WORLD CONFERENCE ON TIMBER ENGINEERING 2025
BRISBANE, AUSTRALIA
Advancing Timber for the Future Built Environment

MODIFICATION OF MELAMINE FORMALDEHYDE-BASED ADHESIVES FOR THE MANUFACTURING OF CROSS-LAMINATED TIMBER (CLT) FROM JABON WOOD

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ABSTRACT: Adhesive is an essential element that requires meticulous consideration throughout the production process of Cross-Laminated Timber (CLT). This study aims to investigate the bonding performance of jabon wood-CLT manufactured with modified melamine formaldehyde (MF)-based adhesives for cold-pressing applications. The commercial MF adhesives (MF-0) and modified ones (MF-1) at three different rates, 250, 280, and 300 g/m^2 were applied in this study. The MF-1 was composed of MF-0 by adding 5% citric acid, 3% polymeric 4,4-methylene diphenyl diisocyanate (pMDI), and 10% wheat flour. The results show that the MF adhesive can be modified by incorporating citric acid and PMDI, enabling it to be utilized in the production of CLT using cold pressing application. Adding these substances enhanced the adhesive viscosity and shortened the gelation time. In comparison to MF-0, MF-1 has a larger contact angle and a lower *K*-value due to its increased viscosity. However, this modification results in adhesive performance of MF-1 (300 g/m^2) that complies with EN 16531:2021. Increased spread of the MF-1 led to improved bonding strength, reduced delamination, and enhanced wood failure. MF-1 demonstrated a higher stress-strain curve and stiffness compared to MF-0 indicates that it provides enhanced performance for bonded wood products such as CLT.

KEYWORDS: cross-laminated timber, jabon wood, melamine formaldehyde, cold-pressing, bonding performance

1 – INTRODUCTION

Cross-laminated timber (CLT) is a modern engineered wood product that can be applied as a component material in wooden building. CLT is manufactured by stacking numerous layers of timber with a specified thickness, in which each layer is positioned orthogonally and glued together using structural adhesives [1,2]. The choice of suitable raw materials and the application of appropriate adhesives influence the quality of the manufactured CLT products. Adhesives are an essential factor in the manufacturing of wood composite products, including CLT [3]. The adhesive cost component could amount to 32% of the overall production expenses for wood composites [4]. Presently, there are no established regulations governing the requirements or specifications for adhesives used in the production of CLT. CLT products are primarily designed as structural components; thus, employing a structural adhesive type is the most suitable method.

2 – BACKGROUND

Common adhesives utilized in the manufacturing of CLT products include phenol-resorcinol-formaldehyde (PRF), melamine-formaldehyde (MF), emulsion polymer isocyanate (EPI), and polyurethane (PU) [5]. The types of adhesives commonly used to produce CLT in Europe are melamine-urea-formaldehyde (MUF) and PU [6,7] Meanwhile, in Japan, CLT is produced using MF, PRF, and isocyanate adhesives [7]. Wood-based

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https://doi.org/10.52202/080513-0595

panel products for exterior and semi-exterior use, including CLT, commonly use MF-based adhesives [8] (Lan et al. 2019). MF is a commonly selected adhesive by glulam or CLT producers because to its cost-effectiveness, transparent adhesive line, and resistance to both heat and moisture [3,9]. As a thermosetting adhesive, MF is generally applied using a hot press, which causes increased production costs due to heat energy consumption. Modifying MF-based adhesives to apply them in a cold press process for CLT product manufacture requires innovative steps. We expect these efforts to reduce heat energy consumption in the manufacture of CLT without compromising its mechanical strength properties. Modifying MF-based adhesives is a highly encouraging method for enhancing the economic advantages of these adhesives while safeguarding the environment [10]. The objective of this study is to modify an MF-based adhesive to make it suitable for the manufacturing of CLT through the cold pressing method. The quality and bonding mechanism of jabon wood and MF-based adhesive on CLT product were investigated in this study.

