Performance of Oriented Strand Board Bonded with a Hybrid Phenol-Formaldehyde/Polymeric Methylene Diphenyl Diisocyanate Adhesives System

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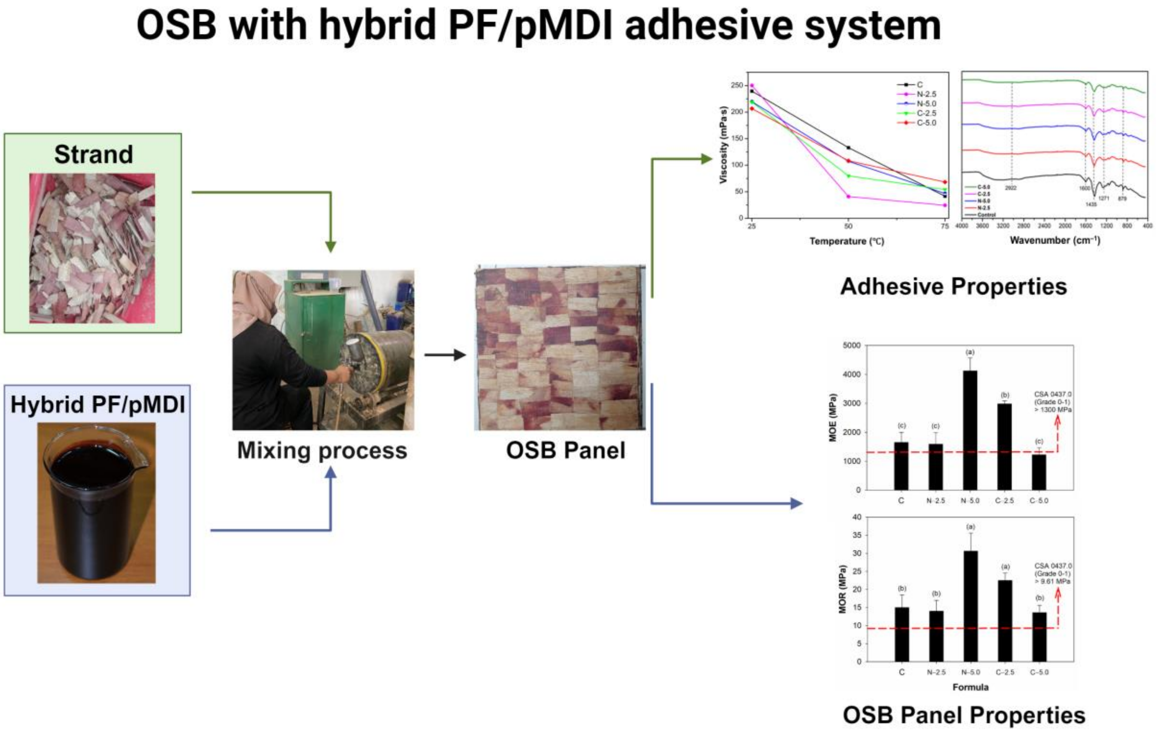
GRAPHICAL ABSTRACT



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A hybrid adhesive system composed of phenol-formaldehyde (PF) resin and polymeric methylene diphenyl diisocyanate (pMDI), modified with two types of alkaline catalysts, namely NaOH and $CaCO_3$ at 20% (w/v), was used for manufacturing the oriented strand board (OSB) from sengon (*Paraserianthes falcataria* L. Nielsen) wood. The catalyst was added at a concentration of 1% of the solids content of PF adhesive, and pMDI was added at 2.5% and 5.0% of the PF adhesive solids content. Adding catalysts and cross-linking agents increased the solids content and viscosity of the adhesive and accelerated the gelation time. The water absorption of OSB increased with the addition of catalysts and crosslinking agents compared to the control PF. Still, the $CaCO_3$ catalyst worked optimally in reducing the thickness swelling of OSB. The mechanical properties of the laboratory-fabricated OSB panels increased with the addition of catalyst and cross-linker, except for the modulus of elasticity parallel to the grain. The optimal performance of OSB was obtained by adding 1% $CaCO_3$ and 2.5% pMDI based on the PF's solids content.

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Keywords: Catalyst; Cross-linker; Mechanical properties; OSB; Physical properties; Thermosetting adhesive

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INTRODUCTION

Wood is a versatile construction material for houses and buildings that is both environmentally friendly and sustainable. Wood's inherent elasticity, strength, and lightweight nature provide a distinct advantage during earthquakes. Wooden structures' ability to flex and revert to their original shape after seismic events has made them a favoured construction choice for centuries in earthquake-prone regions, such as Indonesia (Mahmoud and Abbas 2020). Wood materials have the advantage of a higher strength-to-weight ratio compared to steel and concrete. Currently, the woodworking industry in Indonesia obtains its raw materials from Industrial Plantation Forests (IPF) and community plantation forests. The wood generated is typically a variety of lightweight, rapidly growing wood that possesses traits such as tiny diameter, low to medium density, limited natural resistance to decay, low dimensional stability, and numerous inherent flaws (Cahyono *et al*. 2017; Suri *et al*. 2021).

One alternative to using low-density wood is processing it into wood-based composite products. Directional strand board, also known as Oriented Strand Board (OSB), is a composite product commonly used as a structural material suitable for light, fast-growing wood, particularly in North America. OSB is a type of structural composite that may be manufactured using lignocellulosic materials, such as wood and bamboo. Prior studies indicate that hardwood OSB possesses a sleek exterior, although its capacity to maintain shape and its mechanical characteristics are subpar (Iswanto *et al*. 2010b; Hidayat *et al*. 2011, 2017). The assessment of OSB's superiority as a construction material is contingent upon several factors, including its fundamental properties, strength, resistance to naturally destructive organisms, resistance to fire, and other relevant considerations. Apart from that, OSB has advantages, including an easy manufacturing process, overcoming knot defects in raw materials, homogeneous characteristics, properties isotropic, abundant in supply, cheap in price, and quite high shear strength (Shmulsky and Jones 2011; Febrianto *et al*. 2017).

Sengon (*Paraserianthes falcataria* L. Nielsen) wood is one of the major IPF-harvested materials in Indonesia. The production of sengon wood experienced a significant growth of 62,270 m^3 from 2019 and 2021. Sengon is a rapidly growing plant species that yields a total wood production of 152,014 m^3 (BPS 2022). Sengon wood has been widely used in various wood products and their derivatives such as interior crafts, pallets, fuel (Anis *et al*. 2021), pulp (Winarni *et al*. 2020), fiberboard (Sufi *et al*. 2018), laminated veneer lumber, and barecore. Currently, sengon wood is the primary material for manufacturing OSB and cross-laminated timber (Hidayat *et al*. 2011; Baskara *et al*. 2022). The potential use of sengon wood as raw material for OSB is rated quite good. This is supported by OSB consumption in the United States in 2019-2020, which increased from 19,770 to 20,100 m^3 (Baskara *et al*. 2022).

Previous research suggests that the typical vulnerabilities are linked to mechanical characteristics such as bending strength (modulus of elasticity) and fracture strength (modulus of rupture), as well as internal bonding (IB) in OSB (Baskara *et al*. 2022). Several factors can contribute to this weakness, such as the type of raw material, compression ratio, glue type and content, strand geometry, wood extractive chemicals, and pretreatment (Febrianto *et al*. 2017). A study reported that by using phenol-formaldehyde (PF) adhesive with a mixture of NaOH and $CaCO_3$ catalysts, it was able to provide adhesive strength values that met SNI 01-5008.2 standards (Fitrianum *et al*. 2023). The production of OSB from gmelina wood, employing a 5% concentration of polymeric methylene diphenyl diisocyanate (pMDI) adhesive with pre-evaporation treatment on the strands, resulted in board adhesive strength values that were deemed satisfactory. Applying a pre-evaporation treatment to the strand can enhance the dimensional stability of OSB made from sengon wood (Aisyah *et al*. 2021).

