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Effectiveness of Nanocatalyst in the Improvement of Sorghum Bagasse Particleboard Bonded with Bio-Adhesive

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ABSTRACT

Modification in the manufacturing process of bio-adhesive need to be carried out to improve the properties of sorghum bagasse particleboard, such as adding nanocatalyst into the bio-based adhesive. This study was conducted to determine the effectiveness of nanocatalyst in enhancing the properties of sorghum bagasse particleboard. Each nanocatalyst, such as zinc oxide (ZnO) and graphene oxide (GO), has been added to the citric acid and molasses-based adhesives. The target density of the particleboard was 0.8 g/cm³, with a board size of 350 mm × 350 mm × 6 mm. The particleboard was pressed using a hot-pressing machine at a temperature of 200°C and 10 MPa of pressure for 10 min. The physical and mechanical properties of the particleboard were evaluated, referring to JIS A 5908:2022 standard. The result showed that adding GO into the citric acid-based adhesive obtained a higher modulus of rupture (MOR) and lower thickness swelling (TS) than the others. Consequently, adding GO is more effective than adding ZnO into the bio-based adhesive in enhancing the properties of the particleboard.

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1. Introduction

Regarding the tremendous destruction of trees and environmental pollutants, it is critical to produce an ecologically benign and sustainable composite material (Dušek et al. 2021). Biomass as an alternative raw material for composite goods has received considerable attention due to the decline in wood forest products caused by deforestation (Sutiawan et al. 2022). Biomass made from natural fibers has advantages because it is renewable, biodegradable, inexpensive, and readily available (Wang and Hu 2016). Sorghum bagasse is a biomass that has been effectively used as a raw material in particleboard (Iswanto et al. 2017; Kusumah et al. 2016; Sutiawan et al. 2020; Syahfitri et al. 2022; Tampubolon et al. 2022). According to Fatriasari et al. (2015), sorghum bagasse is a by-product of the sweet sorghum stem sap extraction method, and 30% of the total fresh weight of the extracted sorghum plant is a residue comprising carbohydrate polymers and lignin. Sorghum plantations cover 40.81 million ha globally (Syahfitri et al. 2022) and generate up

to 60 tons of cellulose per ha per year (Iswanto et al. 2017). Furthermore, this biomass can potentially be used as a particleboard raw material.

Iswanto et al. (2014, 2017) used formaldehyde and petroleum-based synthetic adhesives to make sorghum bagasse particleboard. However, the poor dimensional stability of particleboard-bonded synthetic adhesives with common adhesives was obtained. In addition, a synthetic adhesive can impact human health. As a result, bio-based adhesives such as citric acid (Kusumah et al. 2017b; Kusumah et al. 2016) and sucrose (Kusumah et al. 2017a, 2020) have been developed for use in sorghum bagasse particleboard.

The development of bio-based adhesive sorghum bagasse particleboard started with using citric acid. Based on previous research, it is known that citric acid adhesive sorghum bagasse particleboard has good dimensional stability. At the same time, its modulus of elasticity and internal bond values are comparable to particleboard using PF adhesive. Despite these strengths, the modulus of rupture value is still lower than that of PF adhesive particleboard (Kusumah et al. 2016). To overcome this problem, sucrose is added to citric adhesive sorghum bagasse particleboard. Adding sucrose to citric acid adhesive improved the mechanical properties (Kusumah et al. 2017a). However, the sucrose currently used is still synthetic-based, with high raw material costs. Therefore, it is necessary to develop the utilization of other saccharides, such as molasses (Syahfitri et al. 2022). Molasses is a by-product of making granulated sugar that cannot be crystallized (Kambo and Dutta 2014). Molasses is an essential energy source with sugar content in it. Molasses contains 25–40% sucrose and 12–25% reducing sugars with a total sugar content of 50–60% (Kambo and Dutta 2014). Syahfitri et al. (2022) reported that sorghum bagasse particleboard bonded with molasses has an excellent internal bond and achieves the JIS A 5908:2022 standard. However, the modulus of elasticity, rupture, and screw-holding power do not. Therefore, the use of molasses as an adhesive need to be improved.

A hot-pressing procedure is generally used to produce citric acid and sucrose-adhesive sorghum bagasse particleboard. Heat transmission during hot-pressing is necessary for hardening the adhesive in wood-based panels, which works by vapor migration from the panel's surface to its core (Silva et al. 2019). Poor bonding between particles and adhesive is caused by uneven heat transmission during pressing. Because most inorganic materials and organic molecules, such as polymers and wood, are poor conductors of heat and electricity, they are difficult to arrange in vibrational or excitation modes (Kumar et al. 2013). Therefore, modification with the addition of catalyst materials is needed to improve the curing process, which can accelerate the formation of bonds between bio-based adhesives and sorghum bagasse particles to improve the physical and mechanical properties of sorghum bagasse particleboard.

