Effects of Density, Cellulose Nanofibrils Addition Ratio, Pressing Method, and Particle Size on the Bending Properties of Wet-formed Particleboard

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Wet-formed particleboard bonded with cellulose nanofibrils (CNF) was prepared in this work. The effects of density, CNF addition ratio, pressing method, and particle size on the bending strength were evaluated. The results showed that density had the most important effect on the modulus of elasticity (MOE), while the CNF addition ratio had the most important effect on the modulus of rupture (MOR). For panels with low density (< 640 kg/m^3), the MOE and MOR did not change much with the configuration changes between particle size and pressing method. This was due to the synergistic effect of incomplete compression and poor bonding in the core area using a constant thickness (CT) pressing method, and lower face density and higher core density using a constant pressure (CP) pressing method. For panels with medium density (640 kg/m^3 to 800 kg/m^3), the combination of larger particles, higher CNF addition ratio, and CT pressing method contributed to the highest bending strength. Further increase to high density (> 800 kg/m^3), the pressing method's effect was more important, compared to panels with low and medium densities. With increased density and CNF addition ratio, panels were able to meet low-density and some medium-density standard MOE and MOR requirements.

Keywords: Cellulose nanofibrils; CNF; Particleboard; Binderless; Modulus of elasticity; Modulus of rupture

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INTRODUCTION

Particleboard has been widely used in the furniture, cabinets, wall, and ceiling panel industries for several decades (Wang and Sun 2002). In 2014, the particleboard production was 111 M m^3 worldwide. Most commercial particleboard is produced using the dry-forming process, bonded with adhesives, such as urea formaldehyde (UF) resin, phenol formaldehyde (PF) resin (Ayrilmis *et al.* 2012), and even with diphenylmethane diisocyanate (MDI) resin in special conditions (Kojima *et al.* 2009). However, formaldehyde emission has been an ever-increasing health issue. Research has been conducted to develop environmentally friendly adhesives, such as protein-based or soybean-added adhesives (Lorenz *et al.* 1999; Gao *et al.* 2012). Cellulose nanofibrils (CNF) have shown potential as a performance enhancement additive for numerous applications in a wide variety of industries (Carpenter *et al.* 2015). Many composites with CNF addition have shown improved performance characteristics or properties. Preliminary results of small hand-formed panels have shown potential using CNF as the binding agent for the production of particleboard and have been proven feasible (Tajvidi *et al.* 2016).

CNF is defibrillated cellulose fibers with a width in the nano-meter range (Zhang *et al.* 2013). They are mainly obtained through a mechanical treatment, but the production often benefits from a chemical pretreatment. The most popular agent for chemically derived cellulose nano-fiber is called the 2,2,6,6-tetramethylpiperidine-1-oxyl radical (TEMPO), and it is used to prepare oxidized CNF (Saito *et al.* 2007). Mechanically derived CNF is made by repeated refining and a large pressure drop with shearing and impact forces (Nakagaito and Yano 2004). The CNF can be used in drug release and energy storage applications, due to its nanostructure (Dima *et al.* 2016; Pan *et al.* 2016). It can also be mixed with other materials and made into composites, due to its strong mechanical properties and web-like network that can improve the interfacial adhesion (Shao *et al.* 2015; Barari *et al.* 2016a; Barari *et al.* 2016b). The CNF could be a great candidate to produce binderless particleboard. Materials with a high content of lignin, starch, and sugar were deemed favorable to produce binderless boards (Boon *et al.* 2013). Only a few researchers have investigated the production of binderless boards (Baskaran *et al.* 2012; Hashim *et al.* 2012; Boon *et al.* 2013; Euring *et al.* 2016; Tajvidi *et al.* 2016), however, only through the dry-form process. Little research has been conducted using the wet-forming process (Arévalo and Peijs 2016). The wet-forming process could minimize fines and maximize fiber-to-fiber bonding compared to dry-form processes (Hunt and Supan 2005).

