Comparative Effect of Biofillers in pMDI Resin Performance

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Abstract

This study investigated the feasibility of using wood flour (WF) as a partial substitute in polymeric methylene diphenyl diisocyanate (pMDI) resin and compared its performance with soy flour (SF) substituted in pMDI resin. The physical and mechanical properties of experimental particleboards made with WF and SF substituted in pMDI resin at different substitution percentages were evaluated. The viscosity for the WF at different substitution ratios (5%, 10%, 20%, and 30%) ranged from 314.7 to 6,256.3 cP, whereas SF-substituted resin ranged from 249.7 to 1,291.8 cP. During the production of the boards, it was observed that because of the high viscosity of WF substituted in pMDI resin above 10 percent, it was exceedingly difficult to apply it through spraying and brushing, either to wood particles or veneers. Dimensional stability test results established that the incorporation of SF assisted in mitigating board thickness swelling. The results from the study showed that panels made with SF substituted in pMDI resin at 5 and 10 percent exhibited the overall best performance in all the properties considered compared with panels made with WF substituted in pMDI resin.

The global demand for wood composites had been significantly increased in the last two decades (Alawode et al. 2020a). The global production reached 444.1 million m^3 in 2020, with a projection of 658.1 million m^3 in 2027 (CISION PR Newswire 2020). The market analysis published by Grand View Research reported that the global wood-based panel market size was estimated at USD 144.67 billion in 2019 and is anticipated to expand at a revenue-based compound annual growth rate of 6.9 percent from 2020 to 2027 (Grand View Research 2020). The significance of wood adhesives in wood composites production cannot be overemphasized. Wood adhesives are categorized into natural and synthetic adhesives. Formaldehyde-based adhesives (phenol–formaldehyde, urea–formaldehyde, etc.) are the most used synthetic adhesives because of their superior bonding properties (Amini et al. 2013, Alawode et al. 2019, Govender et al. 2020). However, there has been a recent campaign against their use because of the carcinogenicity of formaldehyde (Khosravi et al. 2010, Amini et al. 2013, Zhang et al. 2014). The adverse effects of formaldehyde emission on the environment and human health have been widely reported (Van Langenberg et al. 2010, Dongre et al. 2015, Hemmilä et al. 2017). Furthermore, the oil market heavily influences the price of synthetic adhesives, resulting in price volatility. The depletion of fossil fuel supplies is a significant worry, putting the future availability of synthetic adhesives in doubt (Hemmilä et al. 2017, Antov et al. 2020). Therefore, researchers have made several attempts to develop adhesives free of formaldehyde (Van Langenberg et al. 2010, Sulaiman et al. 2013, Alawode et al. 2020b). Also, quite a few studies have discussed the advantages that the incorporation of soy flour (SF) in polymeric methylene diphenyl diisocyanate (pMDI) resin has brought to the wood adhesive system, such as cost reduction, properties improvement, etc. For instance, Cheng et al. (2019) showed that a partial resin substitution of 15 percent in pMDI resin used to produce oriented strand board and particleboard results in significant cost savings without compromising wet and dry board properties. Asafu-Adjaye et al. (2020a) also reported that incorporating SF into pMDI resin increased its cold tack and improved the prepress integrity of a mat. This innovation offers a strong

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technological and economic case for its application in engineered wood (Asafu-Adjaye et al. 2020a).

Many adhesive systems used currently in the wood composite industry do not emit formaldehyde, though they are fossil fuel based, such as pMDI resin. The use of fillers in pMDI systems helps lower costs, thereby promoting its use, thus reducing the need for formaldehyde-containing adhesive systems. pMDI resin is gaining broader acceptance in the wood composites industry because it does not contain formaldehyde (Asafu-Adjaye et al. 2020a). Despite pMDI's high acceptability, its major concern is its high cost. Various studies are currently ongoing to reduce costs (Cheng et al. 2019, Asafu-Adjaye et al. 2020b, Alawode et al. 2022). Recently, the use of SF was successfully established as a partial substitute in pMDI resin. According to one study, SF could be perfectly substituted at a certain percentage without compromising the strength properties of the wood composites (Asafu-Adjaye et al. 2020b).

Furthermore, several recent types of research on the performance and application of wood flour (WF) as a partial replacement in the adhesive formulation in the composite manufacturing process have also been published. The physical properties of cornstarch-based biodegradable polymers—Mater-Bi family—were examined after adding WF with two granulometries. The authors investigate the effects of WF size, content, and thermal treatment on the composite's mechanical properties. The results of the tensile mechanical testing revealed that increasing the WF content increases the stiffness of the composites (Morreale et al. 2008). The impact of date WF as a filler on the mechanical properties of polyethylene composites was also evaluated. The mechanical parameters, flexural strength (modulus of rupture [MOR]) and tensile strength, dropped as the filler content rose, whereas the flexural modulus (modulus of elasticity [MOE]) increased (Mirmehdi et al. 2014). However, other studies indicate that the impact strength of the composite is maintained with increasing filler content up to about 40 percent loading in weight (Peng et al. 2021).

This paper aims to compare the effects of WF and SF as partial substitutes in pMDI resin. This study was motivated by a recent debate that arose because of the availability of WF in the wood composite industry, with WF being proposed as a better filler option to SF. Therefore, in this study, the performance of particleboards bonded with SF substituted in pMDI resin was compared with that of boards bonded with WF as a partial substitute in pMDI resin.

Materials and Methods

Defatted SF (7B) was provided by Archer Daniels Midland (Chicago, IL); Its dry-basis moisture content (MC) was 6.2 percent. System Three 3110S47 brown WF was purchased from System Three Resin Incorporated, USA. The pMDI resin was MONDUR 541 from Covestro. Wood particles (0.5–1.5 mm) were provided by West Fraser and dried to an ambient 6 to 7 percent MC. Emulsified wax (Hexion Bord'N-Seal FMH-XD) was obtained from Huber Corp.