The thermal properties of nanoparticles as nanocatalysts open up new possibilities for heat transfer enhancement when added to composites by mixing nanocatalysts with adhesives (Kumar et al. 2013). In addition, nanocatalysts of nanotechnology compounds can penetrate the wood particles, effectively changing the surface chemistry and resulting in high protection against moisture (Mantanis and Papadopoulos 2010). Based on research conducted by Silva et al. (2019), adding zinc oxide (ZnO) nanoparticles to *Eucalyptus urophylla* x *grandis* particleboard with urea formaldehyde (UF) adhesive can improve the physical and mechanical properties of particleboard, especially density, thickness swelling, water absorption, modulus of elasticity and modulus of rupture as well as increase heat transfer during tempering thereby improving the interaction between particles and adhesive. Furthermore, graphene is the most appealing filler material for the fabrication of composite structures among all nanomaterials because of its ease of amalgamation,

exceptional yield, low cost, and adaptability with polymers due to surface chemical changes (Gul and Alrobei 2021). According to previous research, adding graphene oxide (GO) nanoparticles into the manufacturing process of medium-density fiberboard (MDF) made from wood fiber and UF adhesive improved the physical and mechanical properties of MDF, such as thickness swelling, water absorption, internal bonding, modulus of elasticity, and modulus of rupture, as well as increasing the heat transfer rate during hot pressing of MDF (Gul and Alrobei 2021). Therefore, this study analyzed the effect of adding ZnO nanoparticles and GO nanoparticles as nanocatalysts into bio-based adhesive sorghum bagasse particleboard.

2. Materials and Methods

2.1. Materials

The sorghum bagasse and citric acid in this study were obtained from the Biomass and Bioproducts Research Center, National Research and Innovation Agency, Cibinong, West Java. The molasses used was commercial molasses sold through e-commerce. Zinc oxide (ZnO) nanocatalysts were obtained by synthesizing materials such as zinc nitrate ($\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$) (HiMedia Laboratories, India), sodium hydroxide (NaOH) (HiMedia Laboratories, India), and ethanol (Supelco Analytical Products, USA) (Talam et al. 2012). In addition, graphene oxide (GO) nanocatalysts were obtained by synthesizing materials such as graphite powder, potassium permanganate (KMnO_4) (Merck, Germany), sodium nitrate (NaNO_3) (Merck, Germany), sulfuric acid (H_2SO_4) (Merck, Germany), hydrogen peroxide (H_2O_2) (Merck, Germany) and hydrochloric acid (HCl) (Merck, Germany) (Handayani et al. 2019).

2.2. Methods

2.2.1. Nanocatalysts preparation

In the wet chemical processing of zinc oxide nanocatalysts, zinc nitrate and sodium hydroxide was employed as precursors. The synthesis began with a magnetic stirrer continually swirling a 0.5 M ethanol solution of zinc nitrate ($\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$) for 1 h until the zinc nitrate was entirely dissolved. Then, a 0.9 M ethanol solution of sodium hydroxide (NaOH) was made similarly for 1 h. Following that, for 45 min, the sodium hydroxide solution was progressively added to the zinc nitrate solution at 280 rpm, and the reaction was sealed for 2 h under these circumstances. To precipitate, the solution was then allowed to rest overnight before the supernatant was carefully separated. The remaining solution and supernatant were centrifuged to separate the precipitate for 10 min. The precipitated ZnO nanocatalysts were rinsed three times with distilled water and ethanol to eliminate by-products attached to the nanocatalysts. Furthermore, the precipitated ZnO nanocatalysts were dried in an oven at 60°C. During the drying process, $\text{Zn}(\text{OH})_2$ was entirely changed into ZnO (Talam et al. 2012).

Meanwhile, 2.5 g of potassium permanganate (KMnO_4), 0.5 g of sodium nitrate (NaNO_3), and 0.5 g of graphite electrode powder were mixed with 23.3 mL of sulfuric acid solution (H_2SO_4) as an oxidizer to create graphene oxide nanocatalysts. For nearly 2 h, all chemicals were combined and dissolved in an ice bath at 20°C. After around 2 h of churning, the mixture's temperature should be increased to 35–40°C in a water bath and regularly stirred at 280 rpm for 14 h. Pure water of 41.67 mL was added to the beaker to cease the reaction. To determine oxidation, 3.3 mL of hydrogen peroxide (H_2O_2) was added to the solution, stirring for 30 min to speed up the process.

A total of 133.3 mL of distilled water and 5% HCl were added to the solution, and the solution was rinsed with distilled water until it achieved a neutral pH before being dried at 60°C. After adding 50 mL of distilled water, 0.1 g of graphite oxide was sonicated for 30 min. After centrifugation, the precipitate was dried in an oven at 60°C (Handayani et al. 2019). Then, the size of the nanocatalysts was measured using X-ray diffraction analysis. It is known that the average particle size of the ZnO nanocatalyst was 16.56 nm, and the GO nanocatalyst was 4.45 nm. After that, the ZnO and GO nanocatalyst were added to the adhesive, as presented in **Table 1**.