The bending (flexural) strength of wood-based panels is one of the most important mechanical properties (Li *et al.* 2013). The bending strength of the specimen depends on the compression/tensile sides (faces), but not much on shear unless the specimen is too short (Hayashi *et al.* 2003; Kowaluk *et al.* 2016). The bending properties of particleboard are influenced by many factors, of which density is one of the most influential. Higher density is favorable to achieve a higher bending strength, because higher density means that there are more particles and a more consolidated panel structure (Sari *et al.* 2013). Material density, while not the mean density, can be correlated with the modulus of elasticity (Wong *et al.* 2003). For panels with the same mean density, the U-shaped density profile results in a higher bending strength, due to higher peak density in the face regions (Cai *et al.* 2004; Nemli and Demirel 2007). The particle size is another important parameter to influence the bending strength. Fine particles have been shown to decrease bending strength (Tabarsa *et al.* 2011), while large particles were shown to achieve higher bending strength (Arabi *et al.* 2011; Rofii *et al.* 2013). Larger particles provide a lower surface area to volume ratio compared to small particles, which results in a higher surface concentration of the binding agent per unit area (Iskanderani 2008; Biswas *et al.* 2011). Larger particles could also reduce the number of defects, as the defects act as initial failure sites (Iskanderani 2008). Research suggests that wood particles preferentially would be large enough to form overlaps sufficient to transfer internal stress from one particle to another (Paridah *et al.* 2010) or an intermediate material to bridge the gaps. It is the assumption that CNF will aid in the intermediate gaps to help transfer load throughout the board.

In this study, mechanically derived wet-slurry of CNF with a solids content of 3% was mixed with dry wood particles to form binderless particleboard panels. The effects of panel density, CNF addition ratio, pressing method, and wood particle size were investigated on the bending properties of the binderless particleboard. Properties of these boards were compared with minimum American National Standards Institute (ANSI) particleboard standards.

EXPERIMENTAL

Materials

The wood particles used in this study consisted of an 80:20 ratio of softwood: hardwood (Timber Products Company, Sutter Creek, USA). The wood particles were screened through a 1.59 mm and a 0.79 mm opening dry vibrating screen to obtain three size fractions. Each size fraction was then collected and the weight percentages were determined, as shown in Table 1. The bulk density of each fraction was also determined. Particles from individual size fractions were spread out on a commercial white printer paper sheet, and photos were taken. Further analyses of the particles using Fiji image analysis software (LOCI, 1st version, Madison, USA) was used to determine the estimated particle surface area. Mechanically derived CNF at 3% solids content was provided by the University of Maine's Process Development Center (Orono, USA). The CNF was stored in the cold room at 2 °C before use.

Methods

Fabrication of CNF bonded particleboard

In this study, particleboard panels with the final dimensions of 305 mm × 305 mm × 12.7 mm were made. The effects of panel density, CNF addition ratio, pressing method, and particle size on the board's bending properties were investigated. Three replicates of each treatment were produced. Wood particles and CNF were mixed uniformly with a Hobart laboratory blender (The Hobart MFG, Co. Troy, USA). The moisture content (MC) of the wood particle-CNF mixture was 329%, 490%, and 650% (dry basis) after mixing, depending on the CNF addition ratio of 10%, 15%, and 20%, respectively. The mixture was blended for 15 min. The CNF was observed to be very uniformly distributed throughout the mixture. The mixture was evenly distributed in a 305-mm square forming box, as shown in Fig. 1. A flat plate was placed on top of the distributed mixture and approximately 65 N/m^2 to 75 N/m^2 vacuum pressure was used to pre-compress the semi-wet mat. The initial vacuum pressure applied to the mixture was able to remove a portion of the free water. After vacuum pressing for all the particle sizes, the MC for the 10% CNF/particle mixture ranged from 231% to 281%; the MC for the 15% CNF/particle mixture for all particle sizes ranged from 223% to 261%; and the MC for the 20% CNF/particle mixture for all particle sizes ranged from 276% to 313% (dry basis). The pre-compressed mats were then transferred onto a screen on top of an aluminum caul. A 305 mm square aluminum frame was carefully placed around the mat to prevent the mat from extruding sideways during hot pressing. A second screen was placed between the top of the mat, and two 25.4-mm thick aluminum plates were placed inside the frame and on top of the wet-mat. The total package was placed into a hot-press (Williams White Co., Moline, USA). The platens were steam heated and set at 185 °C. Two separate press schedules were evaluated for the drying process: constant pressure (CP) or constant thickness (CT), as shown in Table 2. Typical CT and CP schedules are shown in Fig. 3. High pressure occurred initially as the wet mat was consolidated and dewatered as the heat transferred into the mat. For all of the panels, the excess water was pressed out of the mixture as the press closed on the CNF/particle mixture. The panels were pressed to near oven-dry conditions before the panels were removed. For testing, the finished panels were cut according the standard test dimensions where possible and then were conditioned in a 20 °C and 65% relative humidity (RH) conditioning room.

