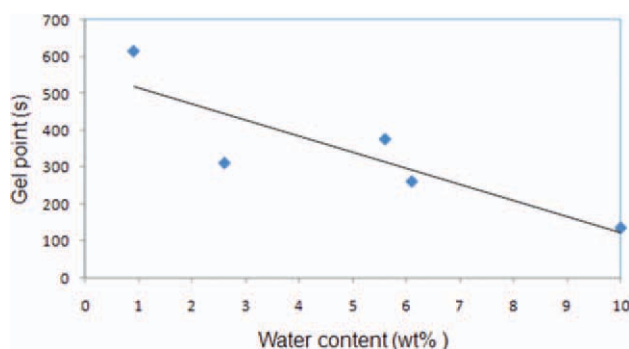


Figure 2 Gel point relative to the added water content. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

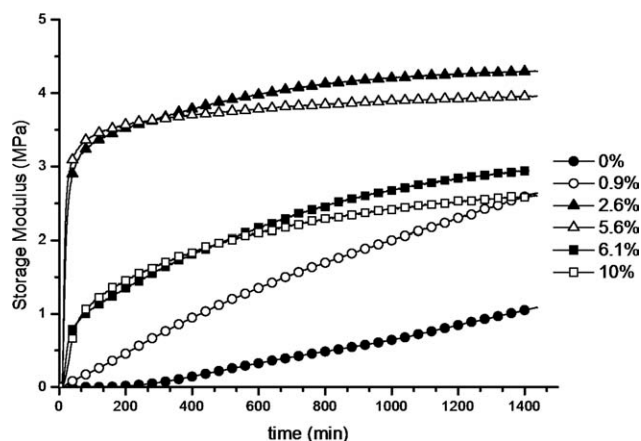


Figure 3 The storage modulus (G') of the MCPU containing various levels of water over time.

To determine the effect of the amount of water on the cure rate of the MCPU, rheometry was performed over 24 h on the uncured liquid MCPU adhesive containing 0–10 wt % water. The gel point, storage modulus, loss modulus, and phase degree were obtained. The gel point of cure is traditionally defined as the point at which the phase degree reaches 45° and lower, where the storage and loss moduli values cross one another, and the polymer network structure becomes irreversible.²⁷ This is shown in Figure 1 in which the adhesive containing 5.6 wt % water reaches a gel point after 375 s.

It was observed that cure rates of the MCPU with various water content between 0.9 and 10 wt % cause the gel point to vary between 10 and 2 min, respectively, with the higher degree of water resulting in significantly faster cure rates. (Fig. 2)

For a more complete view of the curing process, the comparative storage moduli, loss moduli, and phase degree of the adhesives, with 0–10 wt % H₂O, during cure are shown in Figure 3. Figure 3 shows that with the addition of 2.6 and 5.6 wt % water to the MCPU storage moduli increase fastest and to the highest levels within the first 30 min. This implies that the elastic response is highest in these polymers that appear to have the most interaction with water at these levels. Above this level of water, there is a decreased level of elastic response from the polymer, indicating that more than 5.6 wt % addition of water plasticizes the polymer in spite of the gel point being reached. Below 2.6 wt %, there is so little water available that the reaction progresses slower, resulting in a lower initial elastic response.

The adhesive cured without addition of water showed a significantly delayed curing (almost 2 h compared to the test samples with water present), owing to a skin forming and inhibiting further curing by water vapor.

Effect of postheating on the viscoelastic properties (rheometry)

To determine the effect of postheating the MCPU at 70°C, the typical industrial drying temperature for timber, the storage modulus of the adhesive containing 4.1 wt % added water was measured during these conditions, directly after curing. The storage modulus increases from 1 to 1.3 MPa over 4 h, during heat treatment meaning an increase of 20% during heating. This can be attributed to the isocyanate reacting further, chains orienting toward equilibrium conformation and this indicates that a stiffer polymer is produced with heat. The chemical reason for this is further studied by FTIR and DMA. For practical applications such as green gluing, this implies that when timber with high MC (above the fiber saturation point) is glued, the pressing time and drying is important for removal of excess water and increased conversion for stronger adhesion.

Effect of cure conditions on the chemical composition of the cured MCPU (FTIR)

Degree of isocyanate conversion in the cured films

The amount of residual isocyanate in the films can be determined by taking the ratio of the band at 2275 cm⁻¹ before curing (in the liquid adhesive) and after curing (in the cured films) after normalizing. As shown in Figure 4(a), the adhesives cured with lower amounts of water, such as RH30-T20, have higher amounts of residual isocyanate compared to MCPU cured under the conditions of high moisture or high temperature, which have less residual isocyanate. This is most likely a result of the heat providing added mobility and the water allowing for reaction of the residual isocyanate into urea and ordered bidentate groups.