Table 1. Adhesive characteristics

Adhesive	Nanocatalyst	Solid content (%)	pH	Viscosity 20°C (mPa.s)	Torque (mN.m)	Gel time (min)
Citric acid	Zinc oxide (ZnO)	59	1.12	2.81	0.055	-
Molasses			4.62	176.43	3.294	4.5
Citric acid	Graphene oxide (GO)		1.05	2.82	0.056	-
Molasses			4.36	179.71	3.352	4.7

2.2.2. Adhesive preparations

Molasses with a solid content of 71% was combined with distilled water while being constantly stirred until the solid content reached 59 wt% (Syahfitri et al. 2022). The citric acid powder was then dissolved in the same manner as the molasses adhesive at a concentration of 59 wt% (Kusumah et al. 2016). Furthermore, the nanocatalysts of ZnO and GO were individually added to the molasses and citric acid adhesives at 0.5 wt% of the adhesive weight. The gel time of bio-adhesives was measured using the gel time meter (Techne GT-6, Coleparmer, USA) (Aisyah et al. 2023; Hariz et al. 2023). The sample was placed into the tube case, in position with the needle submerged in the sample. At 180°C, the time necessary for the glue to gelatinate was also noted. The adhesive gelatinization timeout is achieved during the auto-stop time. Afterward, the sample of bio-adhesives was placed on a rotating rheometer (RheolabQC, AntonPaar, Austria) to measure the viscosity and torque of bio-adhesives (Aisyah et al. 2023; Hariz et al. 2023). The average viscosity and torque were determined at rotating speeds of 115/s at room temperature. The characteristics of the adhesive can be seen in **Table 1**.

2.2.3. Manufacture of particleboards

The sorghum bagasse particles were ground to a sample size of 4–20 mesh and dried for 12 h at 80°C to achieve a moisture content of less than 8%. The adhesive solution with 20 wt% of resin content was mixed with sorghum bagasse particles. The mixed particles were dried at 103°C to reduce the moisture content (Syahfitri et al. 2022). The oven-dried particles were then formed using a 350 mm × 350 mm wooden mold and then pressed using a hot press at 200°C for 10 min at a pressure of 10 MPa. The thickness of the particleboard was controlled using a standing bar with a thickness of 6 mm. The target density of the particleboard was 0.8 g/cm³. The particleboard was then conditioned for a week. The variation of bio-adhesive and nanocatalyst in particleboard manufacturing is shown in **Table 2**.

Table 2. Type of particleboards bonded with bio-adhesive and nanocatalyst addition

Particleboard type	Type of adhesive (Factor a)	Type of nanocatalyst (Factor b)
CAZ	Citric acid	ZnO
MZ	Molasses	
CAG	Citric acid	GO
MG	Molasses	

2.2.4. Evaluation of particleboard properties

The particleboard was tested based on the Japanese Industrial Standard JIS A 5908:2022 for particleboard (JSA 2022). The mechanical properties were tested using a bending test consisting of modulus of rupture (MOR) and modulus of elasticity (MOE) using a sample measuring $200 \times 30 \times 6 \text{ mm}^3$. The sample will be bent at three points with a loading speed of 10 mm/min and a load range of 15 times the thickness of the sample. The sample was tested using a universal testing machine (Shimadzu 50 KN, Japan). Internal bond (IB) was tested with a sample measuring $50 \times 50 \times 6 \text{ mm}^3$. The test sample was attached to an iron plate connected to a universal testing machine and then pulled vertically to the board surface at a loading speed of 2 mm/min. Meanwhile, screw holding strength (SHS) was measured using a screw of 2.7 mm in diameter and 16 mm long, attached to the board sample of $100 \times 50 \times 6 \text{ mm}^3$ until it reached a depth of $\pm 5 \text{ mm}$. The screw was then pulled in the vertical direction at a loading speed of 2 mm/min, and the maximum load was recorded.

The dimensional stability test of the particleboard was carried out by measuring the change in thickness and weight after being soaked in water at 20°C for 24 h to determine the thickness swelling (TS) and water absorption (WA). Furthermore, cyclic tests were carried out using TS and WA samples that had been dried at 105°C for 10 h, then soaked in water at 70°C for 24 h, dried again at 105°C for 10 h, then soaked in water at 100°C for 4 h and dried again at 105°C for 10 h. The changes in thickness and mass that occurred during the treatment were measured. Each test parameter was conducted in five replicates.

2.2.5. FTIR spectroscopy

FTIR analysis was performed on particleboard before and after cyclic treatment (Kusumah et al. 2016). The particleboard was crushed to obtain particle powder, which was then tested with an FTIR spectrophotometer (FT-IR 4000, PerkinElmer Inc., USA).

2.3. Data Analysis

The mean values of mechanical properties and dimensional stability of the samples were compared with the minimum values required by JIS A 5908:2022 standard (JSA 2022). The type of adhesive (Factor a), type of nanocatalyst (Factor b), and interaction between Factor a and Factor b was analyzed using analysis of variance (ANOVA) at 0.05.

3. Results and Discussion

3.1. Mechanical Properties of Particleboards

Fig. 1 shows the modulus of elasticity (MOE) and modulus of rupture (MOR) values between 754–1,128 MPa and 5.65–9.69 MPa, respectively. Based on these findings, it is clear that

all of the particleboard MOE values did not meet the JIS A 5908:2022 type 8 particleboard standard. However, MOR values of the particleboards with citric acid (CAG) and molasses (MG) adhesives satisfied the JIS A 5908:2022 standard.

