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ORIGINAL PAPER

Preparation and Evaluation of Particleboard with a Soy Flour-Polyethylenimine-Maleic Anhydride Adhesive

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Abstract A soy-based formaldehyde-free adhesive consisting of soy flour (SF), polyethylenimine (PEI), maleic anhydride (MA) and NaOH was investigated for making three-layer particleboard. The weight ratio of SF/PEI/MA/NaOH was 7/1.0/0.32/0.1. Hot-press temperature, hot-press time, particleboard density and adhesive usage were optimized in terms of enhancing the modulus of rupture (MOR), modulus of elasticity (MOE) and internal bond strength (IB) of the resulting particleboard. The MOR, MOE and IB met the minimum industrial requirements of M-2 particleboard under the following variables: hot-press temperature of 170 °C, hot-press time of 270 s, the adhesive usage of surface particles at 10 wt%, the adhesive usage of the core particles at 8 wt%, and the targeted particleboard density of 0.80 g/cm^3.

Keywords Wood adhesive · Particleboard · Soy flour · Polyethylenimine

Introduction

Particleboard is mainly composed of wood particles and an adhesive. The most commonly used particleboard has three layers: two face layers and one core layer. The face layers consist of fine particles, and the core layer is made of coarse particles. Wood particles are first coated with an adhesive, and then formed into a mat that is further hot-pressed to form a panel product [1]. At present, urea–formaldehyde (UF) resin is the most commonly used adhesive for making particleboard. Formaldehyde is considered to be carcinogenic to human [2]. Formaldehyde is emitted in the production and use of particleboard bonded with UF resins. California Air Resource Board has passed a tough regulation on limiting formaldehyde emission from wood composite panel products sold and used in California. The phase II standard for particleboard will take effect on 1 January 2011. Particleboard bonded with the currently used UF resins may not be able to meet the phase II standard. Therefore, there is an urgent need for development of a commercially viable formaldehyde-free adhesive for making particleboard.

Soy flour is abundant, renewable, and inexpensive. The soy-based adhesive was widely used for the commercial production of plywood from the 1930s to the 1960s and then replaced by synthetic resins such as UF resins because of the low water-resistance of plywood panels bonded with the soy-based adhesive [3]. There has been a renewed interest in recent years for the development of water-resistant soy-based wood adhesives. Through mimicking mussel adhesive protein, several formaldehyde-free soy-based adhesives have been developed and one of the soy-based adhesive has been used to replace UF resins for the commercial production of plywood since 2004 [4–6]. One of the newly developed formaldehyde-free adhesives consists of soy flour (SF), polyethylenimine (PEI), and maleic anhydride (MA). This SF–PEI–MA adhesive was successfully used for preparation of type II plywood [7]. This adhesive was investigated in this study to see if it could be used for production of particleboard.

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Materials and Methods

Materials

SF (7% moisture content, 100 mesh, and 90 protein dispersability index) was provided by Cargill Incorporated (Minneapolis, MN, USA); a 50 wt% aqueous PEI solution (Mw = 750,000) and MA were purchased from Sigma–Aldrich (Milwaukee, WI, USA). Douglas fir wood particles (surface and core wood particles) were obtained from Flakeboard (Albany, OR).

Preparation of SF-Coated Wood Particles

The following is a representative procedure for the preparation of SF-coated wood particles. For coating surface wood particles, SF (175 g, dry weight) and water (696 g) were sequentially added into a HOBART A-200 blender (Hobart, Topeka, KS) and stirred for 10 min at room temperature to form 20% SF slurry. Surface wood particles (1,403 g, dry weight) were then added into the SF slurry in the HOBART blender and the resulting mixture was stirred for 10 min. The resulting wet SF-coated wood particles were put in a cloth bag, and dried in a rotary dryer (Speedqueen, Ripon, WI) for 1 h. After drying, the SF-coated surface wood particles had the moisture content of 2%. SF-coated core wood particles (2% moisture content) was prepared by following the same procedure as for the preparation of the SF-coated surface wood particles using SF (245 g dry weight), water (898.9 g), and core wood particles (2,456 g, dry weight).