Additionally, the largest decrease in residual isocyanate content in the film resulted from RH30 to RH65 at a temperature of 20°C. In summary, the residual isocyanate content decreases when more moisture was present during cure and when post-cure heating was applied, which should result in increasing the adhesive strength.

Urea and urethane formation/interactions

The temperature and MC have not only shown to have an effect on the amount of residual isocyanate, but also on the resulting degree of organization of urea and urethane groups in the final films. To investigate the formation of different types of H-bonds and the formation and interaction of SS and HS, MCPU, cured under different conditions, was investigated by FTIR in the region of 1600–1720 cm⁻¹.^{12,28} (Fig. 5) Two peaks were analyzed, charac-

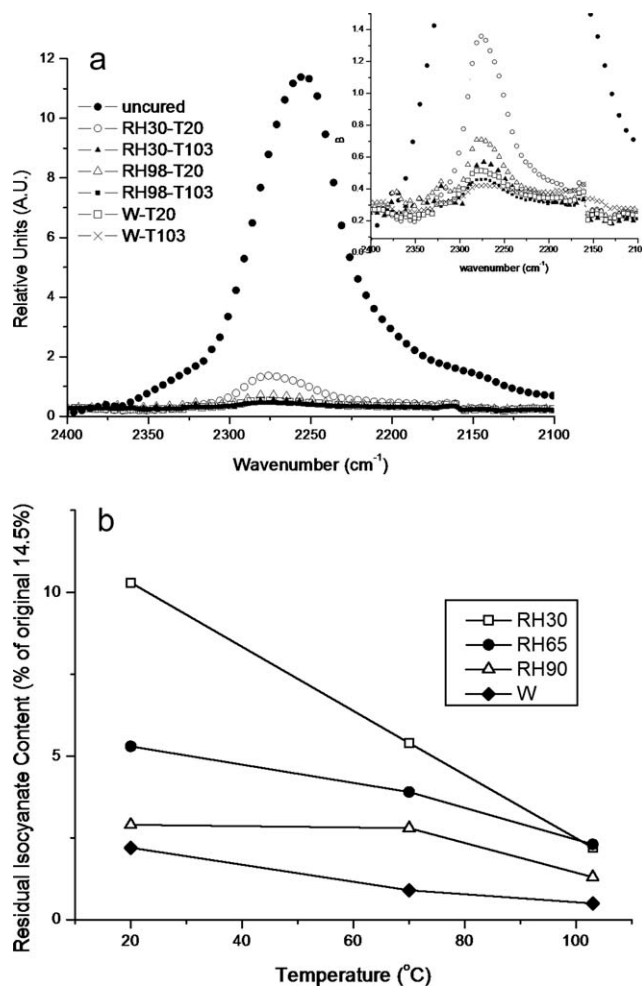


Figure 4 (a) Free isocyanate in uncured and cured adhesive films as a result of different humidity levels at curing and postheat treatment and (b) residual isocyanate as the area of the band at 2275 cm⁻¹ after normalization.

terizing different types of interactions between polymers: (1) the urea peak around 1643 cm⁻¹, indicating the relative amount of hydrogen bonding occurring between the amine group of urea and the carbonyl group of urea, resulting in highly ordered urea (bidentate) (HS), (2) the urethane band around 1710 cm⁻¹ indicating the relative amount of hydrogen bonding between the carbonyl group of the urethane and the amine group of the urethane (HS–HS interaction). Moreover, the peak at 1730 cm⁻¹ indicates the unbound urethane groups, which are not organized in H-bonding, and are present in the prepolymerized adhesive. The additional bidentate content observed to form with postcure heat treatment should also increase the strength of the bond line. The amount of biuret and allaphanate which may also form to a small extent and contribute to the peaks between 1690 and 1730 cm⁻¹ was not studied.