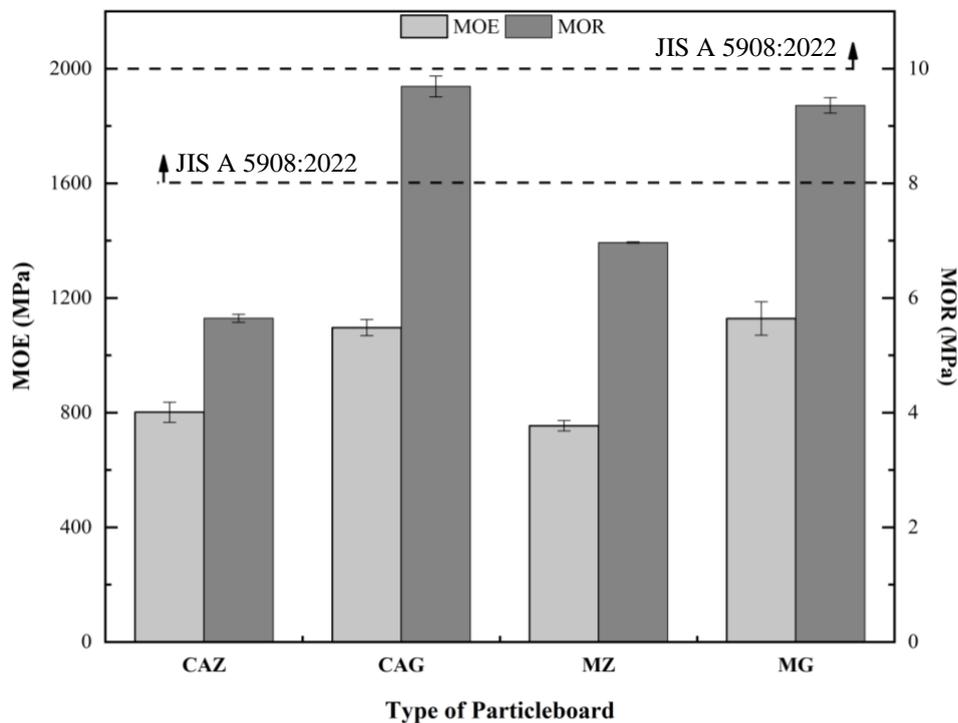


Fig. 1. Bending properties of particleboards.

The results in **Table 3** reveal that the type of nanocatalysts significantly influences particleboard MOE and MOR values. The inclusion of GO nanocatalysts produced superior bending characteristics than the addition of ZnO nanocatalysts. In contrast, previous studies reported that the particleboard without nanocatalysts had a MOE of 1,623 MPa (Syahfitri et al. 2022). The inclusion of nanocatalysts increased the brittleness of the particle as particleboard raw material. In line with previous research, the elevated level of heat conductivity by nanocatalysts causes the curing process to start earlier and faster, increasing the brittleness of the board (Gul and Alrobei 2021). Meanwhile, the addition of GO nanocatalyst for each particleboard bonded with citric acid and molasses had a MOR value higher than the previous research (Syahfitri et al. 2022). Gul and Alrobei (2021) reported that the higher crosslink is affected by adhesive curing.

Table 3. Variance analysis summary of particleboards

Properties of particleboard	Type of adhesive (Factor a)	Type of nanocatalyst (Factor a)	Interaction between Factor a and Factor b
Modulus of elasticity (MOE)	0.947 ^{ns}	0.016 [*]	0.729 ^{ns}
Modulus of rupture (MOR)	0.487 ^{ns}	0.001 [*]	0.259 ^{ns}
Internal bonding (IB)	0.325 ^{ns}	0.228 ^{ns}	0.458 ^{ns}
Screw holding strength (SHS)	0.530 ^{ns}	0.078 ^{ns}	0.398 ^{ns}
Thickness swelling (TS)	0.012 [*]	0.971 ^{ns}	0.850 ^{ns}
Water absorption (WA)	0.017 [*]	0.717 ^{ns}	0.292 ^{ns}

Notes: ^{ns}= not significant, ^{*}= significant.

Particleboards have internal bonding (IB) values of 0.125–0.181 MPa (**Fig. 2**). The IB values met the requirement of the JIS A 5908:2022 standard, except the citric acid (ZnO)-adhesive particleboard. The results revealed that the types of adhesive, types of nanocatalysts, or their interactions had no significant influence on the IB value of particleboard. **Fig. 2** shows that particleboard with molasses adhesive (MG) has the highest IB value in this study. However, the IB value is lower than that reported by [Syahfitri et al. \(2022\)](#), showing an internal bonding value of 1.92 MPa. The decrease in internal bond value could be caused by the over-curing of the adhesive during the pressing process. These results are in accordance with the findings of [Reinprecht et al. \(2018\)](#), stating that the addition of nanocatalysts could have a negative effect on the internal bonding of particleboard.