Although pMDI is highly effective as a high-performance adhesive for OSB production, it is also quite costly. Mixing it with PF could have reduced the overall production without compromising its properties. Therefore, in this study, the performance of OSB from sengon wood was evaluated using hybrid PF/pMDI adhesive systems comprising two types of catalysts, NaOH and $CaCO_3$. Basic properties, chemical properties, and thermo-mechanical properties of hybrid PF/pMDI adhesives were also investigated using several techniques.

EXPERIMENTAL

Materials

A 10-year-old sengon (*P. falcataria* L. Nielsen) tree with a diameter of 28 cm DBH was obtained from a plantation forest in Bogor, Indonesia. A PF adhesive with a solids content of 47.39% was acquired from Pamolite Adhesives Company, located in Probolinggo, Indonesia. The paraffin, distilled water, NaOH (technical grade, 20% w/v), and $CaCO_3$ (technical grade, 20% w/v) were acquired from a chemical store located in Cibinong, Indonesia. Anugerah Raya Kencana Company in Banten, Indonesia provided pMDI with a solids content of 99.47%.

Formulation and Characterization of Hybrid Adhesives

Hybrid PF/pMDI adhesives were created in four different formulations and compared to PF only as a control (Table 1). Two catalysts, NaOH (20% w/v) and $CaCO_3$ (20% w/v) solution were created by combining 200 g of technical grade NaOH and technical grade $CaCO_3$, respectively, with 1000 mL of distilled water. The combination of adhesive and catalyst, together with the pMDI, is mixed based on the solid content of PF adhesive. The catalyst and pMDI were added according to the formulation provided in Table 1.

Table 1. Hybrid PF/pMDI Adhesive in Different Formulations

Type of Hardener	PF resin (g)	NaOH 20% (g)	$CaCO_3$ 20 % (g)	pMDI (g)
Control	300	-	-	-
NaOH-2.5 (N-2.5)	300	7.11	-	3.57
NaOH-5.0 (N-5.0)	300	7.11	-	7.15
$CaCO_3$ -2.5 (C-2.5)	300	-	7.11	3.57
$CaCO_3$ -5.0 (C-5.0)	300	-	7.11	7.15

The fundamental characteristics of hybrid PF/pMDI adhesives, including solids content (SC), viscosity, pH, and gelation time, were assessed based on prior research (Fitrianum *et al.* 2023). The solids content was determined by desiccating 1 g of the sample in an oven at 105 °C for 3 h, and thereafter dividing the weight of the dried sample by its original weight. The mean viscosity of hybrid PF/pMDI adhesives was measured using a rotational rheometer (RheolabQC, AntonPaar, Austria) with a spindle no. 27 at a temperature of 25 ± 2 °C and a consistent shear rate of 100 s^{-1} for a duration of 120 s. The gelation time of hybrid PF/pMDI adhesives was determined by subjecting them to boiling water at a rotational speed of 10 rpm, using a gelation time meter (GT-6, Techne Inc., USA). The pH of hybrid PF/pMDI adhesives was measured at a temperature of 25 ± 2 °C using a digital pH meter (OrionStar A111, ThermoScientific, USA).

The change in functional groups of liquid and cured hybrid PF/pMDI adhesives was investigated using Fourier transform infrared (FTIR) spectroscopy. The SpectrumTwo instrument from Perkin Elmer Inc., Waltham, MA, USA, was utilized for this purpose. The samples were analyzed at a temperature of 25 ± 2 °C using the Attenuated Total Reflection

(ATR) technique throughout a range of 400 to 4000 cm^{-1}, with a resolution of 4 cm^{-1} for each sample. Each formula was examined with three repetitions.

The rheological characteristics of hybrid PF/pMDI adhesives were examined using a rotational rheometer (RheolabQC, AntonPaar, Austria). The adhesive's dynamic viscosity, cohesion strength, and relaxation modulus were assessed within the 30 to 100 °C temperature range. The measurements were conducted at a constant shear rate of 200 s^{-1} and a heating rate of 5 °C min^{-1}.

The thermo-mechanical characteristics of hybrid PF/pMDI adhesives were investigated using Dynamic Mechanical Analysis (DMA 8000, Perkin Elmer Inc., USA). Each adhesive was applied to adhere two Whatman filter papers, with a glue coating weight of 180 grams per square meter, to create a sample of 50 millimetres in length, 8 millimetres in width, and 0.2 millimetres in thickness. Before the DMA analysis, all specimens were subjected to pre-curing in an oven at a temperature of 50 °C for 5 min. The storage modulus (E'), loss modulus (E''), and tan delta of each specimen were measured at a frequency of 1 Hz, strain level of 0.01%, and heating rate of 1, 3, and 5 °C min^{-1} in the temperature range of 30 to 200 °C using the dual cantilever mode.

Pre-treatment and Characterization of Strands

The sengon logs, which had a specific gravity of 0.38, were transformed into strands using a disk flaker with a target size of 70 mm × 25 mm × 0.5 mm. The sengon wood has an initial moisture content of 12.42% and an oven-dried density of 405 ± 42 kg m^{-3}. As displayed in Fig. 1, the sengon wood strands underwent steam treatment in an autoclave at a temperature of 126 °C and a pressure of 0.14 MPa for 1 h. The strands were left to dry in the air for approximately 5 days and then placed in an oven for approximately 14 days until the moisture content reached below 5%. The dimensions of the strand (length, width, thickness), as well as the slenderness ratio (SR) and aspect ratio (AR) for each treatment, were determined by measuring 100 oven-dried strand samples.

Fig. 1. Flowchart of sengon wood strands preparation

Manufacturing of OSB

As depicted in Fig. 2, 250 mm × 250 mm × 12 mm OSB panels were produced using different hybrid PF/pMDI adhesive formulations, specifically N-2.5, N-5.0, C-2.5, and C-5.0. Panels using unmodified PF were also made as controls. A wax content of approximately 1% was incorporated into the hybrid PF/pMDI, according to the weight of the strands. Subsequently, sengon wood strands were mixed with a hybrid PF/pMDI at a concentration of 10% using a rotary drum mixer and spray gun. The strand mats were subsequently created using a shelling ratio 1:2:1 (Maulana *et al.* 2019). The mats underwent a hot-pressing process at a temperature of 150 °C, with a precise pressure of 2.45 MPa applied. The single-stage pressing cycle was 9 min counted from the target board thickness or stopping position reached. The panels were conditioned at a temperature range of 25 to 30 °C and a relative humidity range of 60 to 65% for approximately 14 days until they achieved a state of equilibrium moisture content. A total of 15 panels were produced for three panels per adhesive formula.

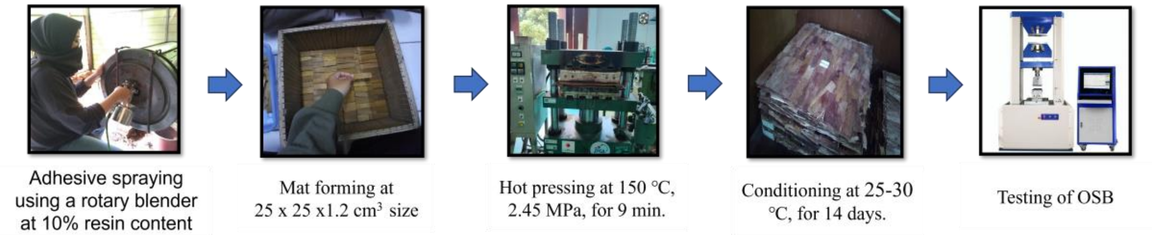

Fig. 2. Preparation of OSB bonded with hybrid PF/pMDI adhesives

Evaluation of OSB Properties

The physical and mechanical characteristics of the OSB were assessed following the JIS A 5908 standards (JSA 2003). These included density, moisture content (MC), water absorption (WA) in 24-hour immersion, thickness swelling (TS) in 24-hour immersion, modulus of elasticity (MOE), modulus of rupture (MOR), and internal bonding (IB). Three samples per adhesive formula were used for each test. The acquired values were subsequently compared to the CSA 0437.0 standard (Grade O-1) for commercial OSB panels (CSA 2011).

