Addition of Polyurethane Foam Waste to Polymeric Diphenyl Methane Diisocyanate to Improve Plywood Binder Performance

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Abstract

The objective of this study was to investigate the feasibility of using polyurethane (PU) foam waste obtained from automobile shredder residue (ASR) in plywood production. The PU foam waste from ASR was cleaned by water and acetone, ground into powder, dried, and mixed with polymeric diphenyl methane diisocyanate (pMDI) resin at various ratios (2.5%, 5%, 7.5%, and 10%, based on the weight of pMDI). The mixed adhesives were examined with Fourier-transform infrared spectroscopy (FTIR) and thermogravimetric (TG) analysis, and evaluated as plywood binders (measured in accordance with Chinese National Standard GB/T 17657-2013). The results indicated that the addition of PU powder into pMDI resin increased adhesive viscosity and efficiency, and also helped solve the problem of deficiency of pMDI on the veneer surface. FTIR results showed various reactions between pMDI and PU powder. This potential cross-linking might contribute to the cohesive strength of the cured adhesive. TG analysis results showed the possible increased thermal stability of the cured mixed adhesive at a temperature range of 150°C to 300°C. The best PU addition ratio was 7.5 percent of the pMDI weight, considering both the bond strength and production practice of plywood. Plywood testing showed that both the dry and wet bond strength reached the peak value at the optimal mixing ratio. The use of PU powder in plywood manufacturing provided a possible way of recycling PU foam waste while improving or maintaining the performance of plywood.

As a polymer material, polyurethane (PU) is used widely in various applications but exhibits considerable volumes of waste. Currently, about 6 million cars and trucks reach the end of their service life annually in China. This will generate automobile shredder residue (ASR) of 9 million tons each year, with 12 to 16 percent composed of PU foam materials (Ni and Chen 2014). In 2016, a Chinese professional standard (SB/T 11160-2016), ''Technical Specification for Crushing of Waste Vehicles'' (Standardization Administration of the People's Republic of China [SAC] 2016), was released. According to this standard, after crushing and sorting, the comprehensive recovery rate of renewable resources should not be less than 90 percent. The increased use of PU foam in automobiles makes recycling a challenge. Currently, only a small fraction of PU foams is recycled; most of them, instead, end up in the landfill, generating a negative environmental impact (Haydary and Dalibor 2015).

The amounts of wood-based panel production and consumption in the year of 2018 in China were 299 and 290 million cubic meters, respectively. Urea-formaldehyde (UF) resin is mainly used in wood-based panel production; however, formaldehyde release and poor water resistance of UF-bonded wood composite panels is a key concern. Replacing UF resin with other formaldehyde-free or low-formaldehyde adhesives has become an important research

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area (Wan et al. 2017, Mao et al. 2018). Polymeric diphenyl methane diisocyanate (pMDI) resin can be used as particleboard and fiberboard binders. Compared with formaldehyde-based thermosetting synthetic resins, pMDI has the advantages of a strong bond, faster curing, good water resistance of the adhesive layer, and no formaldehyde release from the adhesive. However, the small molecules in pMDI might penetrate into the wood, resulting in a starved glue line at the veneer surface (Zhu and Yu 2011, Li et al. 2017). Therefore, pMDI is not directly used in plywood and laminated veneer lumber (LVL) production.

A review of the literature on the application of PU foam–based material in wood bonding produced very limited results (Kwon et al. 2007, Bene et al. 2012). In one study, flexible PU foam powder was added to UF and phenol-formaldehyde (PF) resins to make plywood and particleboard (Mansouri and Pizzi 2007). It was found that the addition of PU powder to UF could serve as an active filler and extender to improve the dry bond strength of the UF resin to wood. The water resistance of these PU foam powder–modified UF and PF resins was also improved. Mao et al. (2014) mixed pMDI resin with recycled rigid PU foam powder at different ratios for particleboard fabrication. The internal bond strength of particleboard was improved when replacing 5 to 15 percent of pMDI with PU powder.