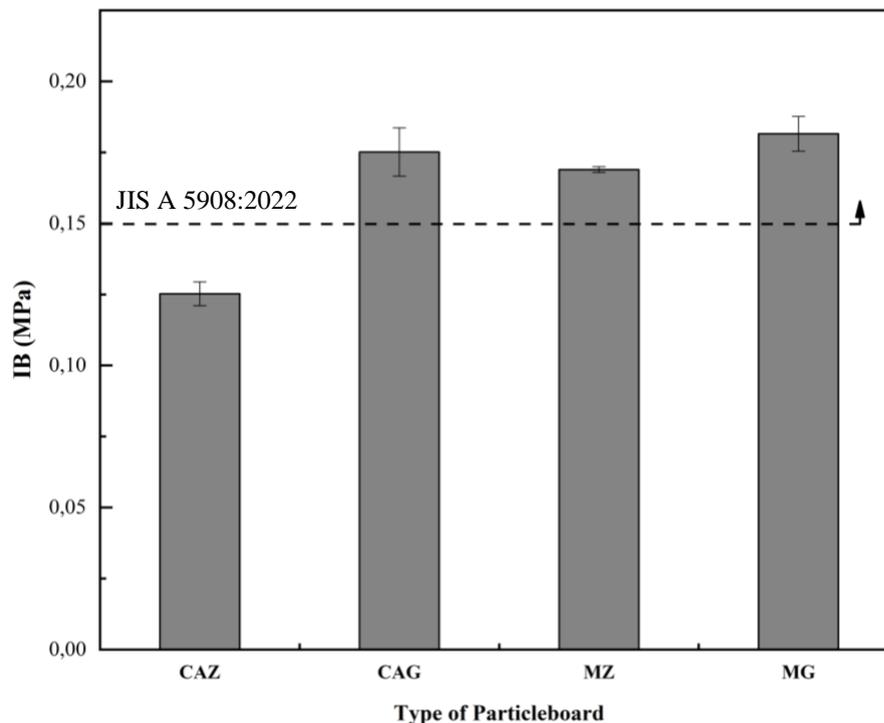


Fig. 2. Internal bonding of particleboards.

Fig. 3 presents the screw-holding strength of sorghum bagasse particleboard, showing the value from 89.76–111.44 N. However, the value of the screw holding strength does not reach the standard of JIS A 5908:2022. Based on the statistical analysis in **Table 3**, it is known that the type of adhesive, the type of nanocatalysts, or the interaction between the two factors do not significantly influence the value of screw-holding strength. The value of screw-holding strength in this study is higher than that in previous research ([Syahfitri et al. 2022](#)). It might be due to the increase in penetration of adhesive as a consequence of the ability of nanocatalysts to transfer heat during hot pressing ([Silva et al. 2019](#)). In addition, nanocatalysts can fill the empty gaps between the particles.

3.2. Dimensional Stability of Particleboards

The average values of TS and WA of particleboards varied from 6.47–11.92% and 40.28–49.26%, respectively (**Fig. 4**). The TS value satisfied the JIS A 5908:2022 standard. According to the statistical analysis results in **Table 3**, the adhesive type significantly affects the TS and WA

values of sorghum bagasse particleboard. [Kusumah et al. \(2017a\)](#) also reported that the adhesive type affected the TS value of particleboard. In addition, the TS and WA values in this study were higher than the TS and WA values in the previous study ([Syahfitri et al. 2022](#)).

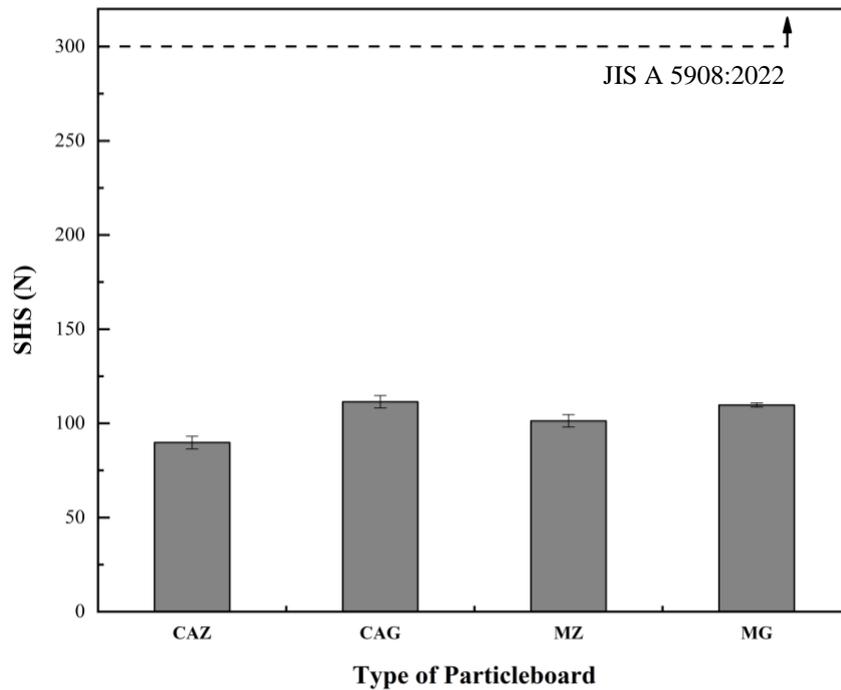


Fig. 3. Screw holding strength of particleboards.