3 – PROJECT DESCRIPTION

This research was conducted from June 2022 to December 2023. Modification and characterization of adhesives, as well as the manufacture of CLT samples, were carried out at the Integrated Laboratory of Bioproducts (iLab), National Research and Innovation Agency (BRIN), Cibinong, Indonesia. Physical-mechanical testing of CLT products was carried out at iLab-BRIN and the Wood Building Engineering and Design Laboratory, Department of Forest Products, Faculty of Forestry and Environment, IPB University, Bogor, Indonesia.

The study utilized commercial MF adhesive (MA-204) sourced from PT Pamolite Adhesive Industry (Probolinggo, Indonesia), pMDI from PT Anugerah Raya Kencana (Tangerang, Indonesia), citric acid, and wheat flour. Jabon wood (*Anthocephalus cadamba* Roxb. Miq.) with a density of 0.44 g/cm^3 and a moisture content of 12 ± 2% was used in this study. Jabon wood was sourced from the community around Bogor, Indonesia. The equipment that was utilized included electric scales, ovens, pH meters, rheometers, gel time meters, dynamic mechanical analyzers (DMA), circular saws, moisture meters, digital calipers, sanders, cold-presses, iron clamps, universal testing machines (UTM), portable stylus-type profilometers (Mitutoyo Surftest® SJ-210, Mitutoyo Corporation, Kanagawa, Japan), and the Dino-Lite digital microscope Basic Am 2111 series (AnMo Electronics Corporation, New Taipei City, Taiwan).

3.1 MODIFICATION OF ADHESIVES

The commercial MF adhesive, as a control (MF-0), was modified incorporating citric acid (20% wt) as a catalyst, pMDI (96.2% wt) as a cross-linker, and wheat flour as a filler (Fig.1). Citric acid and pMDI were included in proportions of 5% and 3%, respectively, relative to the solid content of the MF-0 adhesive. The total mixture included 10% wheat flour. All adhesive components were mixed and stirred evenly for 1-2 minutes at a temperature of 27±2°C, resulting in a modified MF adhesive (MF-1) in paste form, prepared for characterisation and application in the cold pressing process of CLT manufacturing.

3.2 CHARACTERIZATION OF ADHESIVES

The adhesive's pH was measured using a pH meter (Laqua, Horiba, Kyoto, Japan) in accordance with the procedure outlined in previous study [12]. The solid content of the MF-0 and MF-1 adhesives was determined by weighing 2 g of adhesive in an aluminium foil container, drying it in an oven at 105°C for 3 hours, and subsequently reweighing the sample. The solid content was determined using the ratio of the dry weight of the glue post-oven treatment to its the initial weight. The adhesive's viscosity was measured using a rotational rheometer (RheolabQC, Anton Paar, Graz, Austria), following the methodology outlined in previous work [13]. The duration required for the glue to achieve gelation was measured using a gel time meter (Techne GT-6, Coleparmer, Vernon Hills, IL, USA). Approximately 50 g of glue was placed into a test tube with the meter needle submerged in the sample. The duration for the glue to cure at a temperature of 100±3°C was monitored and documented in three trials.

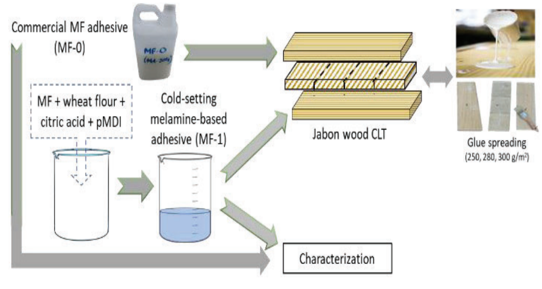

Figure 1. Modification dan formulation of MF-bases adhesive
(Source: Amin et al. 2023 [11], modified)

The thermo-mechanical characteristics of the MF-0 and MF-1 adhesives were examined utilizing a dynamic mechanical analyzer (DMA 8000, Perkin Elmer Inc., Waltham, MA, USA) [14].