The objective of this study was to investigate the feasibility of using PU foam wastes in plywood production. The goal of this research was to provide a theoretical basis and scientific guidance for the rational recycling and application of waste PU materials into pMDI-bonded plywood.

Materials and Methods

Preparation of PU powder

Flexible PU waste foams produced by ASR were obtained from a local vehicle waste stream recycling company in Taian, Shandong Province, China. The color of the foams was light brown. Some rubber, wood, fibers, paper, glass, and other contaminants in the PU foams were removed and the foam was washed by water and then acetone (obtained from Sinopharm Group Co. Ltd, Beijing). The foams were then air-dried under a hood and ground into a 120-mesh powder using a grinding machine (Laboratory Mill, Model WQM; Jingcheng Chemical Machinery Co. Ltd, Jiangyin).

Adhesive preparation

The pMDI (Lupranate M20FB) with the isocyanate (NCO) content of 31.5 percent was obtained from the BASF Chemical Company (Wyandotte, Michigan). It was a dark yellow liquid with a viscosity of 278 cP at 25°C. The mixed adhesives were prepared by physically mixing pMDI with PU powder of 0 percent moisture content at various ratios (2.5%, 5%, 7.5%, and 10%, based on the weight of pMDI resin). The adhesive mixtures were labeled as pMDI-2.5, pMDI-5, pMDI-7.5, and pMDI-10, respectively.

Adhesive property evaluation

Viscosity.—The viscosities of the mixed adhesives were measured at 25°C in accordance with Chinese National Standard GB/T 14074-2017 (SAC 2017) using a rotational Brookfield DVS+ viscometer (Brookfield Engineering Laboratories. Inc., Massachusetts), with three replicates.

Fourier-transform infrared spectroscopy.—Fourier-transform infrared (FTIR) analysis was conducted using a Bruker Tensor II spectrophotometer (Bruker Optik GmbH, Ettlingen, Germany). The liquid samples of the adhesives were added as drops on the observation stage of the spectrophotometer. The mixed adhesives were cured in an oven at 120°C for 3 hours. The solid samples of the PU foam powder and all of the cured mixed adhesives were placed on the observation stage of the spectrophotometer. The FTIR spectra were recorded in the wavelength range of 4,000 to 600 cm^{-1}. A total of 64 scans was averaged for each sample.

Thermogravimetric analysis.—Thermogravimetric (TG) analysis was carried out with a TA Q50 analyzer (TA Instruments, Delaware). All adhesive samples were cured at 100°C for 3 hours in a convective drying oven. Specimens were then placed in alumina crucibles, with an empty alumina crucible used as a reference. Specimens were heated from 25°C to 600°C at the heating rate of 10°C/min. The flow rate of nitrogen purge gas was 20 mL/min.

Panel manufacturing

Three pieces of birch (*Betula* sp.) veneers with a density of about 638 kg/m^3, thickness of 1.6 to 1.7 mm, and a moisture content of 8 to 10 percent for the plywood top, middle, and bottom layers were cut in an end-match manner for plywood manufacturing. This allowed us to minimize the influence of wood quality variation on final panel performance. Plywood was fabricated at 300 by 300 mm using a laboratory hot press (Suzhou Xinxieli Machinery Manufacturer Co. Ltd., Suzhou, China). An adhesive application rate was 220 g/m^2 (single glue line). The adhesives were applied uniformly using a brush on both sides of the middle veneer. The hot press time, temperature, and pressure were 5 minutes, 135°C, and 1.1 ± 0.1 MPa, respectively. Two panels were made for each adhesive formulation. After pressing, the plywood panels were conditioned at 20°C and 60 percent relative humidity for 1 week.

Measurement of the bonding strength

The bond strength was measured in accordance with the Chinese National Standard GB/T 17657-2013 (SAC 2013). Six specimens with dimensions of 100 by 25 mm were cut from each panel to make a total of 12 specimens for each adhesive formulation. From these samples, six specimens were randomly selected and tested for dry and wet strength. Before the testing of the wet strength, specimens were first immersed in boiling water for 4 hours, then dried in an oven at 63°C for 18 hours and immersed in boiling water again for 4 hours. After the second water-immersion treatment step, the specimens were cooled down in water at 25°C and tested for lap shear strength with a CMT 4104 universal testing machine (MTS System Corporation, Minnesota). The wood failure percentage was identified and recorded.