With a lower temperature of 20°C, more urethane interactions and less urea interactions are observed, urea exists in a less ordered form (illustrated by the

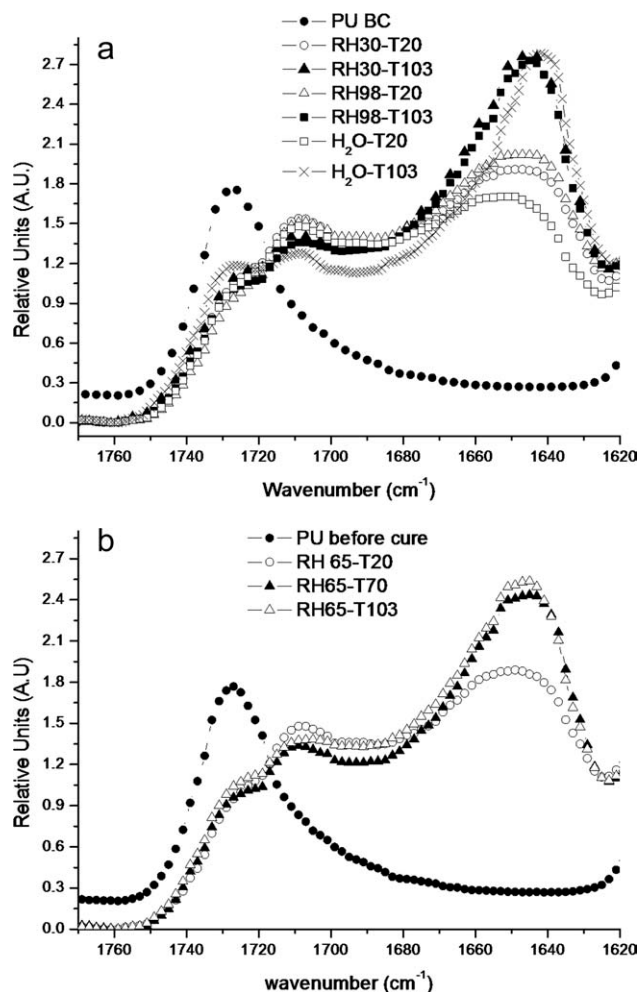


Figure 5 Plot illustrating the relative heights of the peaks of urea and urethane of the PU-films. (a) Effect of moisture and temperature (b) effect of temperature for film cured at 65% RH.

wide peak in the interval $1640\text{--}1670\text{ cm}^{-1}$), with no significant trend from the influence of humidity. All films postheated at the higher temperature of 103°C , W-T103, RH98-T103, and RH30-T103 exhibit a peak associated with the carbonyl in the environment of a high degree of ordered urea groups (1638 cm^{-1}) and a relatively lower degree of urethane groups (1710 cm^{-1}). Again, this is attributed to the high mobility and the large number of urea groups able to organize if the MC and the temperature are both high.

No significant influence of humidity on the amount of H-bonded urea and urethane groups in the cured adhesives could be observed. [Fig. 5(a)]

For simplicity, the samples that were heat treated at 70°C are not compared as no significant difference in the bidentate content, in 70 and 103°C heat-treated MCPU were observed, indicating that 70°C is enough heat to alter the composition of the adhesive [Fig. 5(b)].

Effect of cure conditions on the viscoelastic properties of the cured films (DMTA)

DMTA was performed to determine if the differences in residual isocyanate levels and amount of urea content in the films have an influence on the viscoelastic and thermal properties of the cured adhesive. There appears to be no significant difference in the levels of the storage modulus for the films cured in different environments [Fig. 6(a)]. The small differences observed can be attributed to the microscopic air bubbles that occurred in the film owing to the formation of carbon dioxide.

However, the $\tan\delta$ plot does reveal a trend regarding the T_g values of the films (Fig. 6). With the addition of heat, the T_g is significantly increased regardless of the water content when cured. The heat-treated MCPU has a glass transition temperature of approximately 35°C higher compared to the nonheat-treated film. This is the case for all three humidities, with no significant effect of humidity on the $\tan\delta$ and storage moduli and therefore only the W-T20 and W-T103 are shown for comparison. This increase in T_g with heat is a very significant difference that could be used to tune the properties of the polymer for different applications; especially in structural building applications where a T_g in the range of $75\text{--}85^\circ\text{C}$ would be of great interest. It is interesting to note that the FTIR heat-treated films showing increased amounts of bidentate groups that are highly ordered and contain more hydrogen bonding. This results in a polymer with a higher T_g , as indicated by DMTA, that should provide for a stronger adhesive.