Table 1. Wood Particle Characterization

Wood Particle	Large Particles (> 1.59 mm Opening)	1.59 mm opening > Small Particles > 0.79 mm Opening	Fine Particles (< 0.79 mm Opening)
Weight Percentage (%)	18	41	41
Bulk Density (kg/m^3)	168	202	226
Surface Area (m^2/g)	N/A	0.2	8.08

Table 2. Experimental Design for the Production of Panels

ID	Particle Size*	CNF Addition Ratio (%)	Target Density (actual) (kg/m^3)	Pressing Method**
1	s	10	600 (568)	CT
2	s	10	750 (705)	CP 0.41 MPa
3	s	10	900 (830)	CP 0.55 MPa
4	s	15	600 (636)	CP 0.41 MPa
5	s	15	750 (752)	CP 0.55 MPa
6	s	15	900 (846)	CT
7	s	20	600 (703)	CP 0.55 MPa
8	s	20	750 (709)	CT
9	s	20	900 (816)	CP 0.41 MPa
10	f	10	600 (640)	CP 0.41 MPa
11	f	10	750 (769)	CP 0.55 MPa
12	f	10	900 (849)	CT
13	f	15	600 (709)	CP 0.55 MPa
14	f	15	750 (710)	CT
15	f	15	900 (821)	CP 0.41 MPa
16	f	20	600 (586)	CT
17	f	20	750 (738)	CP 0.41 MPa
18	f	20	900 (855)	CP 0.55 MPa
19	m	10	600 (629)	CP 0.55 MPa
20	m	10	750 (695)	CT
21	m	10	900 (813)	CP 0.41 MPa
22	m	15	600 (580)	CT
23	m	15	750 (714)	CP 0.41 MPa
24	m	15	900 (849)	CP 0.55 MPa
25	m	20	600 (692)	CP 0.41 MPa
26	m	20	750 (774)	CP 0.55 MPa
27	m	20	900 (844)	CT
*s = small size particles; f = fine size particles; m = mixed size particles ** CP = constant pressure pressing method; CT = constant thickness pressing method				

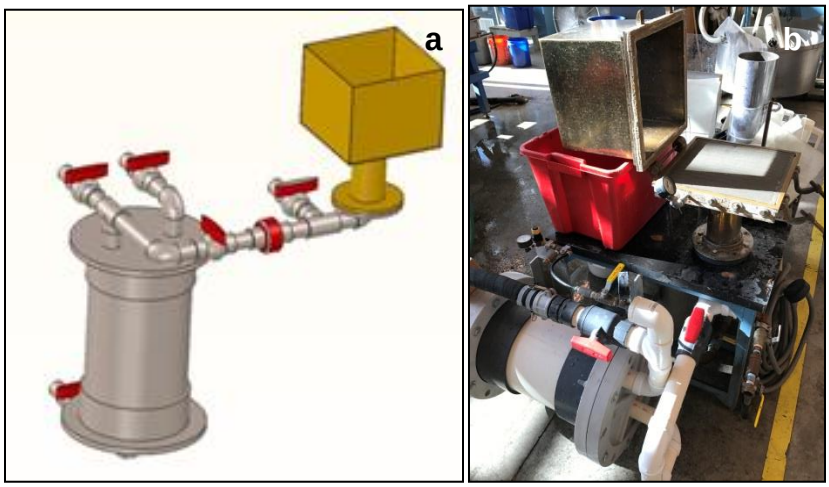

Fig. 1. Mat forming and vacuum system a) diagram; b) actual system

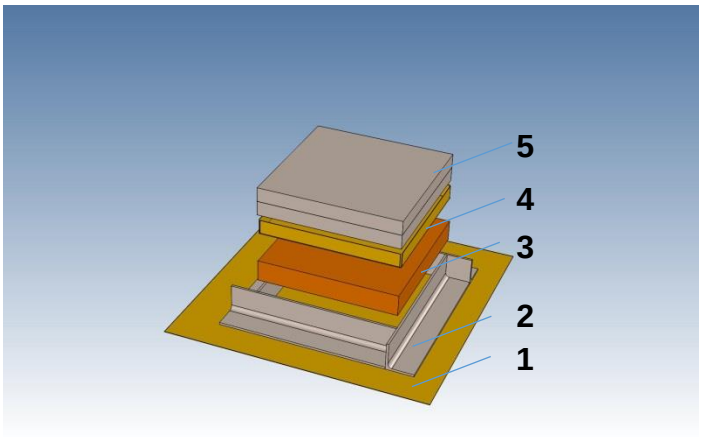

Fig. 2. Diagram of mat hot-pressing setup: 1) bottom caul and screen, 2) aluminum frame, 3) fiber mat, 4) top screen, and 5) two aluminum blocks

Bending test

The bending test was conducted on the 50 kN Instron machine (Norwood, USA), according to ASTM D1037-12 (2012). Typical dimensions for the specimens were 279.4 mm × 76.2 mm × 12.7 mm. While thickness was controlled as best as it possibly could throughout the study; slight differences in the final thickness were measured and accounted for in all of the calculations.