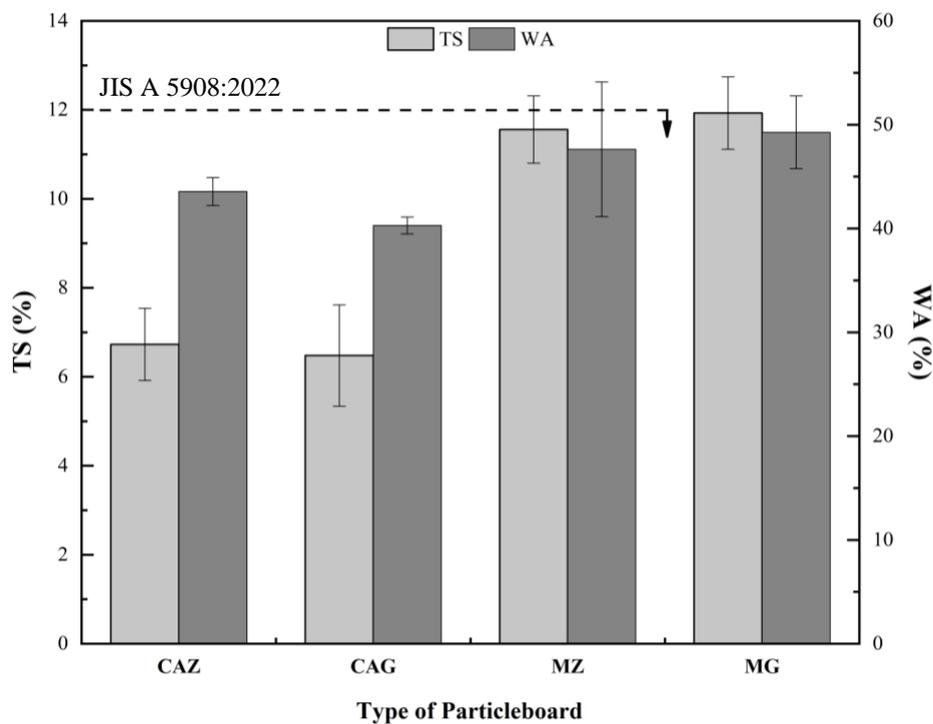


Fig. 4. Effect of adhesive and nanocatalyst on the thickness swelling and water absorption of particleboards.

Fig. 4 shows that the particleboard using citric acid adhesive has a lower thickness change than molasses adhesive. During the cyclic test process, the citric acid-adhesive particleboard

maintained its sample shape until the final drying process (**Fig. 5**). In contrast, the molasses-adhesive particleboard began to disintegrate and decompose under boiling conditions for 4 h. After the final drying, the thickness change of citric acid-adhesive particleboard with ZnO nanocatalysts and GO was 5.2% and 5%, while molasses-adhesive particleboard with ZnO nanocatalysts and GO had a thickness change of 20% and 15%. For citric acid-adhesive particleboard with the addition of ZnO and GO nanocatalysts, the change in thickness tends to be the same, indicating that citric acid adhesive with the addition of both ZnO and GO nanocatalysts produces particleboard with better dimensional stability. The carboxyl groups of citric acid reacted with the hydroxyl groups of the sorghum bagasse to form ester linkages (Kusumah et al. 2016). This result indicates that the ester linkages of citric acid-adhesive particleboard are more significant (**Fig. 7b**), resulting in better dimensional stability. Meanwhile, molasses-adhesive particleboard has poor dimensional stability. Therefore, adding GO nanocatalysts to molasses adhesive produces better dimensional stability than ZnO nanocatalysts.

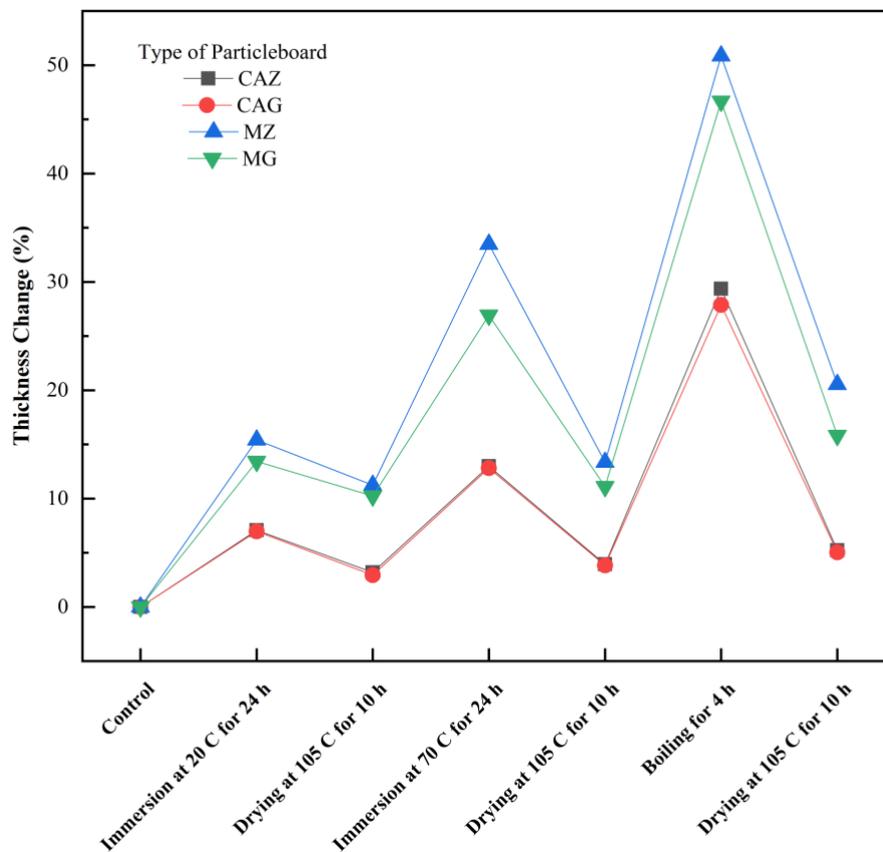


Fig. 5. Thickness change during cyclic aging treatment of particleboards.