Statistical analysis

Means and standard deviations of the data obtained were calculated. Duncan's multiple range tests with SPSS 13.0 software (Chicago, Illinois) were used to determine the differences among different treatments.

Results and Discussion

Characterization of the PU foams

After the PU foams were washed with water and acetone and dried, the color of these foams turned light yellow, and a weight loss of 10 to 20 percent was obtained. The weight loss could be from water-soluble, acetone-soluble substances, and volatile compounds of PU foams collected.

The chemical composition of the ground PU foam powder was characterized using FTIR. The FTIR spectrum (Fig. 1) shows the absorption bands of the PU foam powder. The absorption bands at 1,714.5 and 1,531.8 cm^{-1} correspond to the urethane carbonyl group (C=O in –NH–CO–O–) stretching and N–H bending vibration, respectively. The absorption bands at 1,643.2 and 3,289.4 cm^{-1} correspond to the urea carbonyl group (C=O in –NH–CO–NH–) and the stretching vibration of N–H groups, respectively. These two groups were the products from the reaction between isocyanate and water (Wu et al. 2003, Wang and Chen 2006, Kwon et al. 2007, Bene et al. 2012). The absorption band at 1,449 cm^{-1} corresponds to the aromatic ring and is indicated as a possible aromatic source of the isocyanate as one of the raw materials in the preparation of the PU foams (Lee et al. 2005). The intense stretching vibration of the aliphatic –C–O–C– group at about 1,097.8 cm^{-1} suggests that the main type of raw polyols used in the preparation of the PU foams might be polyether polyols (Mao et al. 2014).

Adhesive viscosity

The viscosity of the adhesives is presented in Table 1. The statistical analysis demonstrates that the viscosity of the pMDI–PU mixing increased with PU powder content (increased from 278 cP for pMDI to 3,280 cP for PMDI-10). In other words, the viscosity of the adhesives was closely related to the amount of PU powder. It was estimated that as the amount of PU powder in the adhesives increased, the foaming structure of the PU powder could absorb more pMDI resin, especially the smaller molecules in the pMDI resin, and this affected the flow during mixing. So, the viscosity of the adhesive would increase accordingly, which helped to maintain pMDI on the veneer surface and prevent the resin from penetrating into the wood cell walls. This could help solve the problem of a starved bond-
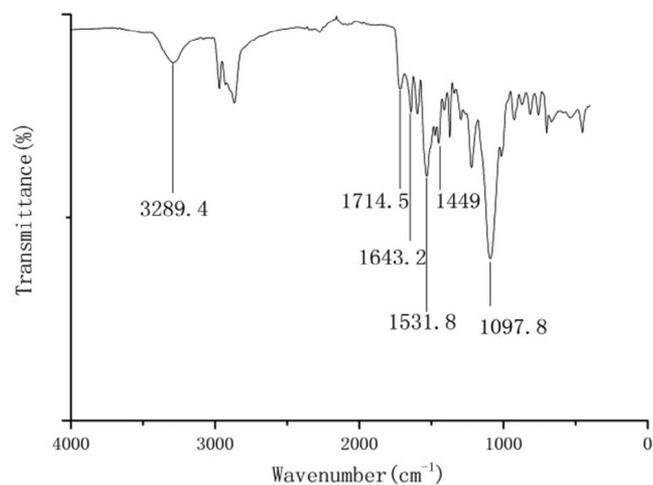

Figure 1.—*Fourier-transform infrared spectrum of polyurethane powders.*

line on the veneer surface when using pMDI as a plywood binder. However, as the amount of PU powder added was increased, the viscosity eventually became too high and made dispersion difficult in a plant setting. According to viscosity results (Table 1) and lab practice, a PU powder content higher than 10 percent might result in viscosities that are too high for application onto plywood or LVL.