Preparation of PEI–MA–NaOH Solution

The following was a representative procedure for the preparation of PEI–MA–NaOH solution. MA (19.2 g) and water (100 g) were put into a 400-mL beaker, and stirred with a magnetic stirrer for 30 min. The resulting MA solution (50 g), the PEI solution (50 g), and 1 N NaOH (65 g) were placed in a KitchenAid mixer, and stirred for 6 min. The resulting solution was then sprayed onto the oven-dried SF-coated surface wood particles (1,578 g, dry weight) in a rotary drum-blender. The resulting adhesive-coated surface wood particles had the adhesive usage of 15 wt% (the adhesive usage was defined as the dry weight of SF, PEI, MA and NaOH divided by the dry weight of the wood particles) and had the moisture content of 9%. A mixture of the PEI solution (70 g), the MA solution (69.2 g), and 1 N NaOH (90 g) was sprayed onto the oven-dried SF-coated core wood particles (2,701 g, dry weight) in the rotary drum-blender. The resulting adhesive-coated core wood particles had an adhesive usage of 12% and had a moisture content of 9%. Adhesive-coated wood particles with a different adhesive usage were prepared in accordance with the previous described procedures while the SF/PEI/MA/NaOH dry weight ratio was maintained at 7/1/0.32/0.1.

Preparation of Three-Layer Particleboard

The adhesive-coated surface wood particles (807 g, dry weight) were placed in a forming box (60.96 × 60.96 cm) by hand to form a uniform layer. The adhesive-coated core wood particles (2,748 g, dry weight) were evenly distributed on top of the surface wood particles layer, followed by a uniform layer of the adhesive-coated surface wood particles (807 g, dry weight). The resulting three-layered wood particle mat was hand-pressed with a flat plywood panel (60.96 × 60.96 cm) and then hot-pressed at 170 °C for 300 s. The target thickness was 17 mm. The hot-press time and hot-press temperature were changed when their effects on particleboard properties were investigated. Two particleboard panels were prepared for each experimental variable.

Evaluation of Mechanical Properties of Particleboard

Particleboard was cut into 7.62 × 46.99 cm rectangular specimens for the measurement of modulus of rupture (MOR) and modulus of elasticity (MOE), and into 5.08 × 5.08 cm specimens for the measurement of internal bond strength (IB) in accordance with ASTM D1037-99 (American Society for Testing and Materials, 1999) using a MTS Sintech 1/G testing machine (MTS Systems Corp., Eden Prairie, MN). The crosshead speeds were 9 mm/min for the measurement of MOR and MOE and 1.5 mm/min for the measurement of IB. For each particleboard panel, four specimens were obtained for the measurement of MOR and MOE, and eight specimens for the measurement of IB.

Statistical Analysis of Data

All data were analyzed with the Welch modified two-sample *t* test with a S-PLUS® statistical software package (Edition version 8.0, Insightful Corp., Seattle, WA, USA). All comparisons were based on a 95% confidence interval.

Results and Discussion

Effects of the hot-press temperature on the MOR and MOE of particleboard panels are shown in Fig. 1a. At all hot-press temperatures studied, the MOR exceeded the minimum industrial requirement of 14.5 MPa (horizontal solid line) for M-2 Grade particleboard. The average MOR value significantly increased by 23.7% when the hot-press