Two specimens were cut from each panel. For the three-point bending test, the span was set to 254 mm, with a cross-head rate of 6 mm/min. The bending test was conducted in the same conditioning room maintained at 20 °C and 65% RH. The testing results were analyzed using an analysis of variance (ANOVA) (SAS, 9.2, Cary, USA). Significant differences of the four main effects and two-way interaction effects were determined at a 5% level. A regression analysis was also performed to demonstrate the relation between the main effects and the bending properties.

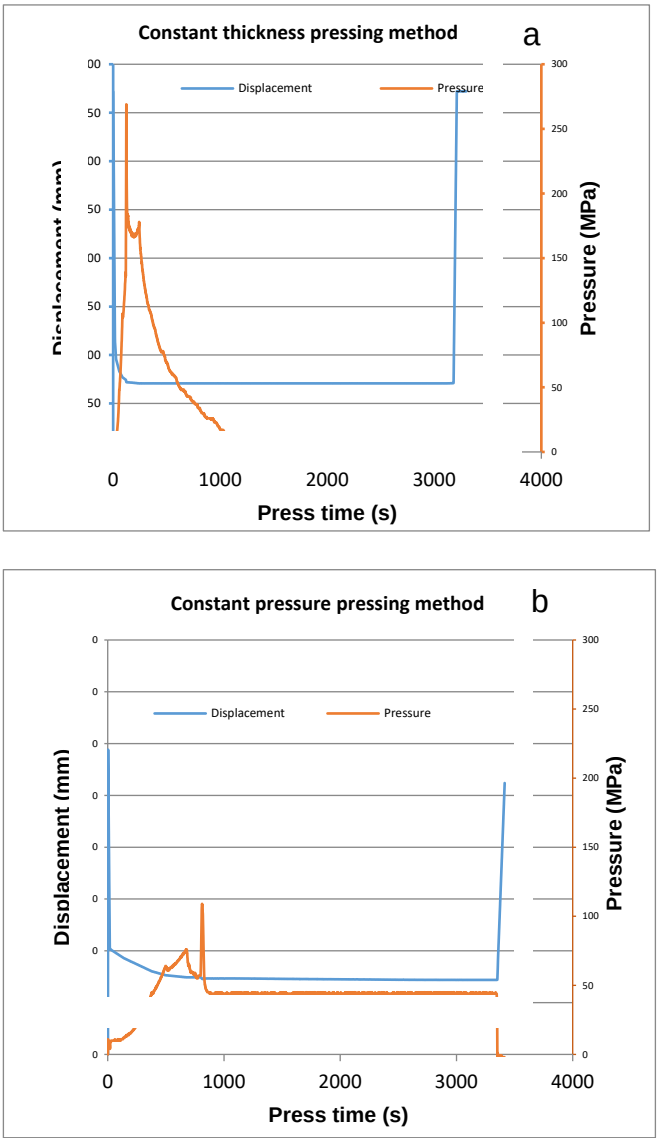

Fig. 3. Typical pressure and displacement curves for two pressing methods: a) constant thickness pressing method; b) constant pressure pressing method

RESULTS AND DISCUSSION

All of the panels produced were placed in their respective sets as expected based on the factorial experimental design, except for panels group No.7, 13, and 25, which were supposed to be in the low-density group (< 640 kg/m^3), but fell into the medium-density group (640 kg/m^3 to 800 kg/m^3). This was attributed to the fact that the constant pressure program yielded a slightly higher density than expected.

It may have been expected that the increased surface area of the smaller fine wood particles (Table 1) may provide additional hydroxyl groups for the water in the CNF to access, and then to provide a release of the higher MC of the CNF. It may also have been possible to decrease the moisture content of the mixture before hot-pressing if the pre-press used higher pressures than the vacuum pressure used in this study. In this study, there were no significant pre-press MC differences between the particle sizes from the pressure

applied by the vacuum. However, the vacuum application still provided huge reductions of MC, from 58% to 375% MC (dry-basis), dependent upon the CNF addition ratio. In addition, it was evident in all of the hot-pressing conditions that the initial consolidation step from the high press pressure yielded a large amount of liquid moisture loss out of the screens. It was possible that higher pressures in the pre-press would have yielded even lower moisture contents and, thus, less water that would have been lost to steam and therefore lower energy costs. A cold or warm pre-press allowed for slower consolidation rates, which allowed more moisture to be pressed out over a longer time span without the concern for pre-drying of the surface layers. The mats were hot-pressed until either the thickness remained essentially constant for the CP panels or the hydraulic pressing pressure remained essentially constant for the CT panels, as shown in Fig. 3.

