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Characteristics of Eco-Friendly and Sustainable Plywood Adhesive derived from Low-Quality Cat's Eye Damar Resin

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ABSTRACT

Wood panel products mainly use formaldehyde-based adhesives that release free formaldehyde and potentially cause health problems. This study aimed to develop a free-formaldehyde adhesive from cat's eye damar (CED) resin as an alternative adhesive for plywood production. The low-quality CED resin was used to increase the added value of the resin. The adhesive formulation consists of a ratio of 30:70 (CED:benzene) dissolved for 15 minutes at 45°C. The plywood was manufactured using glue spread rates of 200, 250, and 300 g/m^2 with an addition of 10% tapioca flour and hot pressed using a pressure of 2.45 MPa at 120°C for 6 minutes. The CED-based adhesive produced has a solid content of 28.76%, a pH value of 5.93, a gel time value of 70.05 minutes, and a viscosity value of 4.02 mPa.s. Fourier-transform infrared spectroscopy analysis stretching of the C-H group, indicating an alkane compound. Plywood's physical and mechanical properties bonded with CED-based adhesive increased with higher glue spread application. Utilizing a glue spread of 300 g/m^2 could produce plywood with comparable physical and mechanical properties to the urea-formaldehyde-bonded plywood.

1. Introduction

Plywood is preferred as a construction material and is conventionally necessary in light frame construction (Demir et al. 2018). An increase in the world's population is making a comeback in the housing and furniture construction industry. Plywood is used for floors, walls, and roofs in house construction and panels (Wang et al. 2021). After Brazil and the Democratic Republic of the Congo, Indonesia contains the third-largest tropical forest in the world, making Indonesia a country that produces various roundwood products, with plywood emerging as the most prominently utilized variant (FAO 2003; Ministry of Environment and Forestry 2021). However, plywood production is currently experiencing a total decline due to the availability of raw materials for making plywood decrease, thus affecting the performance of the plywood industry (Setiawan et al. 2014). Statistics Indonesia (2020) reported that plywood production started declining from 2018 to 2020. In 2018, plywood production was 4,213,557 m^3/year, then decreased in 2019 and 2020 plywood production became 3,862,923 m^3/year.

One way to overcome the shortage of plywood raw materials is to use rubber wood (*Hevea brasiliensis*), which is a type of plantation wood that has not been used optimally to date. Rubber wood is included in the strength class II wood group based on its mechanical properties (Woelan et al. 2012). Rubber trees over 25 years old must be regenerated to increase their latex productivity, and the wood from the trees can be used to make plywood (Setiawan et al. 2014).

The world's wood panel industry mainly uses formaldehyde-based adhesives, where about 90% of the wood panel industry uses these adhesives, and around 80% of the wood panel industries use urea-formaldehyde (UF) adhesives for the production of plywood and particle board (Athariqa et al. 2022). Adhesives combine two or more different materials into a new product (Frihart 2015). The common types of commercial formaldehyde-based adhesives include UF, phenol-formaldehyde (FF), phenol-resorcinol-formaldehyde (FRF), melamine-formaldehyde (MF), and resorcinol-formaldehyde (RF) (Merline 2013). This synthetic adhesive comes from non-renewable resources, namely from petroleum processing (Sulastiningsih et al. 2013). Many wood panel products for interior purposes still use UF adhesives.

UF wood adhesive is mainly used to manufacture wood-based panels because of its low cost, good thermal properties, colorlessness, mold resistance, and ease of use (Gonçalves et al. 2018). However, the weakness of UF wood adhesive is formaldehyde emission, which is readily hydrolyzed in an acidic and humid environment (Lubis and Park 2018). According to research, formaldehyde emission content of just 0.1 ppm can cause health problems, and at high concentrations, it can damage lung function and cause cancer (Liteplo et al. 2002). Pollution and health problems resulting from formaldehyde emissions have spurred the development of using organic materials from natural resources as environmentally friendly, renewable, and biodegradable adhesives (Santoso and Pari 2015). Availability of raw materials, novelty, and environmental friendliness are the main parameters for selecting alternative adhesive raw materials.