Statistical Analysis

Statistical analysis was conducted on the performance of OSB utilizing a completely randomized design, considering the catalyst type and the level of pMDI added to the hybrid PF/pMDI adhesives. Each treatment was replicated three times. Data was processed using Microsoft Excel 2019 software and IBM SPSS Statistics 20. The data analysis was conducted using analysis of variance (ANOVA) at a confidence level of 95%. Duncan's multiple-range test (DMRT) was conducted on parameters that had a substantial impact to ascertain the notable disparity among treatments.

RESULTS AND DISCUSSION

Basic Properties of Hybrid PF/pMDI Adhesives

Table 2 presents the average SC values from the control PF and hybrid PF/pMDI adhesives at different catalysts and levels of pMDI. The average SC of the control PF adhesive was 46.0%, while the hybrid PF/pMDI had an average SC of around 46.8 to 47.6%. Adding a catalyst can enhance the adhesive's shear strength compared to uncatalyzed PF adhesives. The obtained SC value in this study exceeded the current standard range of 40 to 45% (SNI 1998). This is supported by research by Fitrianum *et al.* (2023), which states that the type of catalyst influences the SC value. The ANOVA and DMRT test revealed that only the catalyst type significantly influences the SC values. At the same time, the pMDI content and the interaction between factors did not provide a significant effect. The acidity factor is crucial in the bonding process between wood and adhesive, particularly when the bonded material contains a significant amount of extractive materials. The ANOVA and DMRT test results showed that adding the NaOH catalyst had a more significant effect than the adhesive with the $CaCO_3$ catalyst. This happens because NaOH has stronger basic properties and is very reactive compared to $CaCO_3$ (Amin 2019), making it possible for adhesives with a NaOH catalyst to harden more quickly, and the particle content to be greater is related to the condensation reaction in the adhesive. The SC denotes the amount of solid present in the adhesive, and this value generally correlates with the viscosity, specific gravity, and gelation time. Increased SC leads to elevated adhesive viscosity and specific gravity, resulting in extended gelation time. A higher concentration of resin in the copolymer indicates an elevated number of molecules, which is believed to contribute to the adhesive's intermediate interaction with the adherent. Adhesives that include a high concentration of solids exhibit strong adhesive characteristics and can form optimal bonds, leading to satisfactory adherence (Fitrianum *et al*. 2023).

The results of the gelation time of the hybrid PF/pMDI adhesive can be seen in Table 2. The control PF exhibited the lengthiest gelation period compared to the hybrid PF/pMDI when a catalyst was introduced, specifically lasting 14.2 min. Including the NaOH catalyst accelerated the gelation process, reducing the time required from 8.87 to 9.83 min. In contrast, adding the $CaCO_3$ catalyst resulted in a longer gelation time of 10.7 to 12.5 min. The adhesive's fast gelation time indicates that it does not require an extended period to mature while subjected to hot pressing in producing composite products. Nevertheless, the adhesive's rapid gelation time results in a swift coagulation process, thereby reducing the adhesive's shelf life (Santoso *et al*. 2019). The ANOVA test showed that the catalyst type factor significantly affected the gelation time produced. In contrast, the pMDI concentration and the interaction between the two factors gave insignificant results. Based on DMRT tests, the hybrid PF/pMDI gelation time with NaOH catalyst was more significant than with $CaCO_3$ catalyst and control. The results of this test show that the type of catalyst significantly influences the gelation time produced. This shows that the treated adhesive's gelation time is shorter than the control PF adhesive. SNI 06-4567 states that the gelation time for PF storage is around 30 to 60 min at a temperature of 25 °C (SNI 1998). This is also related to the value of the SC produced, where the value will be directly proportional. The higher the solids content of an adhesive, the longer it takes for the adhesive to change form to gel (Fitrianum *et al*. 2023). Nevertheless, this investigation showed that adhesives with increased SC exhibited a reduced gelation time compared to other treatments. The presence of NaOH, a highly reactive substance, can accelerate the polymerization process and reduce the gelatinization time of adhesives. NaOH functions as a catalyst, enhancing the speed of chemical processes.

Table 2. Basic Properties of PF and Hybrid PF/pMDI Adhesives

Catalyst Type	**Adhesive Characteristics**					
	Solids Content (%)	**Gelation Time (min)**	**Average viscosity (mPa·s)**		**pH**	
			pMDI 2.5%	**pMDI 5.0%**	**pMDI 2.5%**	**pMDI 5.0%**
Control	46.00^{a*}	14.17^{a}	239.48^{b}		12.40^{a}	
NaOH	47.37^{b}	9.35^{b}	250.24^{a}	219.71^{c}	12.35^{b}	12.34^{b}
$CaCO_3$	46.76^{c}	11.62^{c}	218.91^{c}	206.63^{d}	12.12^{d}	12.27^{c}

* Values with different letters are significantly different.

To ensure the stability of the liquid adhesive during storage, it is necessary to preserve the hybrid adhesive under alkaline conditions, which will sustain the curing reaction of the adhesive for an extended period (Santoso *et al*. 2019). The control PF adhesive had the highest pH value compared to the other adhesives, while the lowest was the C2.5 adhesive (Table 2). The addition of $CaCO_3$ causes a decrease in the pH value due to the inherent alkaline nature of this catalyst, which has a pH of 9.91. When combined with PF, it will lower the final pH value. The pH measurements of adhesives subjected to different treatments indicate that the adhesive exhibits alkaline characteristics. The results obtained are based on the standards specified in SNI 06-4567-1998, ranging from approximately 10 to 13 (SNI 1998). Furthermore, the ANOVA test showed that the interaction between catalyst type and pMDI content significantly affects the pH value. DMRT test results revealed that the hybrid PF/pMDI adhesives formulated with different catalysts and pMDI concentrations had significantly different pH values. The hybrid PF/pMDI adhesive must be maintained in alkaline conditions to maintain the adhesive's curing reaction/copolymerization reaction. Thus, the liquid adhesive is relatively stable during storage (Santoso *et al*. 2019).

The viscosity of the adhesive affects its capacity to permeate the wood pores and its shelf life. Maloney (1993) states that adhesives with a high concentration of particles and the appropriate viscosity can create optimal connections, leading to strong adhesion. Table 2 displays the viscosity values of the hybrid PF/pMDI adhesives with different formulations at 25 °C, commonly called room temperature. In addition, identification of the effect of additional temperature was also carried out on the viscosity, cohesive strength, and relaxation modulus on the same adhesive samples (Fig. 1). The ANOVA showed that the interaction between the factors of catalyst type and pMDI content has a significant influence on the viscosity and cohesive strength of the adhesive. The findings of the DMRT test demonstrated that the viscosities of the hybrid PF/pMDI adhesives produced with various catalyst types and pMDI concentrations differed noticeably. Using 2.5% NaOH and pMDI catalyst (N-2.5), the hybrid PF/pMDI adhesive with the highest viscosity was created. It was followed by the Control treatment, N-5.0, C-2.5, and C-5.0 (Table 2).