Fig. 6 shows the weight changes of the particleboard during the cyclic test. Based on the figure, the citric acid-adhesive particleboard has a lower weight change than the molasses-adhesive particleboard in the adhesive test sample. After the first immersion at room temperature, further immersion treatments increased the weight change value of the particleboard. This result indicates that the board became more hydrophilic due to the loss of bond between the adhesive and the particles. After the boiling treatment, some of the molasses-adhesive particleboard samples were damaged. After the final drying, it was found that the weight change of citric acid ZnO and GO particleboards was 35% and 28%, while for molasses-adhesive ZnO and GO particleboards, it was

53% and 41%. This tendency is because when the sucrose in molasses is heated to 200°C, sucrose will convert into caramel, which contains a lot of water-soluble substances (Umemura et al. 2017). It is also known that particleboard with GO nanocatalysts has better weight change values than ZnO nanocatalysts.

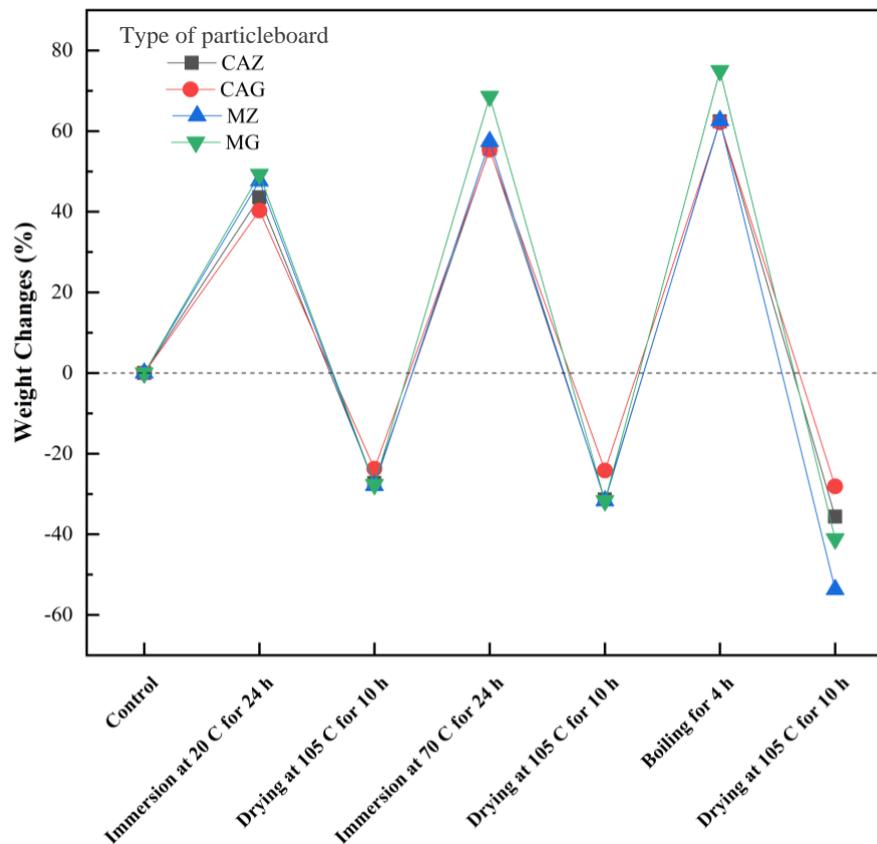


Fig. 6. Weight changes during cyclic aging treatment of particleboards.

3.3. Bonding Mechanism

Analysis of the bonding mechanism of the particleboard was carried out with FTIR, and **Fig. 7** presented the spectra of particleboards before and after cyclic aging treatment. The spectrum of particleboards before and after the cyclic aging treatment showed no new spectral peaks but had differences in intensity. **Fig. 7a** showed an absorption peak at 3340 cm^{-1} corresponded to the hydroxyl group (-OH) and at around 1720 cm^{-1} represented C=O stretching carboxyl group (Ma et al. 2019; Owodunni et al. 2020). The interaction of sorghum bagasse particles as lignocellulose material with citric acid, compared with molasses, before treatment can be seen from the intensity of the hydroxyl and carboxyl group.

Citric acid is rich in carboxyl groups which can be seen as the peak intensity at 1740 cm^{-1} is higher than molasses. This functional group can interact with cellulose through ester linkages (Fatriasari et al. 2021; Kusumah et al. 2016). Previous studies by Kusumah et al. (2016) reported that the carboxyl groups of citric acid reacted with the hydroxyl groups of the sorghum bagasse and formed ester linkages. These interactions are similar to molasses, where an ester bond is formed (Sutiawan et al. 2022).