Krui, Pesisir Barat Regency is Indonesia's primary producer of CED resin, around 80% (Anasis and Sari 2015). The CED resin is derived from the damar (*Shorea javanica*) tree. On an area of 17,500 ha, Krui has roughly 1,750,000 productive damar trees (Febryano and Riniarti 2009). Based on Kurniawan (2021), the damar tree can produce a CED of 1.58 kg/tree within a month. Kurniawan (2021) stated that CED could be used as a raw material for making adhesives, varnishes, batik paints, hardeners, and waxes. There are five classifications of CED quality: Grade A (high quality), with a clear yellowish color and large lumps (3 cm × 3 cm or more), Grade B, with clear yellowish color and slightly smaller pieces (2 cm × 2 cm), Grade C, with small blackish yellow lumps (1 cm × 1 cm), Grade DE, with black color and in the form of small granules, and Grade Dust/Ash (low quality), in the form of dust and dirt. The CED size and quality affect the selling price. The CED quality that can be sold for export is usually CED with Grade A, while Grade Dust/Ash has a low selling value and cannot be exported.

Based on Setiawan et al. (2014), plywood from rubber wood with UF adhesive produced an adhesive strength of 9.98 kg/cm^2. The research results of Kasim et al. (2020) showed that the varnish made from low-quality resin raw materials with mixed formula of 65% resin solution, 25% synthetic alkyd, 8.8% kerosene, 0.3% dry cobalt, and 0.9% dry calcium produces good quality varnish. Based on these previous studies, it explained that rubber wood has the potential utilization to be used as a composite product. In addition, there is still a lack of study on low-quality CED resin as an adhesive for composite products. Therefore, this present study was carried out on plywood from rubber wood as raw material with CED adhesive as an environmentally friendly adhesive.

2. Materials and Methods

2.1. Materials

CED resin of low quality was obtained from Krui, Pesisir Barat Regency, Lampung Province. The rubber (*Hevea brasiliensis*) wood veneers for plywood manufacturing with a size of 30 cm × 30 cm × 0.2 cm were obtained from CV Kota Agung in Pringsewu, Lampung, with an average moisture content of 4.9%.

2.2. Adhesives Preparation

The CED resin with low quality was used for the research. The resin was then sorted and reduced in size by grinding and sifting. CED that passed the 60 mesh sieve was used for the adhesive manufacturing. The dissolving technique made liquid CED adhesive. Solid CED was modified by dissolving in a benzene solvent with a ratio of 30:70 (CED:benzene). The dissolution was stirred for 15 minutes at 45°C and then allowed to settle for ± 24 hours. The preparation and modification of the liquid CED adhesive process is presented in **Fig. 1.**

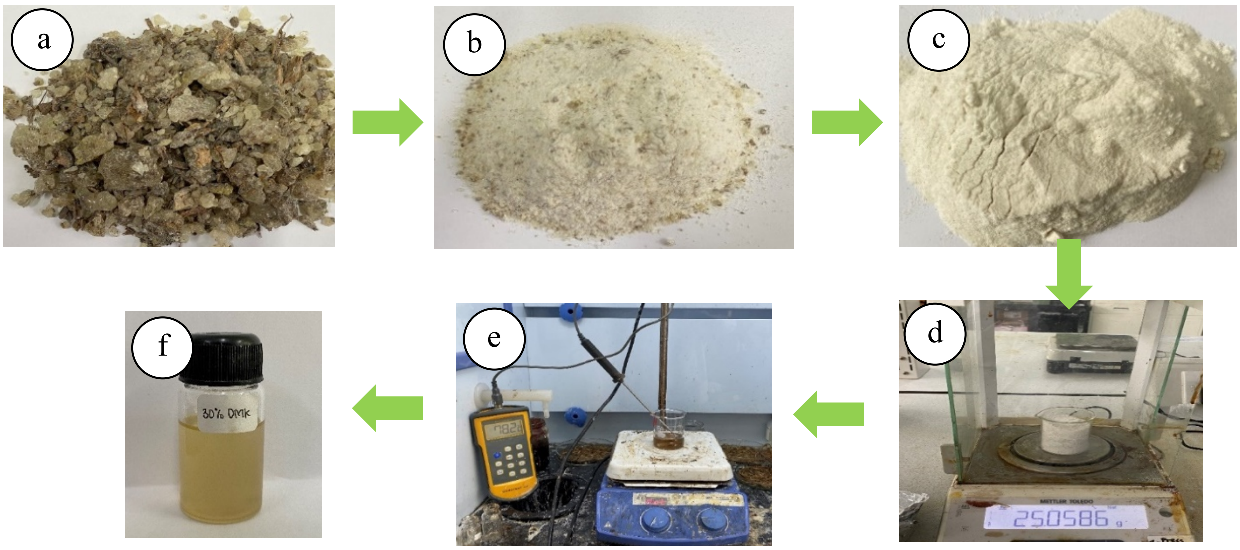


Fig. 1. Preparation and modification of CED adhesives (a) low-quality CED resin, (b) sorted and ground CED, (c) CED resin that passed 60 mesh sieve, (d) weighing CED resin, (e) dissolution of CED resin with benzene, and (f) CED adhesive.