The N-2.5 adhesive formula has the highest viscosity and cohesive strength values, so it hardens more quickly than other types of adhesives (Fig. 3). This can occur due to the addition of a NaOH catalyst, which is very reactive (Amin 2019) and can increase the number of particles contained in the adhesive, as well as the reaction of the PF adhesive with pMDI which influences the viscosity of the adhesive. A high viscosity value can affect the shelf life of the adhesive, where the higher the value, the shorter the shelf life (Shi and Gardner 2001). The standard optimum viscosity value of PF adhesive required by SNI 06-4567 is between 130 and 300 mPa·s (SNI 1998). Test results show that PF and hybrid PF/pMDI adhesives at room temperature (25 °C) have adhesive viscosity values ranging from 207 to 250 mPa·s. This value shows that all adhesive formulas have viscosity values that comply with SNI 06-4567 standards (SNI 1998).

As depicted in Fig. 3, adding a catalyst can reduce the viscosity, cohesive strength, and relaxation modulus of the hybrid PF/pMDI adhesive as a function of temperature (Hong *et al.* 2018). This can happen because the catalyst had a solution concentration of 20%; thus, more hydroxyl groups will be contained in the adhesive when mixing. Adding pMDI provides lower viscosity at room temperature and with additional temperature. The higher the percentage of pMDI content added, the lower the viscosity and cohesive strength produced. The lower viscosity of pMDI can cause this phenomenon compared to PF adhesive, which is around 200 to 212 mPa.s. The viscosity value decreases with increasing temperature, indicating that the ability of the adhesive to distribute on the strand will become lower when the temperature increases. The value of cohesion strength decreases with increasing temperature. Low cohesive strength will facilitate adhesive distribution on the wood strands' surface during the board compression (Aisyah *et al*. 2021). The decrease in cohesive strength value occurs due to damage to the polymer chain bonds at higher temperatures, randomly shaped to reduce interactions between molecular chains. In addition, when the temperature was increased, the relaxation modulus strength value decreased significantly with the same trend for each interaction factor in the adhesive. Relaxation modulus is used to assess stress relaxation as a function of temperature. The higher the temperature, the lower the relaxation modulus value.

The relaxation modulus quantifies the stress relaxation characteristics of a substance by describing the decay of its stress response over time while maintaining a constant strain. An analysis was conducted on the relaxation modulus of the hybrid PF/pMDI adhesive at different temperatures: 25, 50, and 75 °C (Fig. 3c). This analysis aimed to investigate the impact of temperature elevation on the relaxation modulus of the adhesive. Additionally, it provided insights into determining the level of adhesive curing rates and the treatment limits that can be applied. The relaxation modulus value exhibited a negative correlation with rising temperature. Based on data analysis, it has been determined that the PF-C adhesive exhibits the highest relaxation modulus value at a temperature of 25 °C. It is followed by the N-2.5 adhesive sample, C-2.5, N-5.0, and C-5.0.

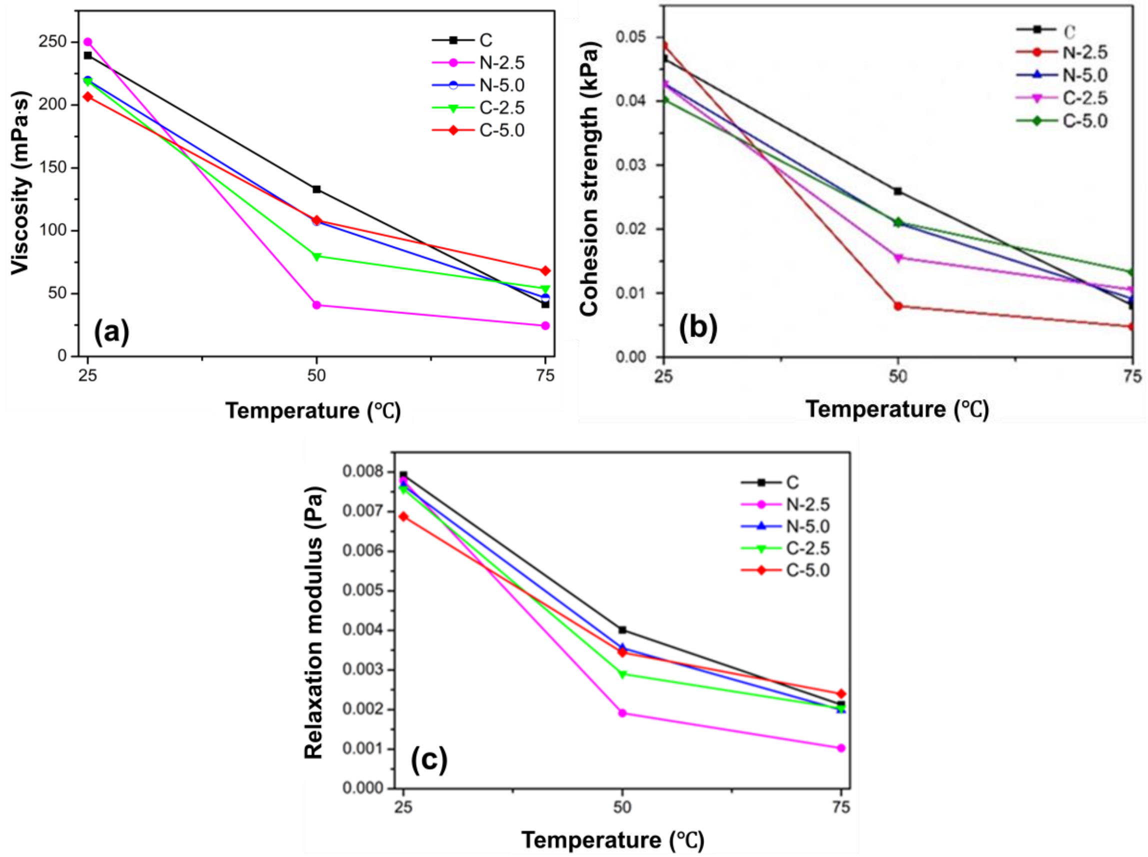

Fig. 3. Viscosity (a), cohesion strength (b), and relaxation modulus (c) of hybrid PF/pMDI adhesive at different formulations and temperatures

FTIR and XRD Analysis of Hybrid PF/pMDI Adhesives

Using an FTIR instrument, functional group analysis was performed on liquid and cured hybrid PF/pMDI adhesive in various formulations (Fig. 4). The presence of O–H group in the hybrid adhesive is indicated by a wavenumber of 3600 to 3200 cm^{-1} (Fig. 4a). The phenolic hydroxyl absorption peak is situated at 3300 cm^{-1}. The C=C stretching vibration of the benzene aromatics in PF and pMDI is responsible for the significant absorption peak observed between 1460 and 1600 cm^{-1}. Furthermore, the vibration of the C–O group can be attributed to the absorption peak at 1000 to 1033 cm^{-1}. There was no discernible variation in intensity between the hybrid PF/pMDI adhesive treatments. The presence of the CH_2 group from the CH_2OH molecule, which results from a reaction between formalin and phenol, is indicated by wavenumbers between 1340 and 1470 cm^{-1}. When comparing analysis utilizing liquid samples to those using solid materials, there is a difference in wave number intensity. A peak in the 3600 to 3200 cm^{-1} range suggested that the hybrid PF/pMDI adhesive contained O–H groups. As shown in Fig. 4b, the absorption intensity is lower, indicating the O–H groups are not detected due to the cured adhesive sample. The C–H group at the wavenumber 2922 cm^{-1} also appears from the CH_2 in the cured adhesives. Another peak at 1435 cm^{-1} can be caused by CH_2 bending vibrations, which indicate the presence of methylene groups in the adhesive due to the reaction between phenol and formalin.