Figures 4 and 5 show the overall relationship between the bending properties and panel density. An analysis of variance (Table 3) showed that particle size, CNF addition ratio, density, and pressing method all had significant effects on the bending properties. It was quite evident that as density increased, the MOE and MOR increased, and as the CNF addition ratio increased, the MOE and MOR also increased. It could be seen that increased density produced higher MOE and MOR, and that the increases were not necessarily a linear relationship. For each panel set, a pre-determined amount of particles and CNF were mixed. Density was manipulated based on the desired range of the density values and an estimated calculated volume for the wood particles with CNF. It was assumed that the finer the particles, the higher the packing density and increased bonding between the materials. Apart from the ANOVA results, it was not evident from the plotted data that there were substantial differences based on the design of the experiment (DOE) for this study. The CT pressing method showed better bending performance with higher CNF addition ratios (15% and 20%), in comparison with the CP pressing method (Fig. 5). The increase for the CT panels was primarily due to the higher faces' density. The CT panels produced a U-shaped density profile with higher density on the surfaces than the core. By contrast, the CP press method had a more uniform density profile through the thickness of the panel. In the literature, most particleboard density profiles show a U-shaped profile. This profile had a significant effect on bending properties and was one of the most influential factors affecting the bending strength (Wilczyński and Kociszewski 2007). A more complete analysis of the density profile from this study will be discussed in a subsequent paper currently in preparation. In addition, there were significant two-factor interactions between the effects of particle size and density ($p = 0.0031$), particle size and pressing method ($p = 0.0017$), and CNF addition ratio and density ($p = 0.0081$) on the MOE. There were also significant interactions between the effects of particle size and pressing method ($p = 0.0375$), CNF addition ratio and density ($p = 0.0111$), and density and pressing method ($p = 0.0260$) on MOR. The results from the ANOVA confirmed that there were no significant interactions between the effects of CNF addition ratio and particle size, and between the effects of CNF addition ratio and the pressing method. There was not much difference for the effects of particle size and pressing method on the bending strength. Three density groups (Table 2) were used to try to help define the effects of particle size and pressing method on the bending properties. It is well known that finer particles and higher face density are used to improve bending properties (Wong *et al.* 2003; Cai *et al.* 2004; Arabi *et al.* 2011; Tabarsa *et al.* 2011). However, the MOE and MOR did not change much with the change between particle size and pressing method for panels with a low CNF addition ratio. For panels with only 10% CNF, there was no significant difference between the pressing methods or particle sizes used to make the panels. The reason might have been that the density was

low, such that the bonding between wood particles and CNF was weaker. Any potential advantages of particle size and pressing method were not achieved for panels with a low CNF addition ratio. As the CNF addition ratio increased, the CT panels outperformed the CP panels, especially at the lower average density ranges.

In addition, the panels pressed with constant 0.55 MPa pressure generated a higher MOE and MOR than those pressed with constant 0.41 MPa pressure. A higher hot-press pressure resulted in slightly higher density and subsequent higher interfiber bonding, as evident in the increased density for all 0.55 MPa pressure panels. The CP data showed that when the MOE or MOR was plotted against density, the MOE and MOR curves followed similar curves that were essentially the same, regardless of how the panel might have been produced with increased material or with pressure. It was possible, if the pressure difference had been greater, there might have been a greater difference in properties.

Table 3. ANOVA for MOE and MOR

Source	F		p-value	
	MOE	**MOR**	**MOE**	**MOR**
Density	369	360	<.0001	<.0001
CNF Addition Ratio	289	527	<.0001	<.0001
Particle Size	8.92	5.66	0.0004	0.0055
Pressing Method	6.88	7.10	0.0020	0.0017
CNF Addition Ratio*Density	7.49	6.86	0.0081	0.0111
Particle Size*Density	6.36	2.90	0.0031	0.0624
Particle Size*Pressing Method	4.91	2.72	0.0017	0.0375
Density*Pressing Method	2.96	3.88	0.0593	0.0260
Particle Size*CNF Addition Ratio	0.69	0.33	0.5054	0.7234
CNF Addition Ratio* Pressing Method	0.36	0.012	0.6982	0.9882