2.3. Characterization of Adhesives

2.3.1. Solids content

A 1 g adhesive sample was placed on aluminum foil in an oven (Memmert Celsius 10.0, Memmert, Germany) for 3 hours at 103.3°C. The aluminum foil was moved to a desiccator and weighed after the sample had dried. Equation 1 was used to calculate the solids content.

$$Solids\ content\ (\%) = \frac{Oven\text{-}dried\ weight\ (g)}{Initial\ weight\ (g)} \times 100\% \quad (1)$$

2.3.2. Viscosity

A glass gauge (C-CC27, AntonPaar, Austria) holding about 20 mL of CED adhesive samples was placed on a rotating rheometer (RheolabQC, AntonPaar, Austria). A concentric cylinder (CC) spindle No. 27, rotating at 150 rpm, was used to measure viscosity. Experiments were conducted between 25–100°C to find out how temperature affected viscosity. For 120 seconds, the dynamic viscosity was measured.

2.3.3. pH value

The pH level is measured using a pH meter (Laqua pH 1200, Horiba, Japan). A few minutes later, the pH level was measured in the water after an electronic pH meter probe was placed in a container sample.

2.3.4. Gelation time

Differently treated CED adhesive was added to the test tube. The Techne GT-6 gel time meter in Coleparmer, USA, was set up with the needle immersed in the sample. The substance in the distilled water bath was utilized to raise the temperature to 100°C. The amount of time needed for the adhesive to gel was then measured. The sticky gelation time limit was determined when the timer automatically stopped and displayed the gelation time number indicated with “gel” on the screen.

2.3.5. Functional group analysis

Fourier Transform Infra-Red (FTIR) spectroscopy (SpectrumTwo, PerkinElmer, USA) with the Universal Attenuated Total Reflection (UATR) method was used to examine the functional groups of CED adhesive. The mean accumulation was measured using 16 scans at 4 cm^{-1} resolution and wavelengths between 4000 and 400 cm^{-1} at 23 ± 2°C. Each spectrum was adjusted using the min-max normalizing feature in the Spectrum software (Version 10.5.3, Perkin Elmer Inc., United States).

2.4. Adhesive Application in Plywood

To evaluate the adhesive strength, the adhesive was applied to a rubber wood veneer that was 30 cm × 30 cm × 0.6 cm and put in a three-ply plywood. Plywood panels were prepared with double-spreading CED adhesive with 200, 250, and 300 g/m^2 on the veneer. The addition of 10% tapioca flour filler from the adhesive will be applied to the plywood. Plywood was then hot pressed at a pressure of 2.45 MPa at 120°C for 6 minutes. Plywood bonded with UF adhesive at 180 g/m^2 of glue spread was prepared as a control.

2.5. Plywood Characteristics Testing

The physical-mechanical properties of plywood were analyzed based on Indonesian National Standard (SNI) 01-5008.7-1999 and Japanese Agricultural Standard (JAS) No. 233, 2003. Three physical characteristics were tested: delamination, moisture content, and density. Meanwhile, the mechanical characteristics tested were adhesive firmness, wood failure percentage, modulus of elasticity (MOE), and modulus of rupture (MOR).

2.5.1. Density

The density test sample with a dimension of (100 × 100 × 0.51) cm^3 was weighed, and the density was calculated using Equation 2.

$$D\ (g/cm^3) = \frac{W}{V} \quad (2)$$

where D is density(g/cm^3), W is the oven-dried weight (g), and V is the volume of the wood sample (cm^3).

2.5.2. Moisture content

The moisture content test sample with a dimension of (10 × 10 × 0.51) cm^3 was weighed to determine the initial weight of the test sample. Then, the test sample was dried in an oven for 24 hours at 103°C. The dry test sample is re-weighed. Moisture content can be calculated using Equation 3.

$$MC\ (\%) = \frac{100 \times (W_1 - W_0)}{W_0} \quad (3)$$

where MC is moisture content (%), W_1 is the initial weight (g), and W_0 is the oven-dried weight of the wood sample (g).