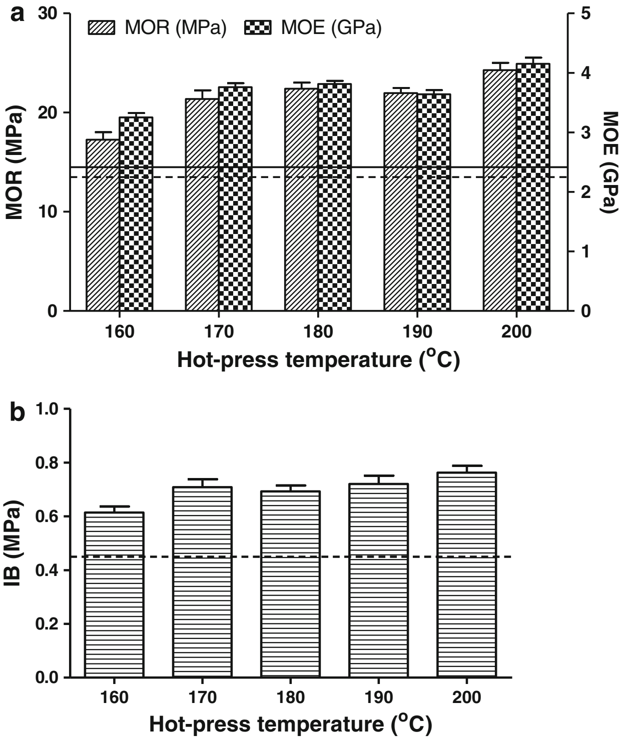

Fig. 1 **a** Effects of hot-press temperature on the MOR and MOE of particleboard. [surface adhesive usage (dry basis on wood particles), 15 wt%; core adhesive usage, 12 wt%; hot-press time, 300 s; the targeted particleboard density, 0.70 g/cm^3. Data are the means of eight replications, and the *error bar* represents one standard error of the mean]. **b** Effect of hot-press time on the IB of particleboard. (Processing parameters and statistical details are the same as those in **a**)

temperature was raised from 160 to 170 °C. In the range of 170–190 °C the MOR did not change significantly. The MOR at 200 °C was statistically higher than that at other hot-press temperatures except that at 180 °C. At all hot-press temperatures studied, the MOE exceeded the minimum industrial requirement of 2.25 GPa (horizontal dashed line) for M-2 Grade particleboard (Fig. 1a). The MOE significantly increased by 10.0% when hot-press temperature was raised from 160 to 170 °C. In the range of 170–190 °C the MOE did not significantly change. However, the average MOE at 200 °C was significantly higher than that at all other temperatures.

The effect of the hot-press temperature on the IB of particleboard is shown in Fig. 1b with the minimum industrial requirement (0.45 MPa) shown as a horizontal dashed line. The IB exceeded the minimum industrial requirement at all hot-press temperatures. The IB significantly increased by 14.6% when the temperature was raised from 160 to 170 °C. The IB values in the 170–200 °C range were not significantly different from each other. With the MOR, MOE and IB all being considered, 170 °C appeared to be the best hot-press temperature and was used for subsequent investigations.