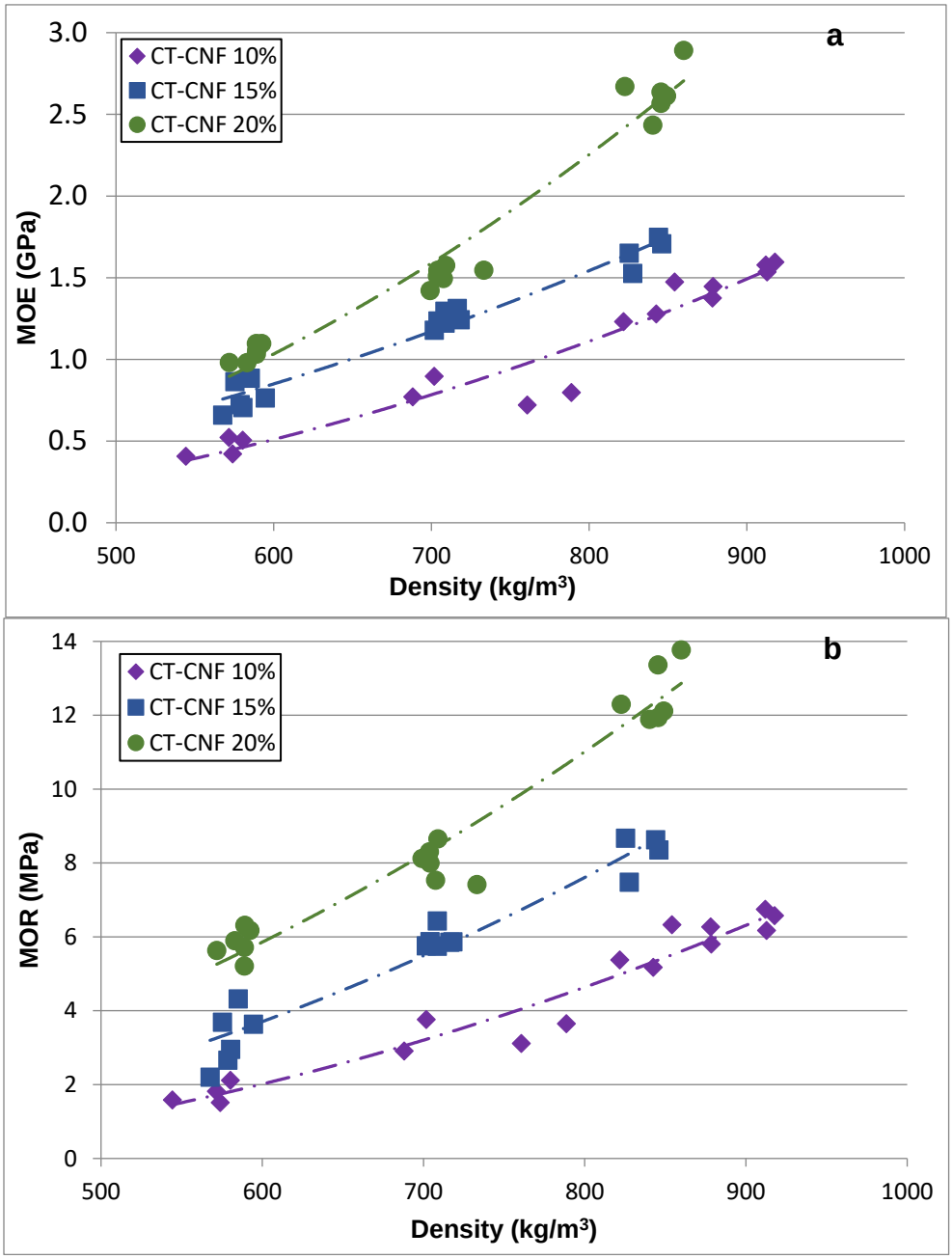

Fig. 4. MOE a) and MOR b) as a function of CNF addition ratio using CT pressing method