Particle size

The particle size measurements for the WF and SF used for the modification in this study were carried out using dynamic light scattering using a Malvern 3000 Zetasizer Nano-ZS (Malvern Instruments, UK).

Viscosity

The viscosity for the WF- and SF-substituted resins at different substitution percentages were conducted using IKA Viscometers ROTAVISC me-vi Complete with ident. no. 0025000310.

Fourier transform infrared spectroscopy analysis

The samples for SF, WF, pMDI, SF-modified pMDI, and WF-modified pMDI resin were analyzed using Fourier transform infrared (FTIR) spectroscopy operating in attenuated total reflectance (ATR) mode. This analysis monitored changes in the modified samples' structure and established that modification occurs. The samples were pressed against the diamond crystal surface with a spring-loaded anvil of a Thermo Nicolet, NexusTM model 470/670/870 FTIR spectrometer equipped with ZnSe lenses. Spectra were collected in ATR mode at a resolution of 4 cm^{-1} and 32 scans per sample within the absorption bands in the 4,000- to 650-cm region. Data collection and further processing were carried out in the Thermo Scientific Origin software.

Particleboard preparation

Southern yellow pine particles were conditioned at 20°C and 65 percent relative humidity (RH) for 96 hours. The equilibrium MC of the materials was determined as 7 percent. About 4 percent of the adhesives and 1 percent of wax on the basis of the target board mass were mixed with the wood particles using a rotary drum mixer. The SF/WF pMDI mixture was prepared by thoroughly stirring SF/WF and pMDI at 40°C and used within a few minutes of preparation. The furnish was poured into a wooden mold measuring 43 × 43 cm, placed on an aluminum steel plate, and a steel bar of 1.1 cm thick was set on top of the plate. The purpose of the steel bar was to compress the composite to a final thickness of 1.1 cm. The setup was transferred to the laboratory press, where it was subjected to a pressure of 2.1 MPa for 3 minutes at 180°C. Before testing, the formed boards were removed from the press and conditioned at 20°C and 65 percent RH for 96 hours. Two panels were made for each modified resin formulation condition, and five particleboard samples were taken from each.

Particleboard evaluation

The properties of the formed panels were evaluated to investigate the effect of WF and SF substitution in pMDI resin on the flexural strength of the panels. Flexural test specimens were tested according to ASTM D 1037-12 standard using an Instron testing machine fitted with a 10-kN load cell, operated at a rate of 5 mm/min. The specimens were tested to failure, and the MOR and MOE were measured. Samples for the flexural test were cut using a horizontal band saw into dimensions of 215 × 75 mm. The specimens were tested for internal bonding following ASTM D 1037-12 standards. The test samples were cut into 2-inch (50-mm) squares with the thickness of finished boards (1.1 cm). Loading blocks of steel 2-inches square and 1 inch (25 mm) in thickness were effectively bonded with a hot melt adhesive (stick glue) to the 2-inch square faces of the samples. Appropriate pressure was used to bond the blocks to the specimen, not to damage the sample. The resulting bond was stronger than the material's cohesive strength perpendicular to the panel's plane. Cross-sectional dimensions of

the specimen were measured to an accuracy of not less than ±0.3 percent. The maximum distance from the center of the universal joint or self-aligning head to the glued surface of the specimen was 3 inches (76 mm). The loading fixtures were attached to the heads of the testing machine (Instron), with the blocks attached to the specimen. The test speed was 2mm/min with a load cell of 10 kN. The samples were stressed by separating the heads of the testing machine until failure occurred. The direction of loading was as nearly perpendicular to the faces of the specimen as possible, and the center of the load was passed through the center of the sample. The applied load was continuous throughout the test at a uniform rate of 0.08 inch/inch thickness per minute. The internal bonding was calculated as a shear strength in MPa from obtained maximum loads. Ten replicates of 2 inches by 2 inches were tested for each treatment.

Samples for dimensional stability were the same size as those used for the flexural test. Water absorption (WA) and thickness swelling properties (TS) were carried out by submerging conditioned specimens horizontally in freshwater for 24 hours. After submersion, the samples were suspended to drain for 10 minutes and excess water was removed from the surface. The specimens were weighed and the thickness was determined as an average of four measurements. The WA of the samples was calculated from the increase in weight and expressed as a percentage of the conditioned weight:

$$\text{WA } (\%) = \frac{\text{Final weight} - \text{Initial weight}}{\text{Initial weight}} \times 100,$$

whereas the TS of the specimen was calculated as a percentage of the conditioned thickness:

$$\text{TS } (\%) = \frac{\text{Final thickness} - \text{Initial thickness}}{\text{Initial thickness}} \times 100$$

Statistical analysis

Statistical analysis was performed using Statistica software (Statsoft v13). The data were analyzed by 1-way analysis of variance at 5 percent significance to determine if the formulation parameters have significant effects on the measured properties. In addition, multiple means were compared with a post hoc Fisher least significant difference test at 5 percent to evaluate if there were significant differences among the panels.