FTIR spectrum of adhesives

Figure 2 shows the FTIR spectrum of the adhesive liquid mixtures at various percentages of PU powder substitution (2.5%, 5%, 7.5%, and 10%). The absorption band at 2,242 cm^{-1} corresponds to the isocyanate group (–NCO). The spectrum of pMDI resin was similar to those of pMDI-2.5, pMDI-5, pMDI-7.5, and pMDI-10 adhesives, indicating that the reaction might not happen immediately after the PU powder was mixed with the pMDI. During plywood and LVL manufacturing, the addition of PU powder into pMDI resin might not affect the pot life of pMDI resin greatly and this will provide enough time for adhesive application.

Figure 3 shows the FTIR spectrum of the cured adhesives obtained by mixing PU powder into pMDI at various weight percentages (2.5%, 5%, 7.5%, and 10%). The subtle change in absorption peaks with the addition of PU powder suggests that the pMDI structure was not altered substantially. This was probably because the PU powder addition amount was still very small (no more than 10%, based on the weight of pMDI resin), or that the FTIR used was not sensitive enough to tell the difference. The absorption band at 1,662 cm^{-1} should correspond to the carbonyl group (–C=O) in substituted urea, which was the reaction product of –NCO groups and water (Mao et al. 2014). The cured mixed adhesives still showed obvious absorption peaks of the –NCO group (absorption bands at 2,252 cm^{-1}) in the spectrum. This result indicates that even though the pMDI and mixed adhesives were cured at 120°C for 3 hours, some residual –NCO groups were still left in various proportions (Li et al. 2017). As the amount of PU powder in the adhesives was increased, the strength of absorption bands of –NCO groups decreased, indicating that some reactions might happen between –NCO groups and other functional groups or materials in PU powder. The potential reactions could be that (1) –NCO groups in pMDI resin reacted with trace water in PU powder or (2) –NCO groups in pMDI resin reacted with –NH– groups in PU powder (Fig. 1). Either reaction might result in the increase of the cross-link density of cured adhesives, which would contribute to the cohesion strength of cured mixed adhesives; or it may be simply that too much PU powder added in might reduce the density of –NCO groups in the mixtures.

TG analysis of the adhesives

The TG curve of the PU powder sample is shown in Figure 4 and the weight loss of PU is shown in Table 2. The PU powder had two different weight-loss regions. The first weight-loss region occurred between 200°C and 300°C and exhibited a weight loss of 28 percent. The thermal degradation of PU materials started at a temperature of 200°C. At this temperature, the C–O bond in the urethane group started to break and generate isocyanate and polyols (Yuan et al. 2005). This process lasted until the temperature reached 300°C. At 300°C, the degradation of the urethane group was nearly completed and the curve became stable at

Table 1.—Viscosities of the adhesives.

	Adhesive sample[a]				
	PMDI	PMDI-2.5	PMDI-5	PMDI-7.5	PMDI-10
Mean (SD) viscosity (cP)	278 (5.2) A[b]	539 (15.9) B	1,069 (18.7) C	2,231 (25.2) D	3,280 (39.8) E

[a] pMDI = polymeric diphenyl methane diisocyanate; pMDI-2.5 = pMDI with 2.25 percent polyurethane; pMDI-5 = pMDI with 5 percent polyurethane; pMDI-7.5 = pMDI with 7.5 percent polyurethane; pMDI-10 = pMDI with 10 percent polyurethane.

[b] Data in the same row with the same letter are not different at $\alpha = 0.05$.

higher temperatures. The second weight-loss region was prevalent at 350°C to 400°C, which had a corresponding weight loss of 55 percent. As the temperature increased to 350°C, the curve started to decrease quickly. The reason could be the degradation of the products from the first degradation reaction: isocyanate and polyol. At temperatures higher than 350°C, the isocyanate and polyol started to degrade to generate amines, olefins, and CO_2 (Duquesne et al. 2001). This could result in an obvious weight loss of PU materials.