3.3 EVALUATION OF SURFACE ROUGHNESS AND WETTABILITY PROPERTIES OF JABON WOOD

Prior to the application of adhesive for CLT manufacturing, the surface roughness and wettability of jabon wood were initially investigated. The surface roughness of jabon wood, sanded with a 100 grit (P100) belt sander, was measured using a portable stylus-type profilometer (Mitutoyo Surftest® SJ-210, Mitutoyo Corporation, Kanagawa, Japan). The contact angle (θ) of MF-0 and MF-1 adhesives on the jabon wood surface was determined employing a method referred to as low-bond axisymmetric drop analysis (LB-ADSA) [15]. The wettability of MF-based adhesives was determined by evaluating the rate of change for a constant contact angle (*K* value) adopting the S/G model [16].

4 – EXPERIMENTAL METHODS

4.1 APPLICATION OF ADHESIVE FOR THE MANUFACTURING OF JABON WOOD CLT

The fabrication of CLT from jabon wood with the application of MF-0 and MF-1 adhesives refers to the EN 16351:2021 standard [17]. CLT is composed of three layers, each of which measures 330 mm (l), 110 mm (w), and 60 mm (t). Each adhesive type is applied three times, leading to the glue spreads of 250, 280, and 300 g/m². The cold pressing procedure involves the production of CLT with the application of MF-1 adhesive, which is maintained at a pressure of 1 MPa for a duration of 2 hours. In the application of MF-0 adhesive, CLT is produced through a cold pressing process at a pressure of 1 MPa. The CLT is subsequently secured, removed from the pressing machine, and heated in an oven at 100°C for 2 hours. All CLT samples that have been made are then conditioned indoors for ± 7-14 days to standardize the water content and release residual stress due to the pressing process.

4.2 PHYSICAL AND MECHANICAL PROPERTIES TESTING OF CLT

The moisture content of CLT was measured using a moisture meter (mini-Ligno E/D, Lignomat, Oregon, USA). The delamination characteristics were evaluated according with EN 16531:2021 [17]. Test specimens measuring 100 mm × 100 mm × 6 mm were submerged in water at a temperature of 10-20°C within a pressure vessel and subjected to a vacuum of 85 kPa for 30 minutes. After the vacuum release, it was subjected to a pressure of 600-700 kPa for a duration of 2 hours. The samples were then removed and dried at a temperature of 65°C for 12-20 hours. Delamination was determined by calculating the percentage of adhesive area detachment between the laminae, delamination ratio, using the following equation:

$$\text{Delamination}_{\text{rat}} = 100\ \frac{l_{\text{tot.delam}}}{l_{\text{tot.glue line}}}$$

where $l_{\text{tot.delam}}$ is the total delamination length, $l_{\text{tot.glueline}}$ is the sum of the perimeters of all glue lines in a delamination specimen.

The mechanical properties testing (adhesion strength) of CLT was conducted utilizing the block-shear strength analysis method, as described in previous study [18]. The test used a universal testing machine (UTM AG-IS 50 kN, Shimadzu, Kyoto, Japan) operating at a speed of 2 mm/min.

5 – RESULTS

5.1 CHARACTERISTICS OF MF-BASED ADHESIVES

The results showed that the solid contents of MF-0 and MF-1 were approximately 51.89% and 48.56%, respectively. The solid content of melamine-based adhesives increased as the concentration of melamine increased [19]. Adding citric acid, pMDI, and wheat flour to the MF adhesive mixture changed the balance of its properties and ingredients, which caused the solid content in the adhesive to decrease. The solid content of MF-1 decreased because of the addition of citric acid, pMDI, and wheat flour to the MF-0 adhesive. The addition of 5% citric acid and 3% pMDI into MF-0 resulted in a lower pH value for MF-1 compared to that of MF-0, with pH values of 6.67 and 7.67, respectively. The reduced pH in MF-1 is attributed to the carboxylic acid groups (–COOH) in citric acid reacting with free formaldehyde (CH_2O) present in the MF-0 adhesive. The duration of time required for the adhesive to transform into a gel is known as the gelatinization time. The bonds between adhesive molecules are made stronger by gel formation, which leads to a strengthened overall bond. The gelatinization time of MF-1 (30.27 minutes) is almost 5 times faster than MF-0 (6.90 minutes). Incorporating 5% citric acid catalyst and 3%