2.5.3. Delamination test

The delamination test was carried out by measuring the length of the adhesive lines on each side, both straight and cross, and adds up the adhesive lines. The test samples were dried for 3 hours at 60 ± 3°C after being submerged in hot water at 70 ± 3°C for 2 hours. By dividing the total length of the adhesive line by the whole length of the delamination line, the delamination percentage was determined. The length of the peeled-off adhesive line on all sides was measured and summed. The delamination percentage was calculated using Equation 4.

$$Delamination\ percentage\ (\%) = \frac{\Sigma\ Delaminated\ glue\ line}{\Sigma\ Total\ glue\ line} \times 100\% \quad (4)$$

2.5.4. Modulus of elasticity (MOE) and modulus of rupture (MOR)

The MOE and MOR tests used the universal testing machine (Model AGX, Shimadzu, Japan), conducted at an average loading speed of 14.7 MPa/minute up to the failure sample. The MOE and MOR values were determined using Equation 5 and Equation 6.

$$MOE\ (MPa) = \frac{\Delta P \times L^3}{4 \times \Delta y \times b \times h^3} \quad (5)$$

$$MOR\ (MPa) = \frac{3 \times P \times L}{2 \times b \times h^3} \quad (6)$$

where P is the maximum load (N), ΔP is the difference between the maximum and minimum load (N), L is the length of the span (mm), Δy is a variance in deflection between the top and bottom load (mm), b is the sample width (mm), and h is the sample thickness (mm).

2.5.5. Tensile shear strength

The tensile shear strength test used samples with a dimension of (5 × 5 × 0.51) cm^3, and the testing was conducted using the universal testing machine (Model AGX, Shimadzu, Japan). The tensile strength was calculated using Equation 7.

$$Tensile\ shear\ strength\ (MPa) = \frac{P}{2A} \quad (7)$$

where P is the highest stress at which the sample breaks (N) and A is the remaining bond area (mm^2).

2.5.6. Wood failure

Wood failure was analyzed using a tensile test sample right after analysis. Wood failure percentage can be calculated using Equation 8.

$$Wood\ failure\ (\%) = \frac{Area\ of\ failure\ to\ the\ adhesive\ area\ after\ testing\ (cm)}{Area\ of\ the\ measured\ area\ (cm)} \times 100\% \quad (8)$$

2.6. Data Analysis

Two variables were used to analyze the collected data: coating weight and adhesive formulation component, in a completely randomized method. Three repetitions of each treatment were performed. IBM SPSS Statistics 21 and Microsoft Excel 2019 were used to process the data. The data were then statistically evaluated using Analysis of Variance with a 95% confidence level (ANOVA). The parameters that indicated a significant effect were subjected to the Duncan Multiple Range Test (DMRT) to find the critical difference between treatments.

3. Result and Discussions

3.1. Characteristics of CED-based Adhesive

The essential characteristics of CED-based adhesives are presented in **Table 1**. The results revealed that the solid content of CED-based adhesive is 28.76%. The CED content in the adhesive will further increase the value of the solids content. This might be due to the higher molecular content of CED. The solid content is the number of molecules in the adhesive that bond with the molecules being bonded (Adelka et al. 2023; Aprilliana et al. 2022).

The pH value of the CED adhesive is 5.9, or weak acid. If the CED content in the adhesive increases, it will decrease the pH value or increase the acidity. CED has an acidic pH, so the adhesive hardens quickly and has a low shelf life (Sribudiani and Somadona, 2021). Gelation time is required for the adhesive to change phase from liquid to gel at a given temperature (Baskara et al. 2023). The gel time value in the results of this study was 70.05 minutes. This result does not meet the SNI 06-0060-1987 that the gel time for UF adhesive is < 60 minutes (Kartika and Pratiwi 2018). The CED adhesive has a viscosity of 4.0 mPa.s. The viscosity value influences how well the adhesive enters the pores in the wood (Hidayat et al. 2022). The standard viscosity value for UF adhesive for coatings required by SNI 06-0060-1987 is between 100–150 mPa.s. The viscosity value produced from CED adhesives has not been lower than that of UF adhesives (Lubis et al. 2019; Lubis and Park 2020), resulting in slower gelation time and lower solids content.