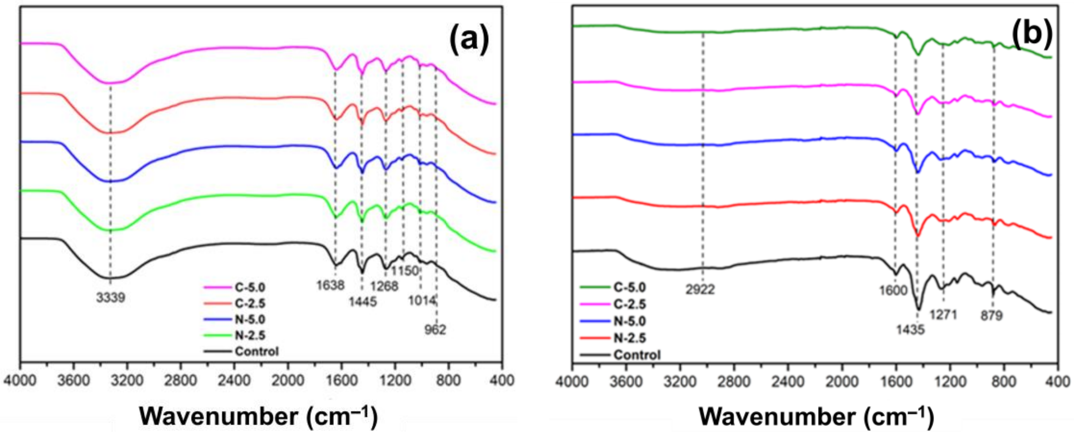

Fig. 4. FTIR spectra of the hybrid PF/pMDI adhesive. (a) Liquid adhesive, (b) solid adhesive

The XRD analysis of the hybrid PF/pMDI adhesive in various formulations can be seen in Fig. 5. The control PF (C) had one peak at the 2θ of 20.8°, the hybrid PF/pMDI with N-2.5 had some peaks at the 2θ of 20.5 to 35°, the hybrid N-5.0 had several peaks at 2θ of 22.34 to 40°, the hybrid C-2.5 with some peaks at the 2θ of 19.8 to 35°, and the hybrid C-5.0 with the highest peak at the 2θ of 20.17 to 45°. The respective peak angles characterize the crystalline form of each adhesive. The hybrid PF/pMDI adhesives combined with various catalysts had more crystalline structures than the control PF (C), which was dominated by an amorphous structure. Further identification of the degree of crystallinity (DOC) of the hybrid PF/pMDI adhesives at different formulas showed that the DOC of the adhesive with the addition of catalyst and pMDI increased compared to the PF control. The addition of the $CaCO_3$ catalyst provided a higher increase in DOC compared to the NaOH catalyst. Adding 5.0% pMDI content resulted in higher DOC than adding 2.5% pMDI content. This is explained by the nature of pMDI, which can cross-link with

PF *via* a urethane bond and increase the cohesive strength of the hybrid adhesive. The hybrid C-5.0 formula had the highest DOC of 59.3%, while the control C formula had the lowest DOC of 38.4%. Due to the strong inter-chain forces and high-order chains that predominate in the polymer structure, a crystalline area can be formed by the chains or parts of the chains approaching each other in a parallel manner. The high DOC shows this. Hydrogen bonds between chains create an attraction. This is brought on by variations in each polymer's molecule's length and irregularity. Irregularities in the chain structure, such as branch structures, will prevent the chains from approaching each other, limiting the crystallization process. However, when it comes to copolymers used in wood adhesive applications, elasticity is primarily needed rather than stiffness or strength. In this scenario, the presence of branch structures is necessary to impede or restrict the movement of the chains, preventing them from coming into proximity. This is done with the expectation that the adhesive will retain its original properties and not become brittle upon curing.

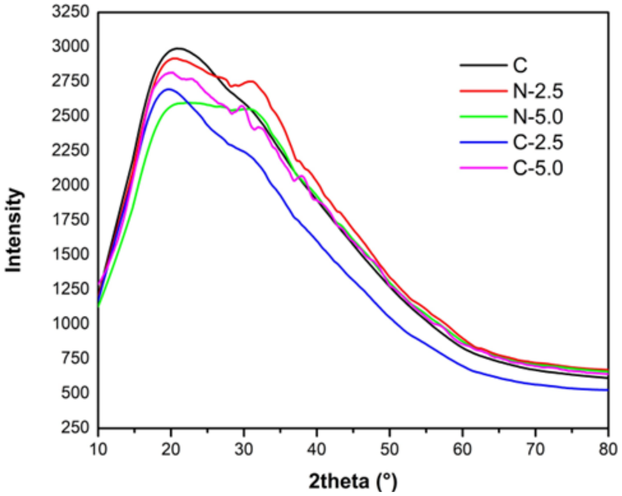

Fig. 5. XRD patterns of hybrid PF/pMDI adhesives at different formulations

Thermo-Mechanical Properties of Hybrid PF/pMDI Adhesives

The results of the DMA of control PF and hybrid PF/pMDI adhesives with the addition of catalysts on two types of catalysts and different pMDI levels are shown in the following graph in Fig. 6. DMA provides information related to storage modulus (E'), loss modulus (E''), and tan delta, namely data related to the thermomechanical and viscoelastic properties of adhesives. The tan delta peak represents the midpoint transition or inflexion point of the decline in the log curve E', while the loss modulus peak shows the initial drop of E' in the glassy state towards the transition stage. The E'' peak at the temperature of glass (T_g) generally indicates the intersection of two tangential curves on the log E' obtained from the glassy region and the transition called the onset temperature (Menczel and Prime 2009). The DMA results of the hybrid PF/pMDI adhesives in various formulations show different viscoelastic properties with different catalysts and pMDI levels. However, it is difficult to analyze the accurate temperature at which curing occurs in the hybrid PF/pMDI adhesives. This is also supported by the nature of thermoset polymers, which do not have a clear T_g; such clear T_g results are expected in the case of thermoplastic polymers. The increase of T_g value does not influence the strength and hardness of thermosets in temperature and deformation rate. The combination analysis of E', E'', and tan delta was able to explain the hardening properties of the hybrid PF/pMDI adhesives to a greater extent than using only the *E′* parameter and the tan delta curve (Lei and Frazier 2015). This is because the increase in temperature only provides a small change in the resulting *E′* value, so it is quite challenging to determine the onset temperature accurately on the adhesives tested.

Storage modulus (*E′*) shows the elasticity value of the material in the form of the material's ability to store energy. Measuring the adhesive's *E′* value indicates the adhesive’s ability to resist deformation. The *E′* values of the five treatments decreased with increasing temperature (Fig. 6a). The *E′* value declined significantly during heating from 30 to 120 °C, due to the softening of the adhesive in this region (Hong *et al*. 2018). Shen *et al*. (2021) state that the softening temperature is achieved at 80 to 120 °C (Shen *et al*. 2021). After softening, then *E′* decreases gradually between 120 and 200 °C, and another significant decrease occurs at a temperature of 200 to 300 °C related to the adhesive phase transition, while at a temperature of 265 to 300 °C a carbonization process occurs in the adhesive. The *E′* value indicates the stiffness properties of the adhesive, as can be seen at temperatures below 200 °C; higher *E′* values and more glassy structure are obtained compared to the properties produced at temperatures above 200 °C. A low *E*' value correlates with low mechanical properties of the adhesive when applied to the board.