https://doi.org/10.52202/080513-0595

pMDI cross-linker can reduce the gelation time of MF-based adhesives. The addition of this catalyst and cross-linker speeds up the time it takes for the adhesive to thicken by helping to create longer molecular chains. A previous investigation [20] revealed that the gelation time of MF-based adhesives can be decreased by the incorporation of 1–2% pMDI. A short gelatinization time will speed up the maturation or hardening process of the adhesive when applied. During the gel formation process, the viscosity of the adhesive increases, and it becomes harder and rougher. The gel point occurs when the viscosity is very high and the adhesive forms a gel. The average viscosity of MF-1 (570.75 mPa·s) was higher than that of MF-0 (484.51 mPa·s) due to the formation of a bigger molecular chain from the interactions of MF, citric acid, and pMDI. The viscosity of an adhesive correlates with its cohesive strength. Increased viscosity signifies enhanced resistance to deformation, leading to strengthened internal connections within the adhesive. The higher viscosity of MF-1 results in higher cohesive strength compared to MF-0. So, CLT bonded with MF-1 adhesive is predicted to exhibit better properties compared to MF-0.

5.2 SURFACE ROUGHNESS AND WETTABILITY PROPERTIES OF JABON WOOD

The investigation of the wood adhesive interface entails an analysis of the interaction between the wood surface and the adhesive substance. Wettability refers to the capability of a liquid (adhesive) to interact with a solid surface (wood) [21]. The adhesive's wettability characteristics on wood are affected by the surface roughness of the wood. Surface roughness measurements demonstrate that jabon wood, after being sanded with a 100-grit belt sander (P-100), has a roughness average (Ra) between 5.62 and 6.94 µm. Increase in surface roughness results in a decrease in the contact angle, which sub-sequently leads to enhanced wettability and bonding performance [22,23]. The adhesive characteristics on wood are affected by the quantity of adhesive disperses and penetrates the wood pores (K-value). In our study, the higher viscosity of MF-1 compared to MF-0 made it more difficult for the adhesives to adhere to the wood surface. This means it takes longer to achieve the equilibrium contact angle. The MF-1 has higher contact angle and lower K-value than that of MF-0. MF-0 adhesive exhibits a higher K value (0.262–0.331) than the MF-1 adhesive (0.136–0.212). As K values increase, the time needs for the adhesive to spread and penetrate, and for the contact angle to reach a state of relative equilibrium is reduced. A high K value indicates excellent wettability, enabling the adhesive's dispersion and absorption into the wood surface's pores. The increased viscosity of MF-1 relative to MF-0 restricts the adhesive's absorption into the wood surface, leading to a greater contact angle compared to MF-0. The MF-1 adhesive requires an extended duration to achieve the equilibrium contact angle when applied to the wood surface of jabon wood.