The TG curves of the adhesives are shown in Figure 4 and the calculation of weight loss in the adhesive is shown in Table 2. From room temperature to 600°C, there were two different weight-loss regions. The first weight-loss region occurred between 150°C and 300°C, and had a weight loss of about 59, 55, 53, 52, and 51 percent for pMDI, pMDI-2.5, pMDI-5, pMDI-7.5, and pMDI-10 adhesives, respectively. The weight loss decreased with increased PU powder substitution. The reactions of pMDI with water and –NH– groups in PU powder appeared to increase the thermal stability of the cured mixed adhesives (pMDI-2.5, pMDI-5, pMDI-7.5, and pMDI-10) between 150°C and 300°C, resulting in less weight loss than that of pMDI resin alone. The second weight-loss range started at around 400°C and lasted to the end of the TG tests (600°C). During exposure to this temperature range, the weight losses were 19, 24, 26, 26, and 27 percent for pMDI, pMDI-2.5, pMDI-5, pMDI-7.5, and pMDI-10 adhesives, respectively. After the PU powder was added into the pMDI resin, the weight loss increased (from 19% to 27%). This result was opposite of

that in the first weight-loss range (from 150°C to 300°C) discussed just above. These weight losses might be from thermal degradation of the PU powder. However, from the TG test results of PU powder, after 400°C, the degradation line already became stable and the PU powder had no obvious weight loss. The probable reason might be that the addition of PU powder into pMDI resin also increased the thermal stability of the PU powder so that the second weight loss of PU powder was postponed to a higher temperature range. The final percentage of degradation remains for all of the adhesives was similar but clearly higher than that of PU powder.

Panel bonding performance

The bond strength and wood failure percentage of the adhesives were determined (Table 3). Both the dry and wet strengths of the mixed adhesives met the requirements of the standard GB/T 17657-2013 (SAC 2013) for outdoor applications (≥ 1.0 MPa for dry strength and ≥ 0.7 MPa for wet strength).

Direct use of the pMDI resin for bonding plywood in this study was met with some difficulties: when pMDI resin was applied to the veneer surface, it penetrated into the veneer quickly (within 2 to 3 min), resulting in a deficiency of adhesive spread onto the veneer surface. To address this issue, the plywood had to be hot-pressed as soon as the pMDI resin was applied to the veneer. This could be done under lab conditions but was not acceptable in the factory.
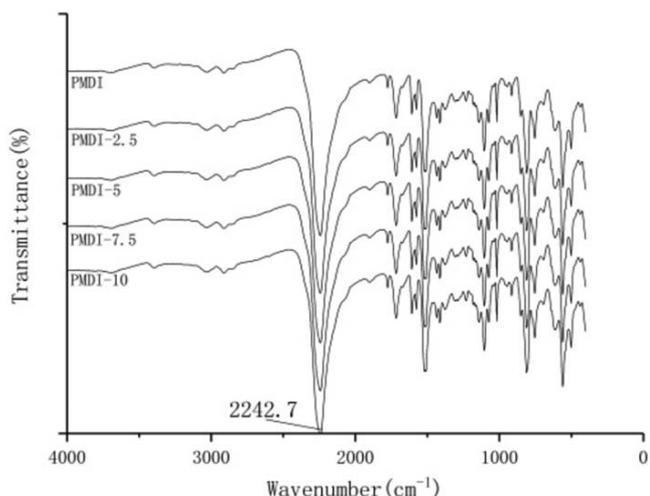

Figure 2.—Fourier-transform infrared spectrum of the adhesives obtained by mixing polymeric diphenyl methane diisocyanate (pMDI) with polyurethane powders at various weight ratios.
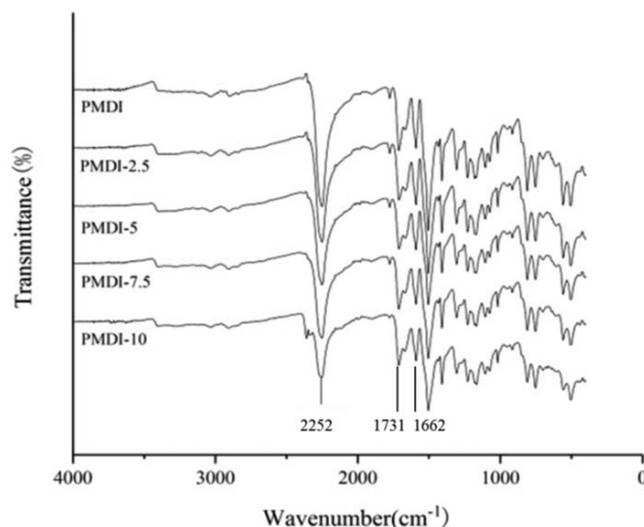