After undergoing the cyclic aging treatment, the intensity in hydroxyl groups at 3340 cm^{-1} in citric acid ZnO and molasses ZnO was increased (**Fig.7b**). This result was probably because the dimensions were unstable, so cellulose was exposed and was no longer compressed. The cellulose molecules have many hydroxyl groups (Bi and Huang 2021). There was no significant change compared to the spectra of particleboards with citric acid and molasses GO. This tendency showed that GO can maintain bonds. However, the higher ester bond interaction in citric acid GO than molasses GO made the dimensions more stable. This tendency is confirmed, resulting in dimensional stability.

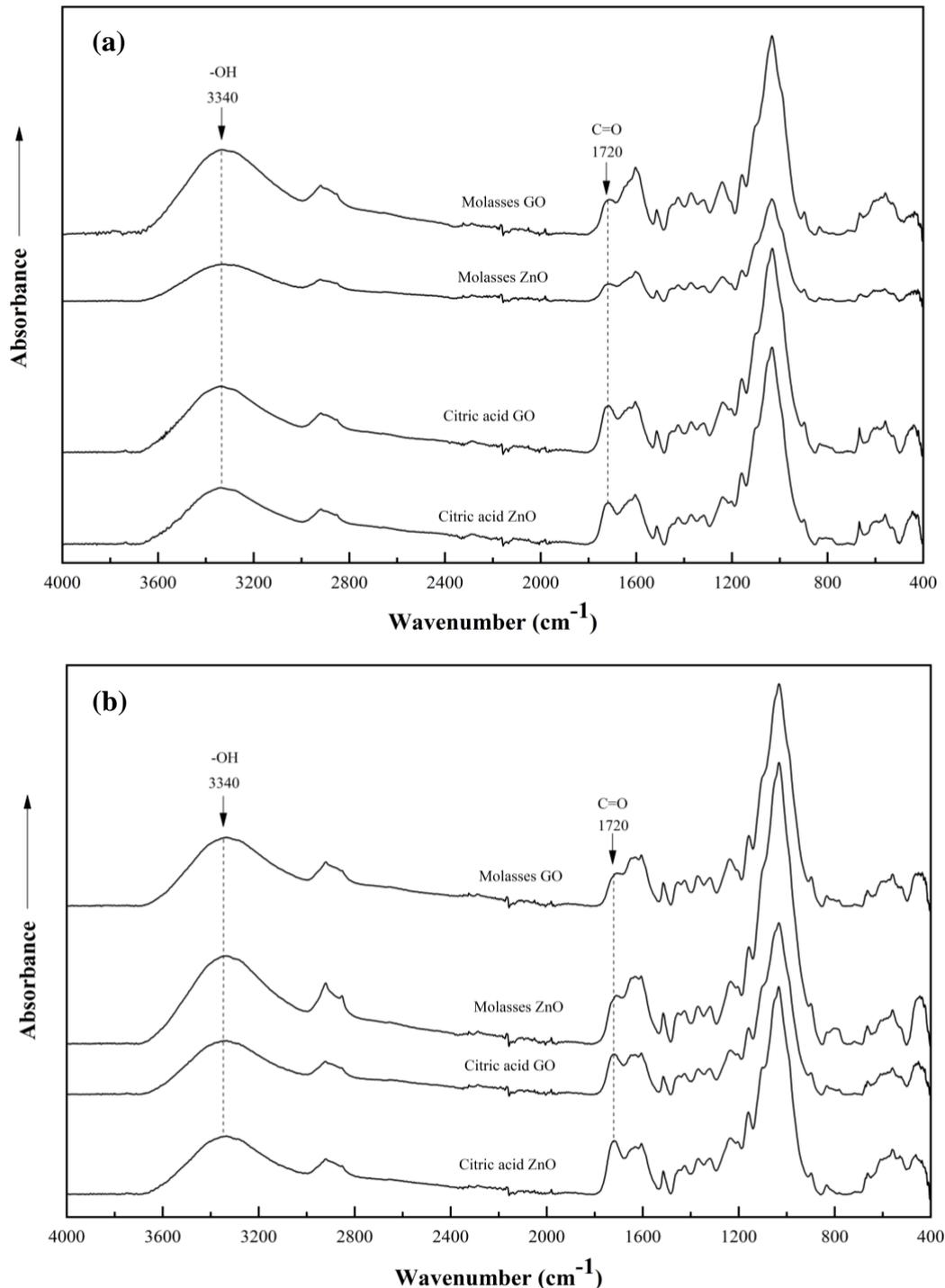


Fig. 7. Fourier transform infrared spectra of particleboards before (a) and after (b) undergoing the cyclic aging treatment.

4. Conclusions

This study revealed that incorporating GO nanocatalysts in the bio-adhesive could enhance the physical and mechanical properties of the particleboard compared to using ZnO nanocatalysts. The particleboard constructed with a GO nanocatalyst-infused adhesive exhibited MOR, IB, and TS values aligned with the JIS A 5908:2022 standard. Furthermore, our experiment found that citric acid adhesive amalgamated with GO nanocatalysts outshone the molasses adhesive in terms of better TS, WA, and MOR values. FTIR analysis further confirmed an increase in the production of ester bonds in particleboards utilizing citric acid adhesive combined with ZnO and GO nanocatalysts, compared with those using molasses adhesive. In conclusion, integrating GO nanocatalysts creates particleboards with superior properties, outperforming the ones generated through ZnO nanocatalysts.

Acknowledgments

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