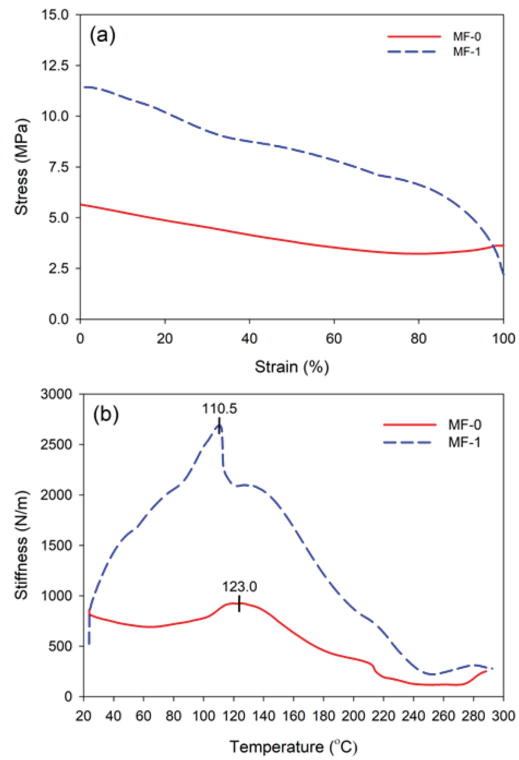

Figure 2. DMA results of wood–MF adhesive: (a) stress–strain curve and (b) stiffness as a function of temperature (Source: Amin et al. 2024 [24])

The thermomechanical (viscoelastic) properties of MF-based adhesives were evaluated through DMA testing. Fig. 2a shows that MF-1 has a higher stress-strain curve value in comparison to MF-0. A higher stress-strain curve represents a higher stiffness in a material, indicating that the material exhibits higher resistance to deformation during dynamic loads. Fig. 2b shows that the interface of jabon wood-MF-0 achieved a maximum stiffness of 957 N/m at 123.0 °C, while jabon wood-MF-1 had a maximum stiffness of 2734 N/m at 110.5 °C. The findings of this investigation indicated that MF-1 exhibited a greater degree of rigidity and could potentially provide better performance in bonded wood products than MF-0. By DMA analysis, at a temperature of 128.9 °C, wood–MF-0 has a maximum storage modulus (E'max) of 12,650 MPa, whereas wood–MF-1 has a maximum storage modulus (E'max) of 22,950 MPa at a temperature of 113.5 °C.

5.3 PHYSICAL AND MECHANICAL PROPERTIES OF JABON WOOD CLT

The integrity of adhesion, covering bond strength and delamination, is a critical consideration for both consumers and CLT manufacturers [25,26]. The bonding strength and delamination are affected by the adhesive type, the wood species, and the wood's surface treatment [5]. This investigation revealed that the commercial MF adhesive (MF-0) was ineffective in the CLT manufacturing trial using the cold pressing method. MF-0 adhesive was effectively used to fabricate CLT from jabon wood, incorporating heat treatment at 100°C for a duration of 2 hours. Meanwhile, MF-1 adhesive applied with cold pressing (1 MPa, 2 hours) successfully produced jabon wood CLT that bonded completely for the three applied glue spreads.

The bonding performance of jabon CLT was evaluated by block shear strength testing, revealing that the adhesive strengths of MF-0 with glue spreads of 250 g/m², 280 g/m², and 300 g/m² were 1.03 MPa, 1.94 MPa, and 2.13 MPa, respectively. The block shear strength of the MF-1 is approximately 50% more than that of the MF-0, with the highest value of 3.14 MPa reached at a glue spread of 300 g/m² (Fig. 3).

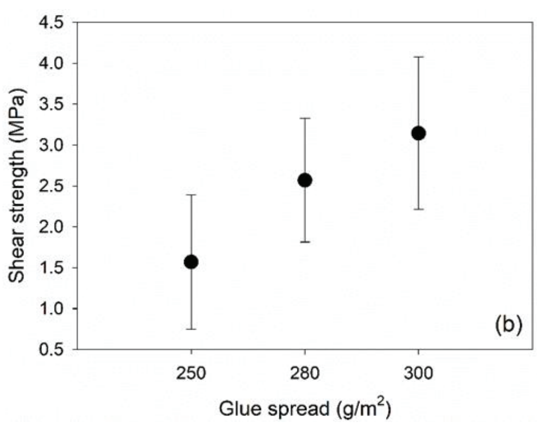

Figure 3. Block shear strength of jabon CLT bonded with MF-1 adhesive at different glue spreads