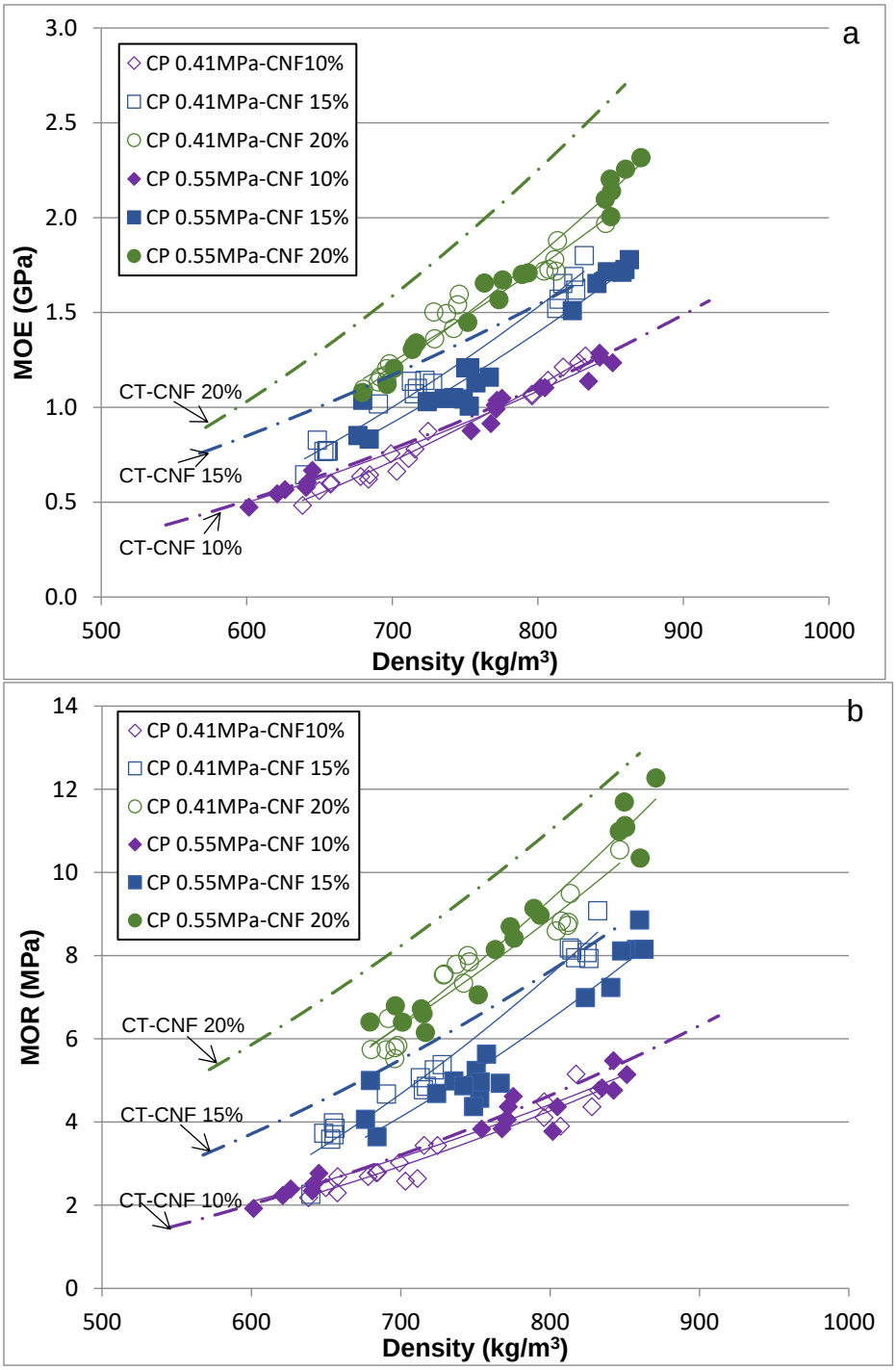

Fig. 5. MOE a) and MOR b) as a function of CNF addition ratio using 0.41 MPa and 0.55 MPa CP pressing method; dotted lines show respective constant thickness properties as a function of CNF addition ratio in comparison with the CP panels

Figure 6 shows the performance data of the MOE and MOR for increased CNF addition ratio and the comparison between the two pressing methods in relationship with ANSI A208.1 (2016) standards for particleboard. It shows that boards can be made that meet some low-, medium-, and high-density standards for several particleboard performance levels for MOE and MOR. When these plots were extrapolated, increased

CNF addition ratio and density levels would provide improved performance to meet or exceed higher standards. It was also clear that the CT pressing method provided improved bending performance, especially at higher CNF addition ratios, as compared with the CP pressing method.