Results and Discussion

The SF and WF percentage substituted into pMDI resin in this study was done on the basis of the weight of the pMDI. The notation for each sample is indicated in Table 1 for convenience purposes, and images for the formulated resins used in this study are presented in Figure 1. The viscosity values for the formulated resin are shown in Table 2. The viscosity for the WF-substituted resin at different substitution ratios (5%, 10%, 20%, and 30%) ranged from 314.7 to 6,256.3 cP. At the same time, it ranged from 249.7 to 1,291.8 cP for SF-substituted resin. We could not produce and compare boards between WF and SF resin at higher substitution percentages because of high viscosity values for WF substituted in pMDI resin at 20 percent and 30 percent substitution (see Figure 1). We observed that it was impossible to spray WF substituted in pMDI resin above 10 percent. An attempt was also made to see the possibility of applying it by brush on veneers for plywood construction. Still it was not feasible because of difficulty obtaining a complete even spread that influenced bond quality. It was established from this study that one of the significant setbacks in using WF as a synthetic resin partial substitute in wood composites industries is its high viscosity.

Table 1.—Formulation descriptions of the samples.

Sample notation	Descriptions
pMDI	Polymeric methylene diphenyl diisocyanate resin
5% WF	5% Wood flour substituted in pMDI resin
10% WF	10% Wood flour substituted in pMDI resin
20% WF	20% Wood flour substituted in pMDI resin
30% WF	30% Wood flour substituted in pMDI resin
5% SF	5% Soy flour substituted in pMDI resin
10% SF	10% Soy flour substituted in pMDI resin
20% SF	20% Soy flour substituted in pMDI resin
30% SF	30% Soy flour substituted in pMDI resin

Particle size measurement

Particle size measurements were carried out on SF and WF used to modify pMDI resin. This analysis is to establish if the variation shown in the performance of the modified resins were due to a considerable difference in the flour's surface area and particle size geometry, which may influence their viscosity. As measured by dynamic light scattering, the particle sizes of SF and WF are shown in Figures 2a and 2b. The particle sizes for SF and WF are 1.32 and 1.55 μm, respectively. This result indicates that particle size as a factor did not influence the viscosity of the modified resins since their particle sizes are almost the same.

FTIR spectroscopy analysis

FTIR spectroscopy was used to monitor changes in the structures of pMDI resin upon modification with SF and WF separately. Figure 3 and Table 3 illustrate the FTIR information of SF, WF, pMDI resin, and modified samples. The emergence of peak 2,261.81 cm^{-1}, which corresponds to N=C=O stretching in the SF-modified pMDI resin, established the modification process. The peak at 1,047.54 cm^{-1} was detected in the SF and was attributed to anhydride and assigned to the C–O, C–C stretching, or C–OH bending in SF (Hashim et al. 2011). Also, the emergence of peaks at 2,267.88 cm^{-1} and 916.70 cm^{-1} in the WF established an interaction between WF and pMDI resin. This peak at

Table 2.—Formulated resin viscosity and corresponding temperatures.

Samples	Viscosity (cP)	Temperature (°C)
pMDI[a]	220.3	25
5% WF	314.7	23.2
5% SF	249.7	22.8
10% WF	622.4	23.2
10% SF	314.8	23.3
20%WF	3,835.3	22.9
20% SF	632.5	23.5
30% WF	6,256.3	23
30% SF	1,291.8	23.5

[a] pMDI = polymeric methylene diphenyl diisocyanate; WF = wood flour; SF = soy flour.