The adhesive's capability to withstand shear stress is of the greatest importance in CLT panel products, as it can result in damage if the adhesive weakens in the layer that crosses the main direction of the panel [28]. The MF-0 adhesive exhibits an average delamination range of 26.30–13.07% with a wood failure percentage below 20%. The delamination and wood failure percentages of MF-1 exceeded those of MF-0. Compared with MF-0 adhesive, the percentage of wood failure in CLT products is almost three to five times higher when using MF-1 adhesive. The application of 300 g/m² of glue (MF-1) resulted lowest delamination and highest wood failure percentages at 5.70% and 98.33%, respectively (Fig. 4). The amount of delamination and wood failure in jabon CLT with MF-1 (300 g/m²) met the standards set by EN 16531:2021, which states that delamination should be under 10% and wood failure should be at least 90% [3].

These results aligned with the DMA findings, which revealed that MF-1 has a greater storage modulus than MF-0. The incorporation of 5% citric acid and 3% pMDI improved the bonding performance of CLT jabon wood by allowing the formation of larger molecular chains through the interaction of MF, citric acid, and pMDI. The creation of extensive molecular chains in the MF-1 adhesive resulted in strong adhesion and possibly improved its bonding to the wood surface [27].

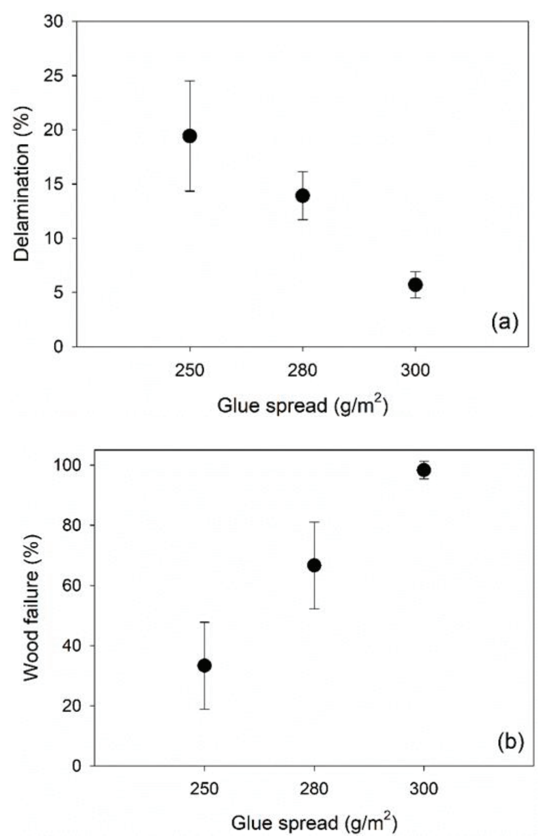

Figure 4. Delamination (a) and wood failure (b) of jabon CLT bonded with MF-1 adhesive at different glue spreads

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6 – CONCLUSION

The potential of MF-based adhesives as cold-setting adhesives for future CLT production has been demonstrated through the modification of these adhesives by combining them with citric acid and pMDI, particularly considering the advantages of thermal energy consumption. The adhesive characteristics and bonding performance of modified MF-based adhesives (MF-1) exhibited improvements in comparison to commercial MF (MF-0). The stress-strain curve and stiffness of MF-1 were higher than those of MF-0, which implies that it provides superior performance for bonded timber products, such as CLT.

7 – ACKNOWLEDGEMENTS

The authors are really grateful for the support provided by the Ministry of Higher Education, Science, and Technology (KEMDIKTISAINTEK) and IPB University through the Doctoral Dissertation Research Grant program for the fiscal years of 2022 and 2023 (contract numbers 3798/IT3.L1/PT.01.03/P/B/2022 and 15857/IT3.D10/PT.01.02/P/T/2023).

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