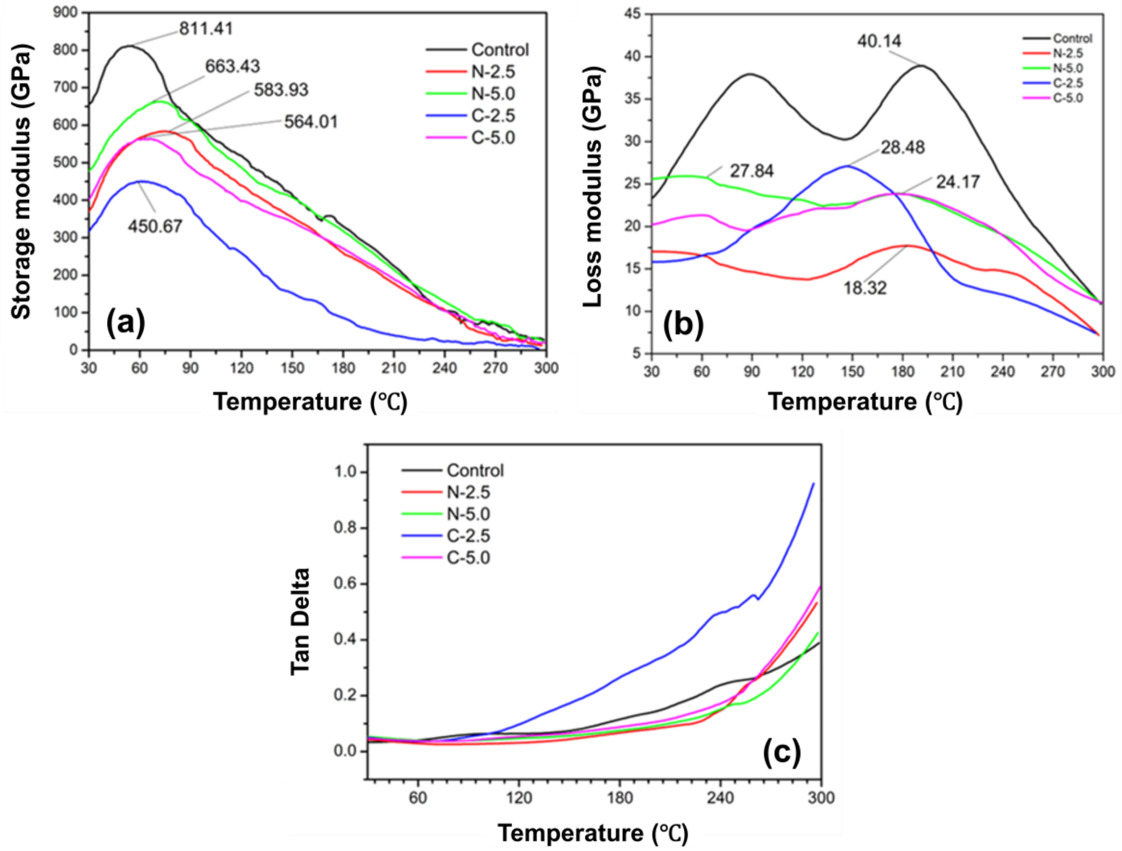

Fig. 6. DMA thermograms of hybrid PF/pMDI adhesives at different catalyst formulations. Storage modulus (a), loss modulus (b), and tan delta (c)

Loss modulus (*E″*) indicates the material’s ability to dissipate energy. Measuring the *E″* value of an adhesive indicates the ability of the adhesive to emit energy as heat. The *E″* values of the hybrid PF/pMDI at five formulations gave varying results at different temperatures (Fig. 6b). The DMA thermogram showed that control PF adhesive releases the most energy as temperature increases. This indicates that the control adhesive has the highest *E″* value compared to the hybrid PF/pMDI adhesives at different catalysts. The hybrid adhesive produces two clear peaks and requires higher temperatures (70 and 190 °C) to release the energy stored in the adhesive. In contrast, adhesive with catalyst and pMDI can accelerate the energy release rate in the adhesive, namely at temperatures around 60 to 180 °C. Adding NaOH and $CaCO_3$ as catalysts and pMDI as a cross-linker agent can speed up the hybrid adhesive maturation process.

Tan delta shows the balance between the elastic and viscous phases of the polymeric materials (Hassan *et al*. 2011). The tan delta (damping ability) of the hybrid PF/pMDI adhesives with various formulations is shown in Fig. 6c. Kamarudin *et al*. (2020) stated that an elastic phase occurs in the adhesive if the tan delta is close to 0, while a viscous phase occurs when the tan delta value approaches 1 (Kamarudin *et al*. 2020). The peak of the tan delta value for each treatment appeared at a temperature of 240 to 260 °C. This is also supported by previous research using differential thermal analysis (DTA), which stated that there was a change in the melting phase transition temperature due to the addition of reactants, so a new compound with different properties was produced (Santoso et al., 2019). The standard melt phase transition temperature for PF adhesives is approximately 260 °C.

Characteristics of Sengon Wood Strand

Strand geometry is one of the most critical factors in improving the properties of OSB because it can provide a wider actual contact area and better stress transfer. OSB properties directly influenced by strand geometry include mechanical properties, board surface characteristics, response to water vapour, and machining properties such as ease of sawing. Table 3 shows the geometric distribution characteristics of sengon wood strands. Strand geometry is one of the factors that affect the properties of OSB. The mean AR value of the strands was 2.82, whereas the SR values were 113. The contact area determines the SR value of the strand, the mechanical properties of the board, and the amount of adhesive used. As the SR value increases, the contact area between strands improves, enhancing the board's mechanical properties (Iswanto *et al*. 2010a). Previous research showed that strands with AR values exceeding 3 and SR exceeding 100 produced good mechanical properties in OSB using PF. The strand geometry in this study is sufficient to produce OSB with good properties. An aspect ratio of 2 is sufficient to produce good OSB properties (Kuklewski *et al*. 1985; Maulana *et al*. 2023). AR value of more than 1 facilitates orientation during mat formation (Maloney 1993). The SR value of the strand is related to the panel’s contact area, the panel's mechanical properties, and the adhesive consumption. Higher SR values increase the contact area between the strands and the mechanical properties of the board (Maloney 1993; Iswanto *et al*. 2010a).

Table 3. Geometry of Sengon Strands for Manufacturing OSB Panels

Parameters	Maximum	Minimum	Average	Standard deviation
Length (cm)	8.38	6.51	6.81	0.17
Width (cm)	3.10	2.09	2.42	0.14
Thickness (cm)	0.11	0.03	0.06	0.02
SR	175.87	60.55	112.83	26.37
AR	3.53	2.20	2.82	0.17

Physical Properties of OSB bonded with Hybrid PF/pMDI adhesives

The average density value of the OSB ranged from 0.55 to 0.64 g.cm^{-3} (Table 4) and met the target density of 0.60 g.cm^{-3}. The ANOVA results showed that only pMDI levels significantly influenced the density values. DMRT results revealed that the density of OSB with adhesive using 5.0% pMDI was the highest and was significantly different from others, followed by the control PF adhesive, and the lowest density was the hybrid adhesive using 2.5% pMDI. This can occur due to the good adhesive strength of pMDI. pMDI can form a solid bond between the substrate and adhesive, thereby improving the physical and mechanical properties of the board, one of which is the resulting density. Higher pMDI content in the hybrid adhesives resulted in a higher density of OSB. Table 4 also shows that the hybrid PF/pMDI adhesive formulas could meet the target density of 0.6 g.cm^{-3}, except for the hybrid PF/pMDI with C-2.5. This is probably due to uneven strand distribution in the OSB manufacturing process. However, the density of OSB bonded with hybrid PF/pMDI adhesives at various formulas provided values that align with the desired targets.