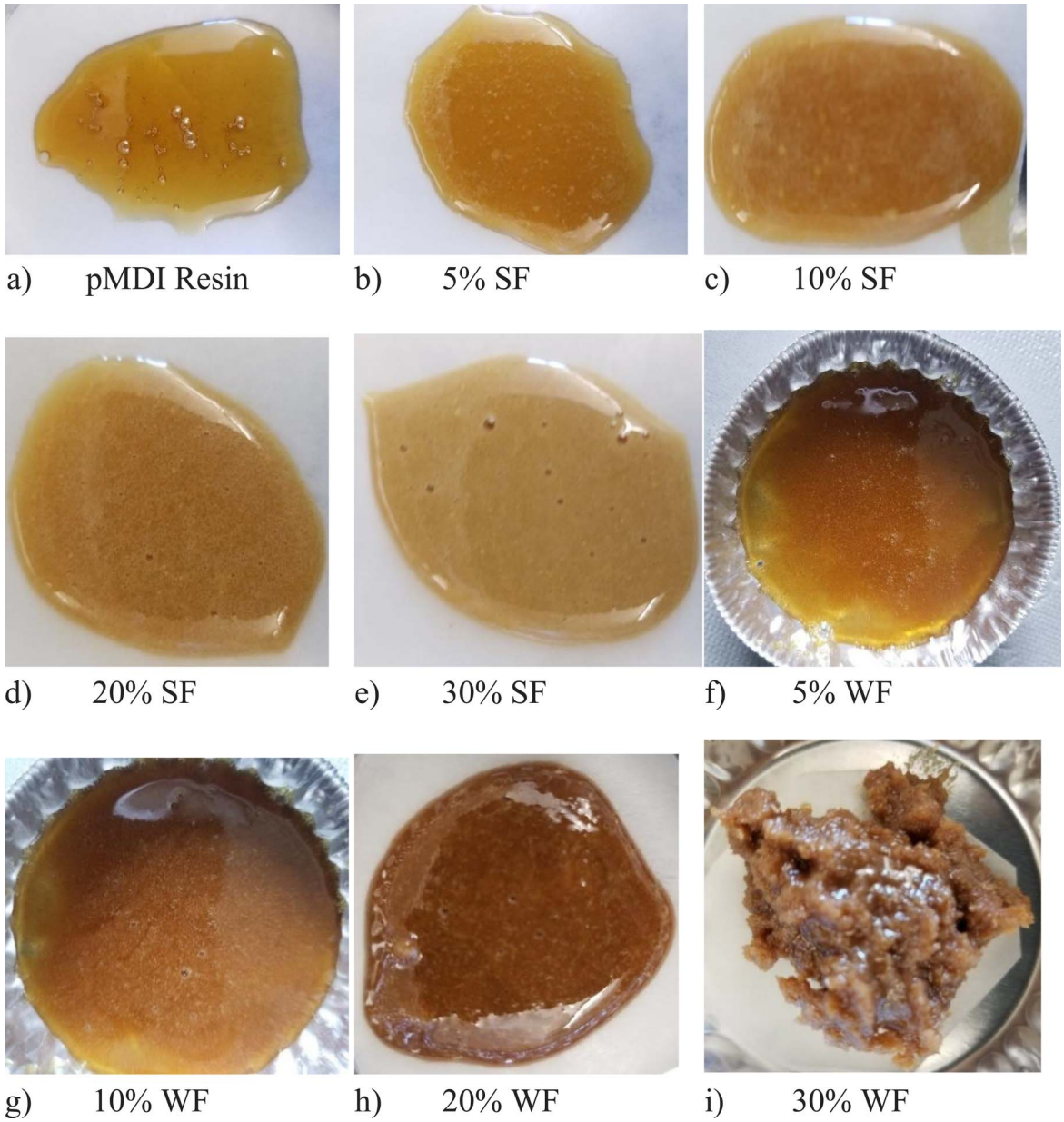

Figure 1.—Images of formulated resins.

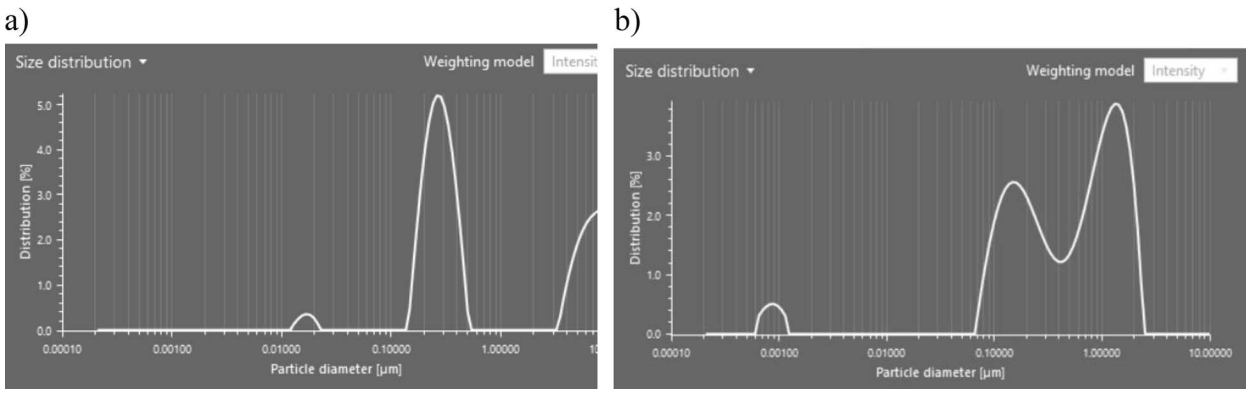

Figure 2.—Size distribution of (a) soy flour (SF) particle and (b) wood flour (WF) particle.

Table 3.—FTIR band assignment in modified resins samples.[a]

Wave number (cm^{-1})	Assignment/functional groups
3,340–3,277	O–H stretching
2,267.88	N=C=O stretching, isocyanate (pMDI resin)
1,664.18	C=O stretching (conjugated ketone)
1,505.72	N–O stretching, nitro compound (SF)
1,408.69	O–H bending, carboxylic acid (WF and SF)
1,305.51	C–N stretching, aromatic amine (SF)
1,228.77	C–N stretching, amine (SF)
1,178–1,076	–CO stretching of polysaccharide moiety
1,047.54	C–O, C–C stretching in SF
961.70	C=C bending, alkene

[a] pMDI = polymeric methylene diphenyl diisocyanate; WF = wood flour; SF = soy flour.

2,267.88 cm^{-1} corresponds to N=C=O stretching. There was a sharp peak in the pMDI resin at 2,243.23 cm^{-1} attributed to isocyanate (N=C=O stretching). It was observed that this peak with high intensity at 2,243.23 cm^{-1} in the pMDI resin spectra was obviously reduced in both modified SF and WF pMDI resin samples. The intensity of OH functional groups, indicated with the peaks between 3,277 and 3,340.58 cm^{-1}, is obviously higher in the SF spectra than in WF. The absorption band between 900 and 1,200 cm^{-1} was characteristic of the –CO stretching of the polysaccharide moiety (Hosseinpourpia et al. 2018, Alawode et al. 2020b).

Particleboard density

Density is crucial in particleboard development because it influences other important properties of the product. The mean values of density measured for the panels made with pMDI (control) and WF- and SF-substituted pMDI resin are presented in Figure 4. The actual density values shown for

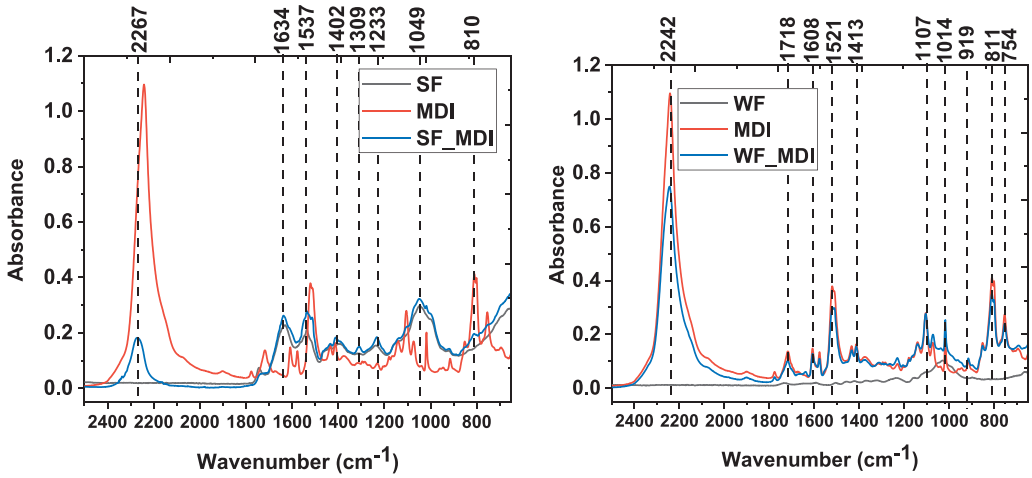

Figure 3.—Attenuated total reflectance–Fourier transform infrared (ATR-FTIR) spectra of samples.