Figure 3.—Fourier-transform infrared spectrum of the cured adhesives obtained by mixing polymeric diphenyl methane diisocyanate (pMDI) with polyurethane powders at various weight ratios.

Table 2.—Thermogravimetric (TG) parameter of polyurethane (PU) and the adhesives with different PU additions.

Adhesive[a]	Temperature range (°C)	Weight loss (%)	Residual percentage at the end of TG test (%)
PU	200–300	28	8
	350–400	55	
PMDI	150–300	59	22
	400–600	19	
PMDI-2.5	150–300	55	21
	400–600	24	
PMDI-5	150–300	53	21
	400–600	26	
PMDI-7.5	150–300	52	22
	400–600	26	
PMDI-10	150–300	51	22
	400–600	27	

[a] pMDI = polymeric diphenyl methane diisocyanate; pMDI-2.5 = pMDI with 2.25 percent PU; pMDI-5 = pMDI with 5 percent PU; pMDI-7.5 = pMDI with 7.5 percent PU; pMDI-10 = pMDI with 10 percent PU.

Figure 4.—Thermogravimetric (TG) analysis of the polyurethane (PU) powders and the adhesives. pMDI = polymeric diphenyl methane diisocyanate; pMDI-2.5 = pMDI with 2.25 percent PU; pMDI-5 = pMDI with 5 percent PU; pMDI-7.5 = pMDI with 7.5 percent PU; pMDI-10 = pMDI with 10 percent PU.

In a mill, usually, after the adhesive is applied, at least 30 minutes can expire before plywood hot-pressing.
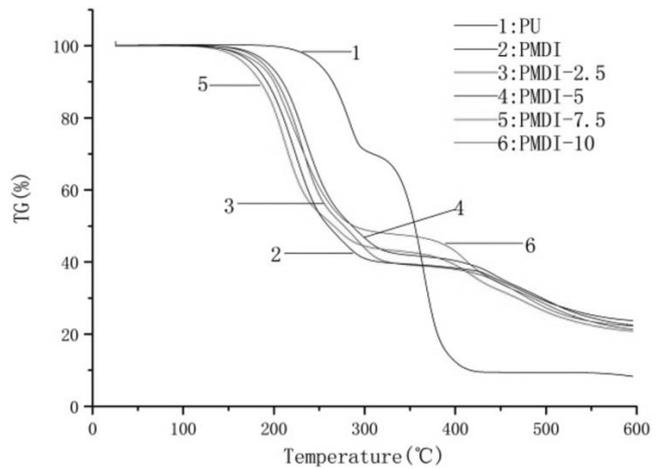
Statistical analysis revealed that the addition of PU powder affected the dry strength of the plywood specimens significantly. The dry bond strength of adhesives increased with PU powder substitution (from 2.5% to 7.5%). At a PU content of 7.5 percent, the strength value reached the highest point (2.68 MPa). As the PU content increased from 7.5 to 10 percent, the dry bond strength value decreased to 1.87 MPa. The dry bond strength is affected by the homogeneity and fluidity of the adhesives (Jin et al. 2010).The addition of PU foam powder into pMDI resin increased adhesive viscosity. Even with a PU foam powder content of 7.5 percent, the pMDI-7.5 adhesive still had an appropriate viscosity and could be spread well on the veneer surface. (In a mill, the adhesive should have viscosity around 2,000 cP.) This could contribute to good dry bond strength. However, when the PU powder content increased from 7.5 to 10 percent, the adhesive viscosity increased quickly from 2,231 to 3,280 cP (Table 1). High viscosity would affect the wetting ability of the adhesive, the spread of the adhesive to the wood surface, and also the depth of penetration of adhesive into wood, in a way that is counterproductive. These are all important factors for the formation of a good dry bond. Therefore, when PU content increased to 10 percent, both the dry bond strength and wood failure

percentage decreased obviously, indicating an inappropriate content of PU foam powder used at 10 percent.