Table 4. Density (g.cm^{-3}) of OSBs with Different Levels of pMDI and Types of Catalyst

Type of Catalysts	**Level of pMDI**			
	0%	**2.5%**	**5.0%**	**Average**
Control *	0.62±0.03	-	-	0.62±0.03
NaOH	-	0.60±0.01	0.64±0.02	0.62±0.02
$CaCO_3$	-	0.55±0.05	0.62±0.05	0.59±0.05
Average	0.62±0.03a	0.58±0.03b	0.63±.03c	

* Control = Commercial PF without the addition of catalyst and pMDI

The MC of OSB obtained from this research was homogeneous, namely around 7.15 to 7.45% (Table 5). The homogeneous MC value was attributed to the board being conditioned at the same temperature and time, namely at room temperature. Sengon wood OSB MC value meets JIS A 5908 standards, which requires MC of OSB in the 5 to 13% (JSA 2003). The results of ANOVA showed that the type of catalyst and pMDI content, as well as the interaction between the two, did not affect OSB moisture content. This is because the water content of the sengon wood strands was relatively the same for all adhesives applied to OSB.

Table 5. Moisture Content (%) of OSBs with Different Levels of pMDI and Types of Catalyst

Type of Catalysts	**Level of pMDI**			
	0%	**2.5%**	**5.0%**	**Average**
Control *	7.23±0.33	-	-	7.23±0.33
NaOH	-	7.26±0.34	7.15±0.43	7.21±0.38
$CaCO_3$	-	7.45±0.36	7.15±0.23	7.31±0.30
Average	7.23±0.33	7.36±0.35	7.15±0.33	

* Control = Commercial PF without the addition of catalyst and pMDI

The WA value of OSB bonded with hybrid PF/pMDI adhesives ranged from 64.4 to 83.1% after 24-h soaking (Fig. 7). The ANOVA showed that the interaction between catalyst type and pMDI content had no significant effect on the WA of OSB. However, each factor had a significant and significant impact on the WA of OSB. DMRT results showed that adding the $CaCO_3$ catalyst type was significantly different from adding the NaOH catalyst and adding 5.0% pMDI was significantly different from adding 2.5% pMDI. This shows that adding pMDI to the PF adhesive can decrease the WA values of OSB due to the formation of a urethane bond between pMDI and PF adhesive (Lubis *et al*. 2019a). The $CaCO_3$ catalyst type provided a lower WA value than the NaOH catalyst. This might be because $CaCO_3$ decomposes into calcium ions and carbonate that are not highly hydrophilic. At the same time, NaOH produces sodium ions and hydroxyl ions that tend to attract water.

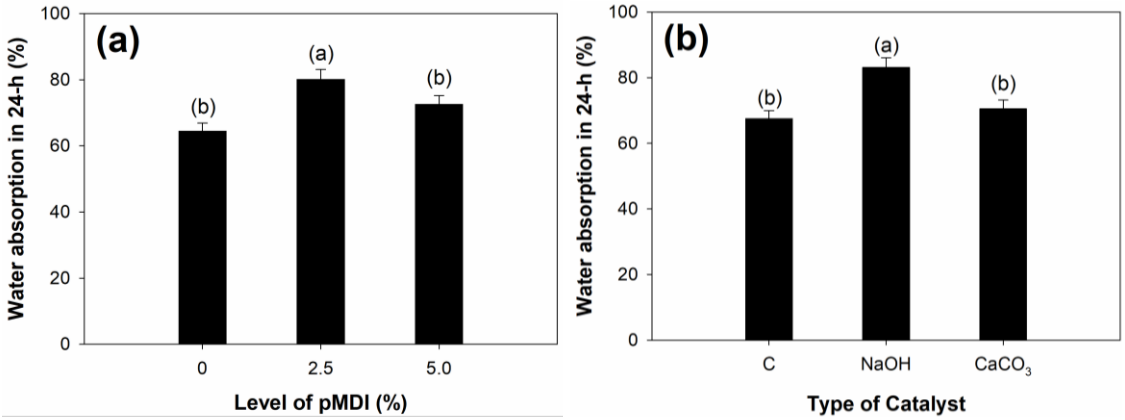

Fig. 7. Water absorption in 24 h of OSBs using PF with different levels of pMDI (a), and different types of catalyst (b)

The TS of OSB was 16.1 to 25.5% after 24 h of soaking (Table 6). As the soaking time increased, the TS value was directly proportional to the WA value. This occurs because the amount of water absorbed by the test sample increases, resulting in the expansion of cell walls and changes in sample dimensions. The adhesive system impacts hydrolytic stability in addition to the wood strands (Sari *et al*. 2024). Hybrid PF/pMDI adhesives with a $CaCO_3$ catalyst obtain significantly lower OSB's TS values.

Table 6. Thickness Swelling of OSB Bonded with Hybrid PF/pMDI Adhesives at Different Formulations

Thickness Swelling in 24 h (%)				
Type of Catalyst	**Level of pMDI**			
	0%	**2.5%**	**5.0%**	**Average**
Control *	20.58±3.64	-	-	20.58±3.64a
NaOH	-	22.39±3.11	25.52±3.63	23.96±3.37b
$CaCO_3$	-	16.13±3.92	19.41±2.66	17.77±3.29c
Average	20.58±3.64	19.26±3.52	22.47±3.15	

*Control = Commercial PF without the addition of catalyst and pMDI

The results of ANOVA showed that the catalyst type factor significantly influenced TS. In contrast, the pMDI content and the interaction between the two factors did not significantly influence the TS of OSB. The DMRT test showed that adding the $CaCO_3$

catalyst gave the lowest TS value and was significantly different from adding NaOH to the adhesive. This correlates with the DSA properties of OSB, which also showed that with the $CaCO_3$ catalyst, the value decreased, and the physical properties of the board increased. Adhesives with the addition of $CaCO_3$ catalyst provided higher TS values than adhesives with NaOH. This correlates with the properties of $CaCO_3$, which can increase the water absorption resistance of the board and improve the interfacial bond on the board, which prevents the formation of cavities in the board, thereby increasing the effectiveness of its adhesive strength. OSB with C-2.5 and C-5.0 adhesive were able to meet the standard PT requirements (soaking 2 h) for OSB according to CSO 0437.01 (Grade O-1) (CSA 2011).

Mechanical Properties of OSB bonded with Hybrid PF/pMDI Adhesives

The MOE perpendicular (⊥) to the grain value of OSB ranged from 1220 to 4120 MPa (Fig. 8). Except for OSB prepared with a C-5.0 adhesive formula, all adhesives used to make OSB achieved the standards required by CSA 0437.0 (*Grade* O-1) for MOE ⊥ to grain, namely 1300 MPa (CSA 2011). The analysis of variance showed that the interaction of the two factors had a real influence on the MOE value. Duncan's further tests showed that OSB using N-5.0 formula adhesive had the highest MOE ⊥ to grain value and was significantly different from OSB using other adhesives, followed by OSB with C-2.5 adhesive. OSB using C-5.0 adhesive had the lowest MOE ⊥ to grain value, but it was not significantly different from OSB using N-2.5 adhesive or control adhesive (C). Adding pMDI as a cross-linker in the adhesive provides optimal values for the N-5.0 and C-2.5 formulas. This was related to the nature of pMDI, which can form urethane bonds from the reaction between the -NCO group in pMDI with -OH groups in sengon wood (Lubis *et al.* 2019a), so that it can produce good adhesion bonds.