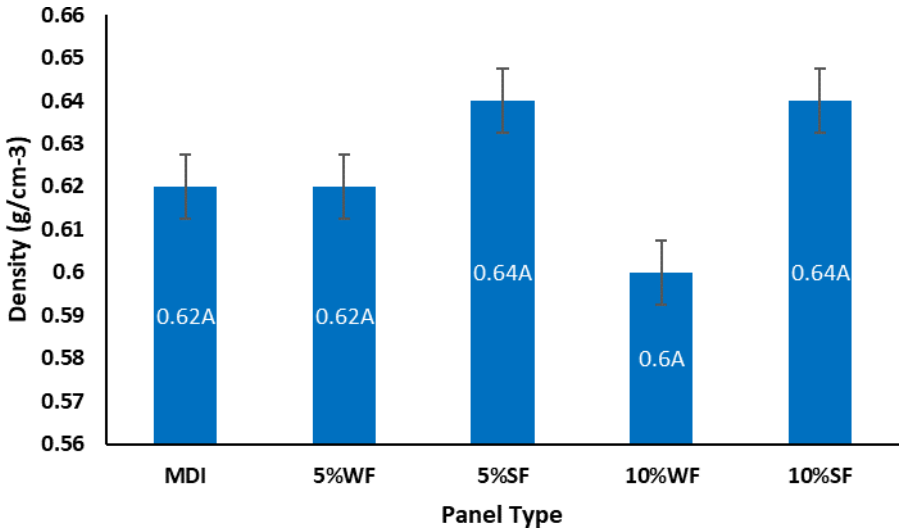

Figure 4.—Density of boards made with different resin formulations. Error bars indicate standard deviation of the average density values of 10 particleboard sample measurements. Means that do not share a letter are significantly different (Fisher least significant difference [LSD] test).