Statistical analysis also shows that the addition of PU powder affected the wet strength of plywood specimens. The wet strength of plywood bonded by pMDI resin was 1.07 MPa. When the PU content increased from 2.5 to 7.5 percent, the wet strength value of plywood increased from 1.72 to 2.26 MPa. After a boiling-water treatment, the plywood samples only had a minor decrease in bond strength value when compared with those of dry samples. This indicated that the addition of PU powder into pMDI resin increased both bonding and water resistance of the plywood samples. This might be attributed to the reaction of PU foam powder with pMDI resin, as indicated by FTIR results (Fig. 3). However, when the PU powder content increased to 10 percent, the wet strength value decreased to 1.67 MPa, indicating that too much powder could overwhelm the pMDI system and minimize the cross-link density. This could result in a worse wetting ability of adhesive to veneer surface, lacking in fluency of adhesive spreading, and insufficient penetration of adhesive into wood because of high adhesive viscosity. Moreover, the uniformity of the adhesive layer would also be disturbed by excessive PU powder. Based on bond strength testing, the best addition amount of recycled PU powder was 7.5

Table 3.—Bonding performance of the plywood.

Adhesive samples[a]	Dry		Wet (boiling water treatment)	
	Mean (SD) bonding strength (MPa)	Wood failure (%)	Mean (SD) bonding strength (MPa)	Wood failure (%)
PMDI	1.28 (0.11) A[b]	30	1.07 (0.04) A	20
PMDI-2.5	1.77 (0.12) B	80	1.72 (0.11) B	60
PMDI-5	2.11 (0.09) C	100	1.88 (0.07) C	100
PMDI-7.5	2.68 (0.14) D	100	2.26 (0.13) D	100
PMDI-10	1.87 (0.07) B	60	1.67 (0.09) B	50

[a] pMDI = polymeric diphenyl methane diisocyanate; pMDI-2.5 = pMDI with 2.25 percent polyurethane; pMDI-5 = pMDI with 5 percent polyurethane; pMDI-7.5 = pMDI with 7.5 percent polyurethane; pMDI-10 = pMDI with 10 percent polyurethane.
[b] Data in the same column with the same letter are not different at $\alpha = 0.05$.

percent considering both the bond strength and production practice of plywood.

Conclusions

In this study, PU foam wastes from ASR were cleaned, ground, dried, and mixed with pMDI at PU powder weight percentages of 2.5, 5, 7.5, and 10 percent. The mixed adhesives were examined with FTIR and TG, and evaluated as plywood binders, with the following findings:

1. The addition of PU powder into pMDI resin increased the adhesive viscosity and solved the problem of deficiency of the adhesive on the veneer surface when using pMDI as plywood binders. At a PU foam powder substitution higher than 10 percent, the adhesive application became difficult due to excessive viscosity.
2. FTIR results indicated that some reactions might happen between pMDI resin and PU foam powder. This could help increase the cross-link density of cured adhesives and thus contribute to the cohesion strength.
3. The addition of PU powder into the pMDI resin decreased the weight loss of the cured adhesives and appeared to increase the thermal stability between 150°C and 300°C.
4. Plywood tests results revealed that both the dry and wet bond strength were increased. The optimal addition of PU foam powder was 7.5 percent based on bond strength data.

In summary, the addition of PU powder into pMDI resin provided a new way to utilize recycled PU foam waste and made pMDI resin a potential suitable plywood binder by preventing penetration into the wood structure. Future studies will be focused on the reaction mechanism between pMDI resin and PU foam powder as well as optimization of adhesive formation and plywood production.

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