Table 1. Characteristics of CED-based adhesive 30% (w/v) compared to commercial UF adhesive

Properties	CED adhesive	UF adhesive (Lubis et al. 2019; Lubis and Park 2020)
Solids content	28.7	58.2
pH (25°C)	5.9	8.2
Gel Time (min) 100°C	70.1	3.4
Viscosity (mPa·s, T= 25-100°C)	4.0	181.5

High viscosity adhesives have a shorter shelf life because they pack more quickly and have poorer adhesive quality, which prevents them from penetrating the pores of the wood (Fang et al. 2014). The best bond for greater adhesion can be formed by adhesives with a high solids content and an appropriate viscosity. **Fig. 2** shows the viscosity value of CED-based adhesive at 30% (w/v). The highest viscosity value occurs at 20.18°C, decreases drastically to 63°C, and slowly rises to 100°C. The decrease in viscosity illustrates the shortening of the molecular chains, while the considerable molecular weight indicates the adhesive power produced (Nurhayati 2018). Adhesives with high viscosity will quickly harden, and the quality of the adhesive will increase.

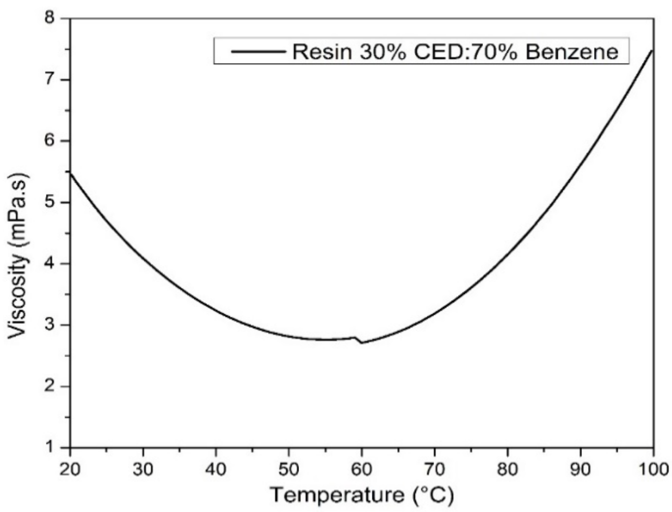
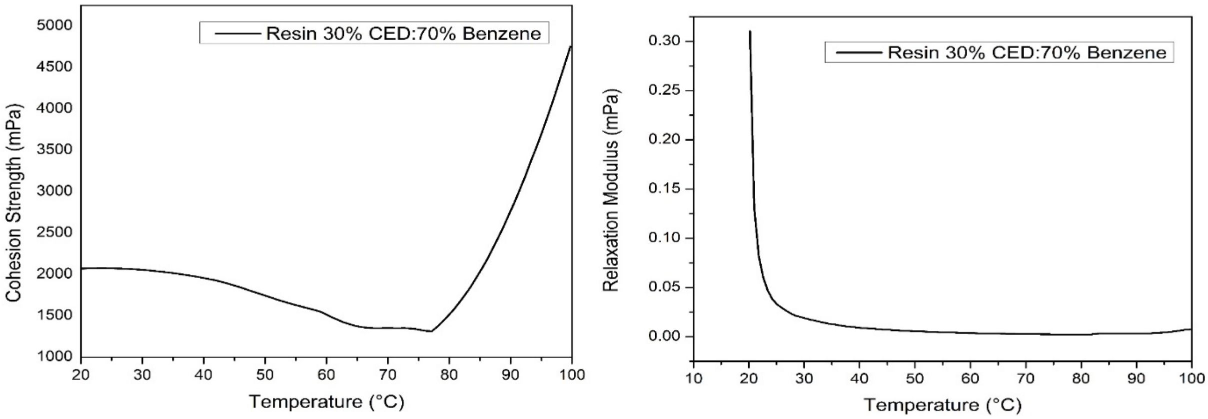


Fig. 2. Effect of temperature on the viscosity of CED-based adhesive 30% (w/v).

The research results show the value of the cohesive strength and relaxation modulus of the CED-based adhesive 30% at temperatures of 20–100ºC (**Fig. 3**). The adhesive will apply well to wood because of its proper viscosity and cohesive strength. The cohesive strength value decreased with increasing temperature up to 78ºC. After that, the cohesive strength value increased drastically (**Fig. 3a**). If the amount of coated adhesive is too much, the resulting adhesion force is weaker than the cohesion force. Hence, the adhesive power decreases (Wibowo et al. 2020). The graph shows that at 20°C, the CED-based adhesive has the highest relaxation modulus value (**Fig. 3b**). When the temperature is added, the strength value of the relaxation modulus decreases significantly up to 27°C and at subsequent temperatures tends to maintain its weight up to 100°C, the decrease is not significant.