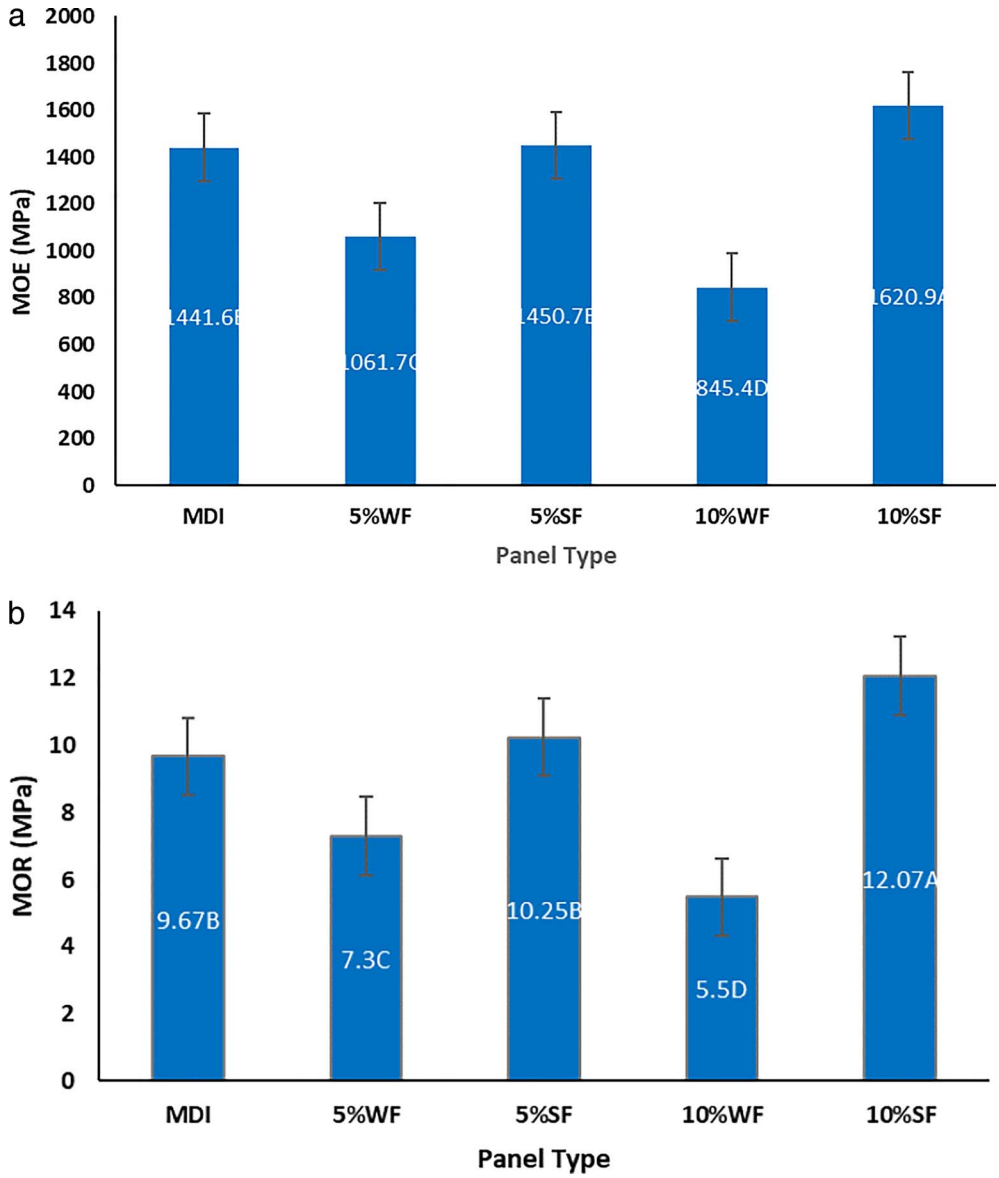

Figure 5.—(a) Dry modulus of elasticity (MOE) results of boards made with different resin formulations. Error bars indicate standard deviation of the average MOE values of 10 particleboard samples measurements. Means that do not share a letter are significantly different (Fisher least significant difference [LSD] test). (b) Dry modulus of rupture (MOR) results of boards made with different resin formulations. Error bars indicate standard deviation of the average MOR values of 10 particleboard sample measurements. Means that do not share a letter are significantly different (Fisher LSD test).

all the samples are very close, if not the same, with the target board density, which is 0.641 g/cm^3. These established that the boards were accurately produced. Panels with a density between 0.60 and 0.80 g/cm^3 are classified as medium-density panels according to American National Standards Institute (ANSI 2016). Furthermore, it was observed that there was no significant difference in the density among the panels produced, and this proved that they were on the same basis for comparison.

Particleboard dry strength

As presented in Figures 5a and 5b, the dry flexural and stiffness properties of boards bonded with SF and WF resin formulations were evaluated by measuring the MOE and MOR, respectively. The MOE values for boards produced with 5 percent WF and 5 percent SF resins are 1,061.7 MPa and 1,450.7 MPa, respectively, whereas for boards made with 10 percent WF and 10 percent SF resins are 845.4 MPa and 1,620.9 MPa, respectively. MOR values for panels bonded with 5 percent WF and 5 percent SF resin are 7.3 MPa and 10.25 MPa, respectively; 5.5 MPa and 12.07 MPa are for panels bonded with 10 percent WF and 10 percent SF resin, respectively. Compared with WF panels, the dry MOE for boards bonded with SF substitution was 36.6 percent higher at 5 percent substitution and 91.7 percent higher at 10 percent substitution. This performance could be attributed to the formation of urethane groups due to interactions between isocyanates and hydroxyl-containing compounds

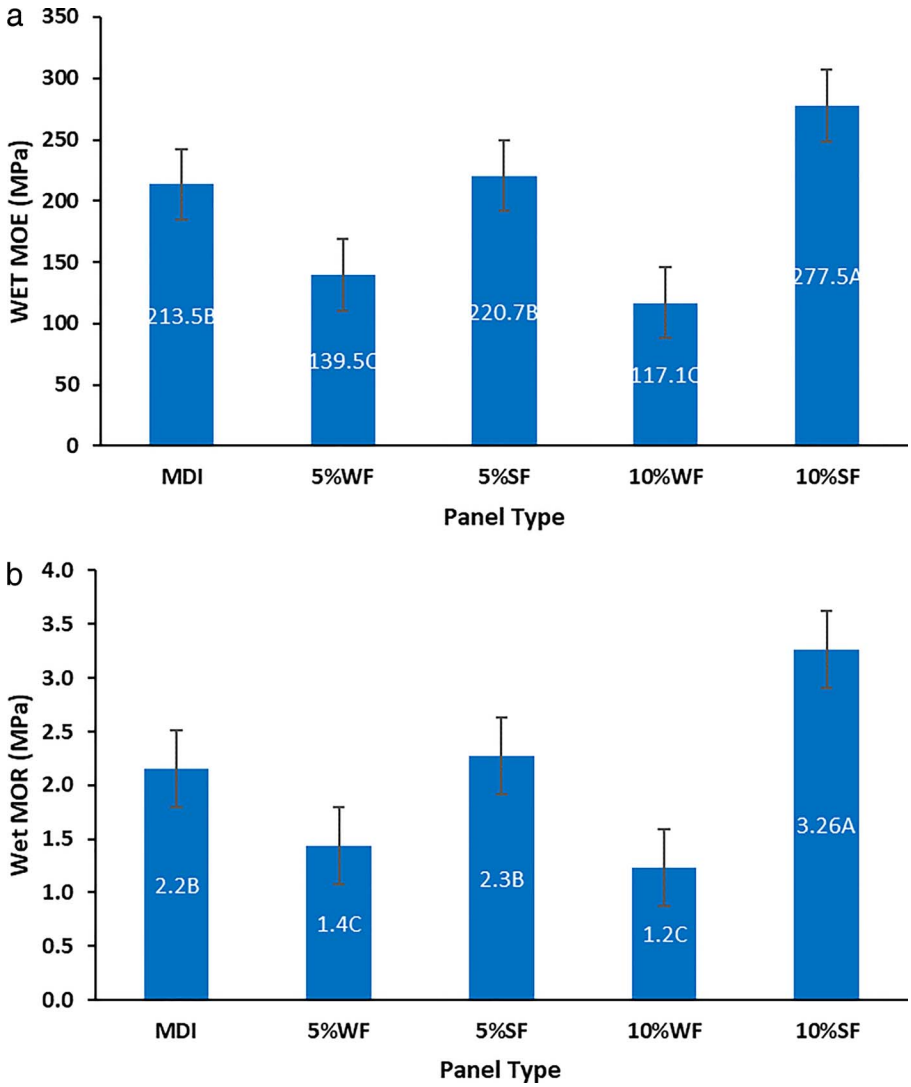

Figure 6.—(a) Wet modulus of elasticity (MOE) results of boards made with different resin formulations. Error bars indicate standard deviation of the average MOE values of 10 particleboard sample measurements. Means that do not share a letter are significantly different (Fisher least significant difference [LSD] test). (b) Wet modulus of rupture (MOR) results of boards made with different resin formulations. Error bars indicate standard deviation of the average MOR values of 10 particleboard sample measurements. Means that do not share a letter are significantly different (Fisher LSD test).