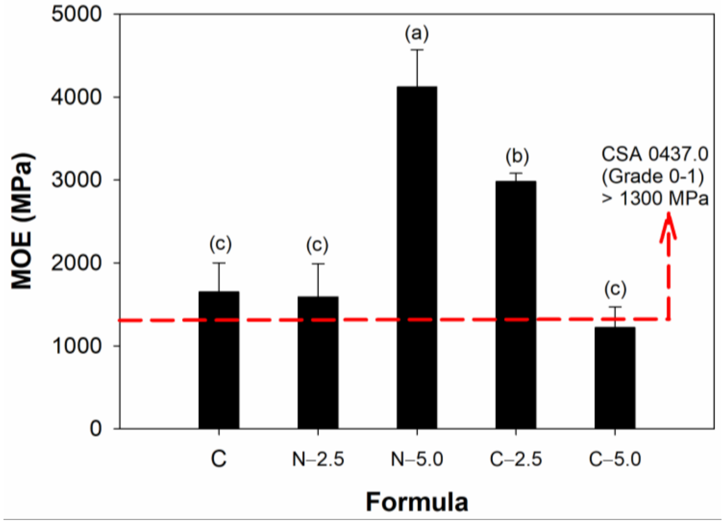

Fig. 8. MOE perpendicular (⊥) to the grain of OSBs bonded with control PF (C) and hybrid PF/pMDI at different formulations

The MOR ⊥ to grain value obtained ranged from 13.6 to 30.6 MPa (Fig. 9). The analysis of variance showed that the factors of catalyst type and pMDI content do not significantly influence the MOR ⊥ to grain value. In contrast, the interaction of these two types of factors had a significant effect on the MOR value. Duncan's further test results showed that OSB using N-5 adhesive had the highest MOR ⊥ to grain value but was relatively the same as OSB using C-2.5 adhesive. Both adhesives produced MOR values significantly different from those of OSB using other adhesives. C-5.0 adhesive provided the lowest MOR ⊥ to grain value for OSB but was not significantly different from OSB using control, N-2.5, and C-5.0 adhesives. Test results showed that all OSBs that use all adhesive formulas met the MOR ⊥ to grain standard required by CSA 0437.01 (Grade O-1), namely above 9.61 MPa (CSA 2011).

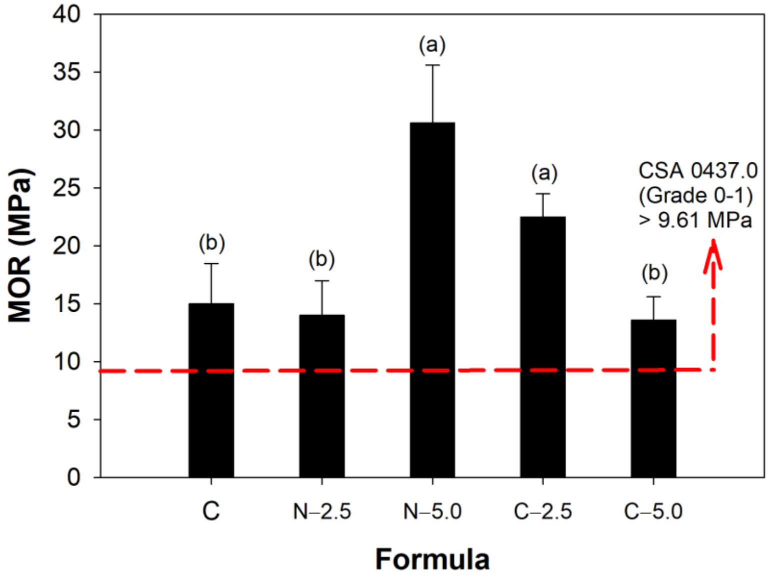

Fig. 9. MOR perpendicular (⊥) to the grain of OSBs bonded with control PF (C) and hybrid PF/pMDI at different formulations

OSB's internal bonding (IB) strength ranged from 0.25 to 0.46 MPa (Table 7). According to the CSA 0437.0 standard, the IB strength for OSB (Grade O-1) is over 0.35 MPa (CSA 2011). Therefore, only OSB bonded with hybrid PF/pMDI adhesive mixed with C-2.5 and C-5.0 was able to meet the minimum IB requirements, while other formulas did not. The results of ANOVA showed that the type of catalyst significantly affected the IB strength. In contrast, the pMDI level and the interaction between the two factors did not significantly affect the resulting IB strength. DMRT test showed that adding a catalyst increased the IB strength significantly. Using the $CaCO_3$ catalyst provided a higher IB strength of OSB compared to adhesives with the addition of NaOH.

Table 7. Internal Bonding (MPa) of OSBs with Hybrid PF/pMDI Based on Different Levels of pMDI and Types of Catalysts

Type of Catalyst	Level of pMDI			
	0%	2.5%	5.0%	Average
Control *	0.25±0.05	-	-	0.25±0.05a
NaOH	-	0.32±0.04	0.27±0.14	0.30±0.08b
$CaCO_3$	-	0.40±0.01	0.46±0.08	0.43±0.04c
Average	0.25±0.05	0.36±0.02	0.37±0.11	

*Control = Commercial PF without the addition of catalyst and pMDI

Statistical analysis showed that adding pMDI and catalyst increased OSB's physical and mechanical properties. This can occur due to the nature of pMDI, which can bond well with hydroxyl groups in wood through urethane bonds, thus producing good adhesion bonds, which is correlated with improving the mechanical properties of wood (Lubis *et al*. 2019b). Hybrid PF/pMDI adhesives with the addition of $CaCO_3$ catalyst provided higher IB values than adhesives with NaOH. This correlates with the properties of $CaCO_3$, which can increase the water absorption resistance of the board and improve the interfacial bond on the board, which prevents the formation of cavities in the board, thereby increasing the effectiveness of its adhesive strength (Laksana and Waluyo 2021). Therefore, the choice of catalyst and adhesive greatly influences the resulting adhesive properties (Berdnikova *et al*. 2021).

The performance assessment of OSB bonded with control PF and hybrid PF/pMDI adhesives showed the most optimal adhesive formula and can be recommended for future use (Table 8). The type of adhesive formula with the highest total assessment showed the best characteristics compared to other types of formula. The highest rating was obtained for the C-2.5 adhesive formula type, namely 23 points. This shows that the PF formula with the addition of $CaCO_3$ catalyst and 2.5% pMDI content provided the most optimal characteristics compared to other formulations when applied to OSB.

Table 8. Determination of Optimum Hybrid PF/pMDI Adhesive Formula for OSB Made of Sengon Wood Strand

Parameters	**Formulas**				
	C	**N-2.5**	**N-5.0**	**C-2.5**	**C-5.0**
Density	3	2	3	1	2
Moisture content	2	2	3	3	1
Water absorption	3	1	2	2	3
Thickness swelling	2	1	1	3	3
MOE	1	1	3	3	1
MOR	1	1	3	2	1
Internal bonding	1	2	1	3	3
Total	20	18	22	23	19

*Mark 1 = bad, 2 = medium, and 3 = good

CONCLUSIONS

1. This study developed a hybrid phenol-formaldehyde/poly(methylenediphenyl-diisocyanate) (PF/pMDI) adhesive system to produce oriented strand board (OSB) by utilizing various catalysts and different levels of pMDI.
2. It was found that the choice of catalyst and concentration of pMDI can enhance the hybrid adhesive's solids content, accelerate gelatinization, and elevate its viscosity.
3. The addition of catalyst and pMDI was observed to decrease OSB's physical properties, including moisture content (MC), water absorption (WA), and thickness swelling (TS). A $CaCO_3$ catalyst proved highly effective in reducing OSB's WA and TS characteristics.

4. The inclusion of catalysts and pMDI in the hybrid adhesives increased the average mechanical properties. Hybrid PF/pMDI formulations can enhance adhesive properties by incorporating the NaOH catalyst. In OSB applications, using hybrid PF/pMDI formulations that include $CaCO_3$ improves physical and mechanical properties.
5. Based on the solids content of the PF resin, the optimal performance of OSB panels was achieved with the adhesive system comprising 20% (w/v) $CaCO_3$ at 1% and 2.5% pMDI.

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