Adhesion performance of the MCPU to wood veneers (shear lap test)

In an attempt to correlate the abovedescribed chemical and viscoelastic properties to adhesive joint strength

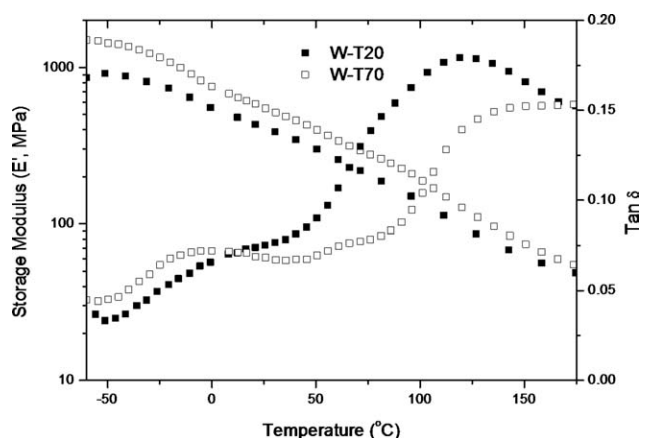


Figure 6 Results from DMTA testing of adhesive films cured at different humidities and postheat treated, tested in tension mode: storage modulus (E') and $\tan\delta$.

TABLE II
Shear Strength of Adhesive Bonds

Sample name	Shear strength (MPa) (\pm)	Location of failure	Percentage of wood failure (%) Min-max
Dry glued	12.4 (2.3)	Wood-adhesive, wood and adhesive	0-80 (mean wood failure, >25%)
Wet glued, dried at 20°C	8.6 (2.1)	Adhesive : adhesive	0
Wet glued, dried at 70°C	11.5 (2.0)	Adhesive : adhesive	0

in wood applications, shear strength tests were performed (Table II). It is of most interest to determine the temperature and moisture influence on the bond shear strength. The shear strength testing was carried out on samples that would most clearly show the effect of water content in the wood on adhesion, and therefore, samples chosen included the wet veneer in comparison to the dry veneer. To most clearly demonstrate the influence of temperature, the heated and unheated specimens were tested.

The samples that were glued in the dry state failed partially within the zone of the wood impregnated with adhesive, that is, the wood-adhesive interphase (Fig. 7). Further, it appeared that more adhesive was taken up by the wood in these samples, most likely owing to the low MC which allowed for a later gel point as observed by rheometry. The strength of this bond is slightly higher than the wet wood samples, indicating that the residual isocyanate content observed by FTIR with less MC and no heat is not a significant factor in the shear strength bond line in this case.

The shear test samples that were wet glued, both those dried in ambient (7b) and higher temperatures (7c), failed within the adhesive layer, that is, cohesive adhesive failure, with no wood failure observed. This could be an effect of lower penetration because the gel point occurred faster owing to the high MC as was observed by rheometry studies

with MCPU containing 10 wt % MC. The wet glued bond dried in ambient temperature (7b) had significantly lower strength. The chemical composition of the adhesive in the wet and dry glued bonds cured at 20°C is comparable with similar urea content (as observed by FTIR) and T_g (as observed by DMTA). Thus, their different behavior observed by shear bond strength is not an effect of the chemical composition, but the physical phenomenon of water plasticizing the polymer, as it was observed by rheometry. However, the wet glued bond subjected to heat treatment (7c) had similar strength to the dry glued bond (7a), but higher strength to the wet glued bond without heat treatment (7b). The heat treatment provided a significant increase in the strength of the wet glued adhesive. This is explained by the increased ordered bidentate content (FTIR), the increased T_g (DMTA), and the higher modulus of the MCPU (rheometry) when the heat is applied.

It should be kept in mind that the beech veneer was received in the dried state, whereas in practice green gluing is usually carried out on freshly sawn spruce wood with open pores. Additional investigations on the adhesion of MCPUs to undried wood and the influence of heat treatment would be interesting to investigate in further studies as it has been shown in recent study that MCPU green glued bonds can be stronger than dry glued bonds.^{13,15}



Figure 7 Fractured surfaces (from left) dry glued (a), green glued dried at 20°C (b) and green glued dried at 70°C (c). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

CONCLUSIONS

An investigation has been done toward correlating quantified water content and postheating to the resulting chemistry, mechanical properties, and bond shear strength of a moisture cured polyurethane adhesive. The cure rate of the adhesive is dependent on the MC present during cure, with 2.6 and 5.6% moisture, resulting in the highest initial elastic modulus.

Postheating of the cured MCPU adhesive had a larger effect on the adhesive properties than the amount of moisture in the environment during cure. Postcure heating resulted in the formation of highly ordered bidentate groups, which are not present in the adhesive cured at ambient temperature. Moreover, the adhesive with postheating had a significantly higher T_g , as observed by DMTA, and higher storage modulus, as observed by rheometry. This was confirmed by increase in the shear bond strength when the wet veneers were adhered and heat treated.

With the present study, a step is taken toward understanding the formation of wood adhesive bonds based on MCPUs in the green gluing process.

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