in SF. It could also be due to immediate interactions between amine groups in SF and isocyanates, resulting in the formation of substituted urea (Frisch et al. 1983, Asafu-Adjaye et al. 2020a;). The trend for dry MOR is similar, with 5 percent and 10 percent substitution. Compared with boards created with WF replacement in pMDI resin, panels bonded with SF substitution were 40.4 percent and 119.5 percent higher, respectively. The dry MOR values of panels bonded with a 5 percent substitution for SF were marginally higher than those bonded with pMDI (control).

Particleboard wet strength

Figures 6a and 6b show the wet MOE and MOR values for panels manufactured with various resin compositions. The panels manufactured with SF substitution were 58.2 percent higher than those made with WF substitution, according to the data shown in Figure 6a for the wet MOE at 5 percent substitution.

Noticeably, at 10 percent substitution, it was shown that the wet MOE for panels made with SF substitution was significantly different from panels produced with WF substitution, with the results indicating 137 percent greater in value than WF's panels. Also, boards made with 5 percent SF were 3.4 percent higher than panels with control (pMDI resin only). Panels made with 10 percent SF substitution gave the highest wet MOE, whereas panels made with 5 percent WF substitution had the least mean wet MOE values.

Similarly, the results for wet MOR, as shown in Figure 6b, followed the same pattern as the wet MOE results. Panels made with SF were 64.3 percent and 171.6 percent higher than panels made with WF at 5 percent and 10 percent substitution, respectively. At 5 percent there was no

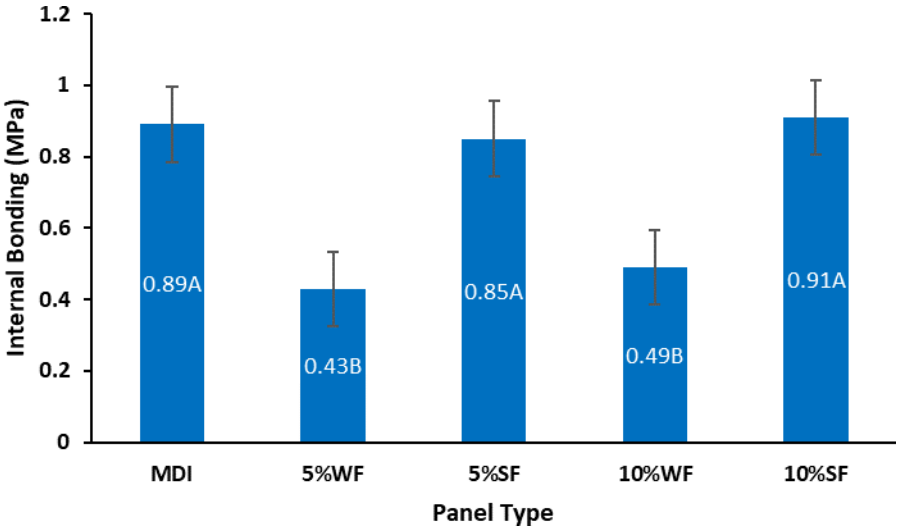

Figure 7.—Internal bonding (IB) results of boards made with different resin formulations. Error bars indicate standard deviation of the average IB values of 10 particleboard sample measurements. Means that do not share a letter are significantly different (Fisher least significant difference [LSD] test).

statistical difference between panels manufactured with 5 percent SF substitution and control panels. In comparison, panels manufactured with 5 percent and 10 percent SF substitutions showed a significant difference from those made with 5 percent and 10 percent WF substitutions.

Particleboard internal bonding

The mean values of internal bonding (IB) for boards made with different resin formulations are shown in Figure 7. The panels made with 5 percent and 10 percent SF substitutions in pMDI resin did not show any significant differences from panels made with only pMDI resin (control). Still, they differed from the boards made with WF substituted in pMDI resin at 5 percent and 10 percent substitution ratios, which had the lowest mean values. The IB values for 5 percent SF resin boards were 97.7 percent higher than for 5 percent WF boards. At 10 percent substitution, the panels manufactured with SF were 85.7 percent higher than those made with WF. The panels manufactured with 10 percent SF substitution yielded the greatest IB mean values, whereas panels made with 5 percent WF produced the lowest mean values.

Thickness swelling

Figure 8a shows the mean and standard deviation of TS values for boards manufactured with various resin formulations. Five percent WF and 5 percent SF resin boards have TS values of 17 and 18 percent, respectively. For panels bonded with 10 percent WF and 10 percent SF, the TS values are 25.6 percent and 18.5 percent, respectively. According to the analysis of the results, the average TS value of panels bonded with 5 percent SF was 5 percent greater than panels produced with 5 percent WF. With 10 percent substitution, the trend is reversed; the mean TS values of panels bonded with 10 percent WF were 38.4 percent higher than those of panels bonded with 10 percent SF. It is important to note that the lower the TS values, the greater the particleboard's ability to resist moisture. Compared with boards bonded with WF substituted in pMDI resin, panels bonded with SF substituted in pMDI resin had lower values, making them more promising when considering outdoor applications.