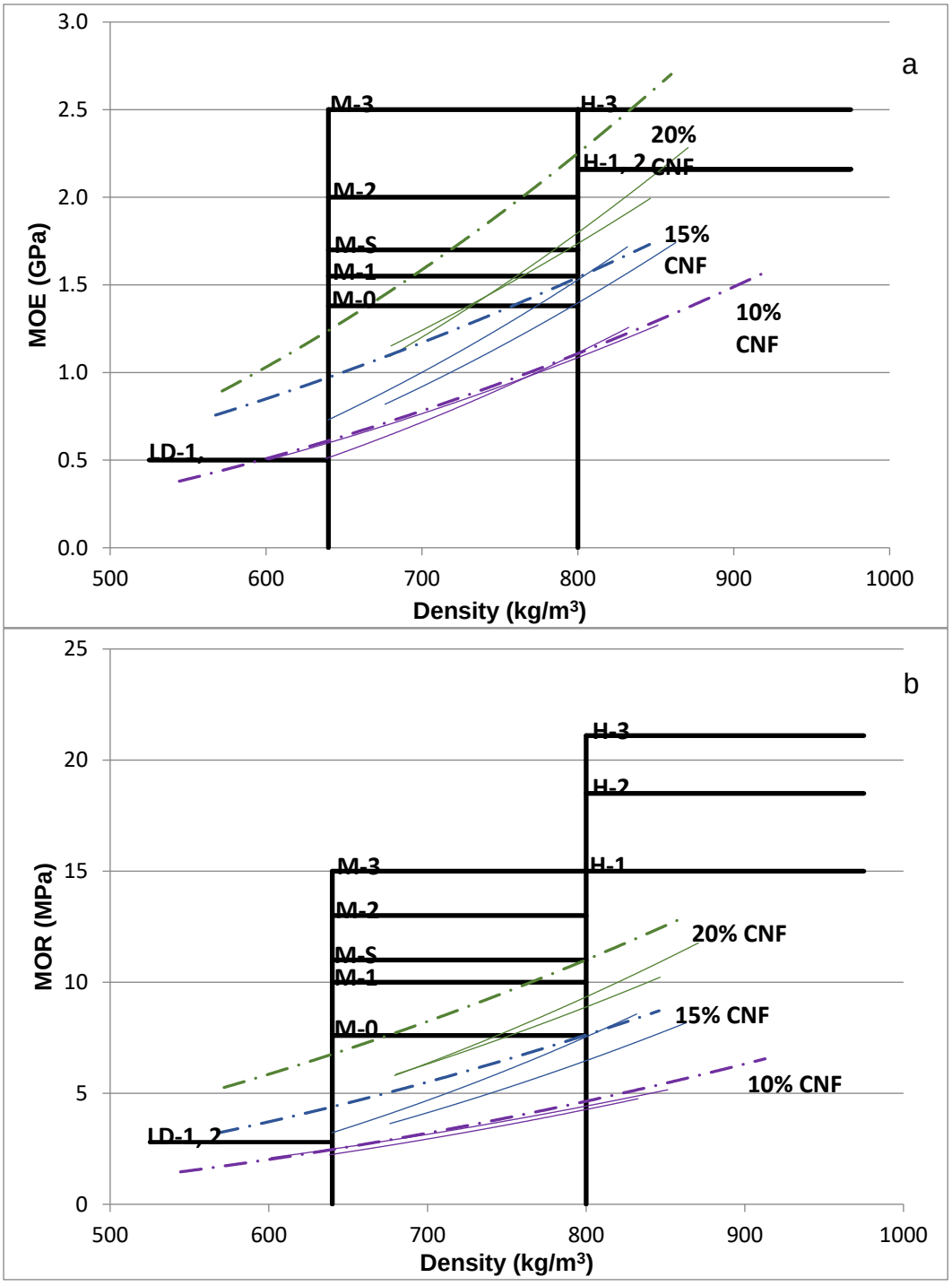

Fig. 6. MOE a) and MOR b) plots for CP (solid) and CT (dashed) pressing methods with increasing CNF addition ratio in relationship with ANSI 208.1 standard (2016)

CONCLUSIONS

1. Both density and CNF addition ratio had the most significant positive effects on MOE and MOR. The pressing method also significantly affected the bending properties.
2. The plots showed increased degree of differences as the CNF addition ratio increased, especially at lower densities and at 20% CNF addition ratio.
3. Preliminary data indicated there was a U-shaped density profile developed using the CT pressing method, which influenced the bending properties, due to higher densities on the outer faces.
4. Slight effects were observed for the MOE and MOR with changes between particle sizes. While panels pressed with a higher CP of 0.55 MPa showed increased properties at higher densities, these still followed similar plot lines as a function of density within the same CNF addition ratio.
5. Further increase to higher densities and higher CNF addition ratios will help meet the standard MOE and MOR requirements, according to ANSI A208.1 (2016).

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