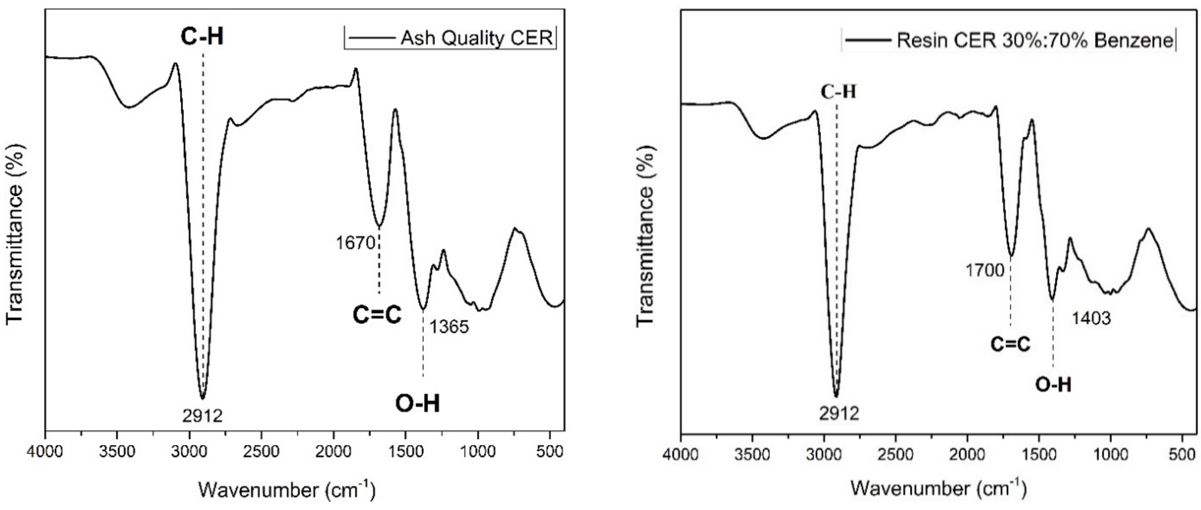


(a) (b)

Fig. 3. Effect of temperature on the cohesion strength (a) and relaxation modulus (b) of CED-based adhesive 30% (w/v).

3.2. Functional Group Analysis

Functional group analysis of low-quality CED adhesive (**Fig. 4**) showed that the absorption peak at wave number 2931.22 cm^{-1} is a stretching of the C-H group, indicating an alkane compound, the peak will shift towards the maximum if there is a change in the C-H bond structure. The wave number 1697.72 cm^{-1} shows the tension of the C=C group, which is a double vibration, showing the peak of alkene. The wave number 1373.51 cm^{-1} is the O-H flexible group, representing phenol compounds.



(a) (b)

Fig. 4. Typical FTIR spectra (a) low-quality CED material, (b) CED-based adhesive 30% (w/v).

Functional group analysis on 30% CED adhesive shows a high concentration of alkane groups (C-H) at wave number 2912 cm^{-1}. The peak will shift towards the maximum if there is a change in the C-H bond structure. Wave number 1700 cm^{-1} indicates muscular stretching of the C=C group, which may be caused by CED oxidation during dissolution. Wave number 1403 cm^{-1} is a stretch of the O-H group with phenol compounds.

3.3. Dynamic Mechanical Analysis (DMA)

DMA is a valuable technique for characterizing the mechanical properties of materials, including wood adhesives. DMA can provide insights into how these adhesives respond to various temperature, frequency, and time conditions, helping optimize the adhesive formulations and assess their performance (Aisyah et al. 2023; Hariz et al. 2023). As depicted in **Fig. 5**, DMA data provides the CED adhesive's viscoelastic behavior, such as its glass transition temperature (*Tg*), storage modulus, loss modulus, and damping properties (tan delta). These properties are critical for assessing the CED adhesive's performance in different applications. The *Tg* was observed at 70.5°C, showing that the CED adhesive became rubbery after reaching that temperature.