Water absorption

Figure 8b shows the mean and standard deviation of WA values for boards produced with various resin compositions. The WA values for 5 percent WF and 5 percent SF resin boards are 31.7 percent and 25.8 percent, respectively. At the same time, the WA values for panels bonded with 10 percent WF and 10 percent SF are 40.8 percent and 37.1 percent, respectively. According to the findings, the average WA of panels bonded with 5 percent WF was 22.9 percent higher than panels produced with 5 percent SF. The pattern is similar with 10 percent substitution; the mean WA of panels bonded with 10 percent WF was 10 percent greater than that of panels bonded with 10 percent SF. It is important to note that the lower the WA values for particleboard moisture resistance ability, the better. According to the results, panels bonded with SF-substituted pMDI resin had lower values, making them more promising for outdoor applications than boards bonded with WF-substituted pMDI resin.

Conclusion

This study showed that boards made with SF substituted in pMDI at 5 and 10 percent showed the best overall performance in all attributes evaluated in this study. This performance was ascribed to a better resin distribution and an interaction between the SF proteins and the pMDI resin's isocyanate groups. On the other hand, the performance of WF substitution in pMDI resin is limited to 10 percent; above that, increased viscosity makes resin distribution problematic. Because of a competing reaction of the pMDI (isocyanate) with the hydroxyl groups of the WF and wood particles, the overall mechanical and physical qualities of the panel made with WF-substituted pMDI resin were low.

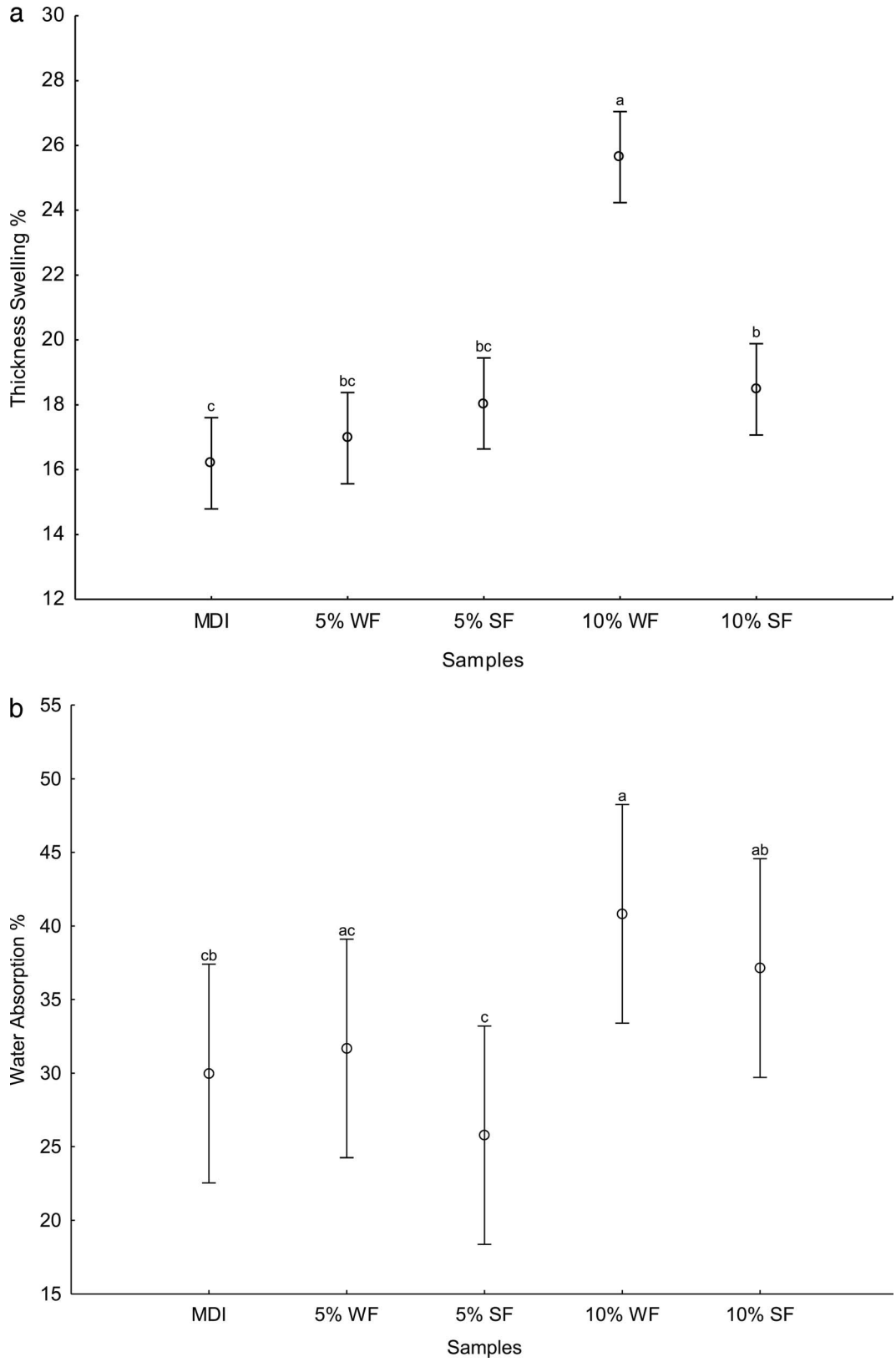

Figure 8.—(a) Thickness swelling (TS) of boards made with different resin formulations. Error bars indicate standard deviation of the average TS values of 10 particleboard sample measurements. Means that do not share a letter are significantly different (Fisher least significant difference [LSD] test). (b) Water absorption (WA) of boards made with different resin formulations. Error bars indicate standard deviation of the average WA values of 10 particleboard sample measurements. Means that do not share a letter are significantly different (Fisher LSD test).

Acknowledgments

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