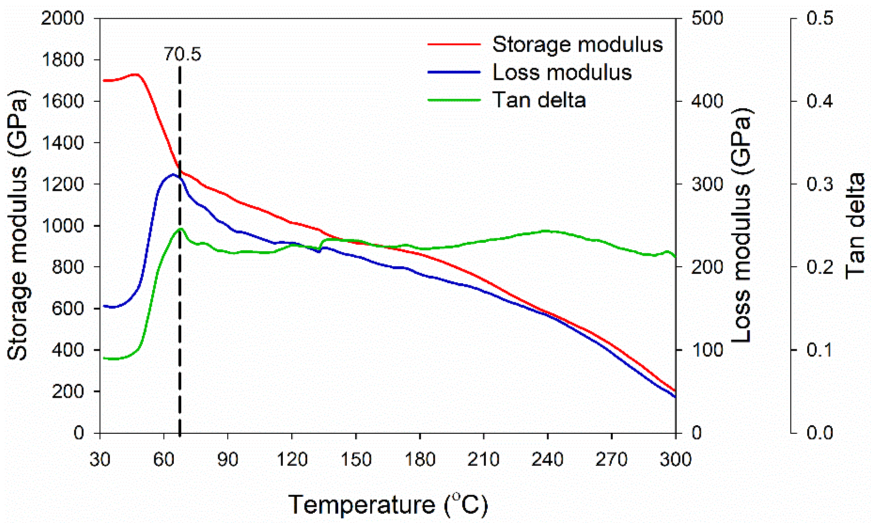


Fig. 5. DMA thermogram of CED-based adhesives 30% (w/v).

3.4. Properties of Plywood Bonded with CED-based Adhesives

Plywood is a flexible engineered wood product made from several layers of wood veneer glued together. These layers are usually oriented perpendicular to one another, which imparts plywood with unique properties and advantages. **Table 2** presents the physical properties of plywood bonded with CED-based adhesives at different glue spreads compared to commercial UF adhesives.

Table 2. Physical properties of plywood bonded with CED-based adhesives at different glue spreads compared to commercial UF adhesive

Properties of Plywood	CED adhesive (g/m^2)			UF adhesive (Lubis et al. 2019; Lubis and Park 2020)
	200	250	300	
Density (g/cm^3)	0.41	0.43	0.45	0.47
Moisture content (%)	6.83	7.15	7.36	5.70
Delamination (%)	100.0	100.0	100.0	100.00

The results show that, with greater glue spread application, the physical characteristics of plywood bonded with CED-based adhesives improved. Plywood bonded with CED-based adhesives had physical characteristics similar to those bonded with commercial UF adhesives. The density was in the range of 0.41–0.45 g/cm^3, with a moisture content of 6.83–7.36%. However, the plywood panel possessed 100% delamination, meaning the adhesive was not water-resistant. The research has met the standards required by SNI and JAS for water content < 14% and a delamination value of 100%, while the density value has not met the standard > 0.56 g/cm^3. Previous studies on plywood manufacturing made from rubber wood veneer and using UF adhesive had a density value of 0.63–0.65 g/cm^3 and moisture content of 14.43–14.60% (Setiawan et al. 2014).

Fig. 6 illustrates the mechanical characteristics of plywood bonded with CED-based adhesives at various glue spreads in comparison to commercial UF adhesives. In general, MOE and MOR values of plywood bonded with CED-based adhesives increased with higher glue spread application. The physical properties of plywood bonded with CED-based adhesives were comparable to those bonded with commercial UF adhesives. In previous studies on plywood manufacturing, Setiawan et al. (2014) found that plywood made from rubber wood veneer and using UF adhesive had a MOE and MOR value of 0.98 MPa and 2.1 MPa. These results indicated that CED-based adhesive could be an alternative adhesive for plywood.

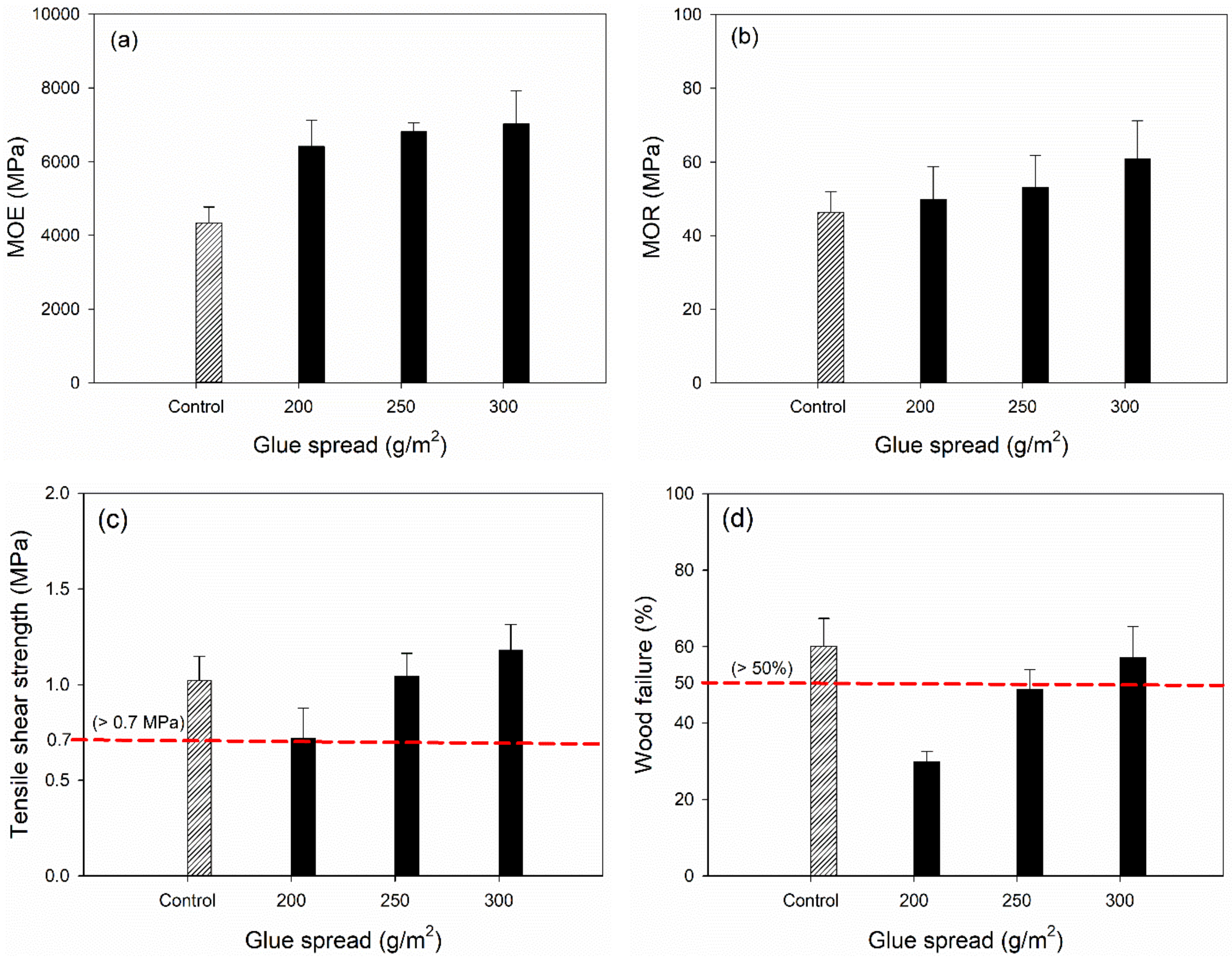


Fig. 6. Properties of Plywood bonded with CED-based adhesive. (a) MOE, (b) MOR, (c) Tensile shear strength, and (d) Wood failure percentage.

Furthermore, the tensile shear strength value of all plywood had met the minimum requirement of 0.7 MPa for plywood. However, only utilizing 300 g/m^2 of glue spread could result in the percentage of wood failure over 50%. Based on previous research related to Setiawan et al.

(2014), plywood made from rubber wood veneer and UF adhesive had a tensile shear strength value of 0.97 MPa.

4. Conclusions

Cat's eye damar (CED) resin adhesive derived from the meranti tree (*Shorea javanica*) has been successfully prepared. The CED-based adhesive has been applied as an alternative for plywood at glue spread of 200, 250, and 300 g/m^2. The results disclosed a solids content value of 28.76%, a pH value of 5.93, a gel time value of 70.05 minutes, a viscosity value of 4.02 mPa·s, and FTIR analysis and DMA led to an understanding of the use of CED as a plywood adhesive. Plywood-bonded physical and mechanical properties with CED adhesive increased with higher glue dispersion application. Using 300 g/m^2 of adhesive spread could produce plywood with the same physical and mechanical properties as UF-bonded plywood.

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