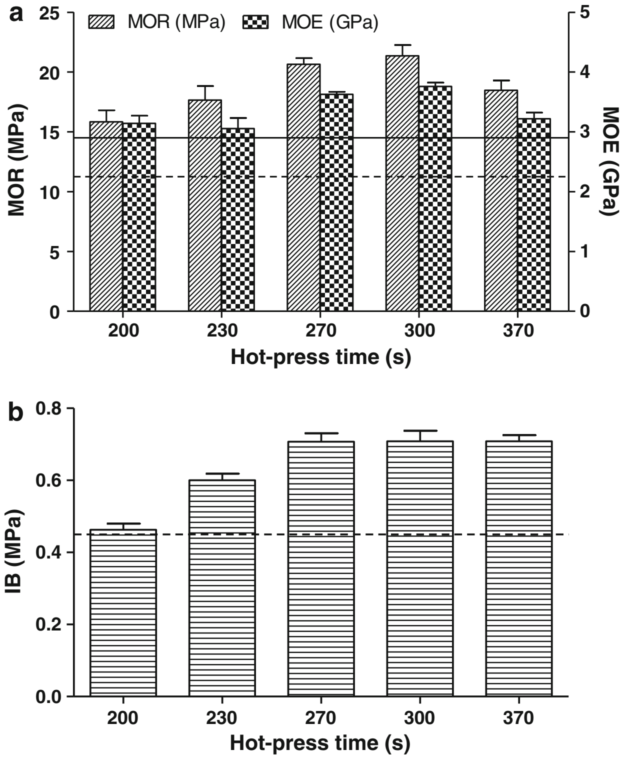

Fig. 2 **a** Effects of hot-press time on the MOR and MOE of particleboard. [surface adhesive usage (dry basis on wood particles), 15 wt%; core adhesive usage, 12 wt%; hot-press temperature, 170 °C; the targeted particleboard density, 0.70 g/cm^3. Data are the means of eight replications, and the *error bar* represents one standard error of the mean]. **b** Effect of hot-press time on the IB of particleboard. (Processing parameters and statistical details are the same as those in **a**)

Effects of hot-press time on the MOR and MOE of particleboard are shown in Fig. 2a. The MOR did not significantly change when the hot-press time was raised from 200 to 230 s. The MOR significantly increased when the hot-press time was raised from 230 to 270 s. Further increases in the hot-press time from 270 to 300 s did not significantly increase the MOR although the average MOR at 300 s was higher than that at 270 s. The MOR markedly decreased when the hot-press time was increased from 300 to 370 s. The MOR at all hot-press times tested met the minimum industrial requirement (horizontal solid line, Fig. 2a). The MOE remained the same when the hot-press time was increased from 200 to 230 s. However, the MOE significantly increased when the hot-press time was increased from 230 to 270 s. The MOE remained the same when the hot-press time was increased from 270 to 300 s, and then significantly decreased when the hot-press time was further increased from 300 to 370 s. The MOE at all hot-press temperature tested met the minimum industrial requirement (horizontal dashed line, Fig. 2a).

The IB significantly increased when the hot-press time was increased from 200 to 270 s (Fig. 2b). The IB remained the same when the hot-press time was in the range of 270 to 370 s. The IB at all hot-press temperature tested met the minimum industrial requirement (Fig. 2b). The 270 s was used as the optimum hot-press time for subsequent evaluation.

The effect of the core adhesive usage on the MOR of particleboard panels made at two different density levels is shown in Fig. 3a. At the high density level (0.80 g/cm^3), the MOR remained the same when the core adhesive usage was reduced from 11 to 10 wt% and then significantly increased when the core adhesive usage was further reduced from 10 to 9 wt% (Fig. 3a). At the high density level, the MOR rapidly decreased when the adhesive usage was reduced from 9 to 7 wt%. The MOR at all adhesive usages tested exceeded the minimum industrial requirement (horizontal solid line, Fig. 3a). At the low density level (0.70 g/cm^3), the MOR was statistically the same when the adhesive usage was reduced from 11 to 8 wt%. The MOR at the core adhesive usage of 9 wt% was significantly higher than that at 7 wt%, and the MOR at 8 wt% was statistically the same as that at 7 wt%. The MOR at the adhesive usage of 11 to 8 wt% met the minimum industrial requirement while the MOR at 7 wt% was very close to meeting the requirement (horizontal solid line, Fig. 3a). At the same adhesive usage, the MOR at the low density level was significantly lower than that at the high density level.

The effect of the core adhesive usage on the MOE of particleboard panels made at two different density levels is shown in Fig. 3b. At the high density level (0.80 g/cm^3), the MOE remained the same when the adhesive usage was reduced from 11 to 9 wt% and then significantly decreased when the adhesive usage was further reduced from 9 to 7 wt%. All MOE values at the high density level exceeded the minimum industrial requirement (horizontal solid line, Fig. 3b). At the low density level (0.70 g/cm^3), the MOE significantly increased when the core adhesive usage was reduced from 11 to 10 wt% and then significantly decreased when the adhesive usage was reduced from 10 to 8 wt%. The MOE somehow significantly increased when the adhesive usage was further reduced from 8 to 7 wt%. All MOE at this density level exceeded the minimum industrial requirement (horizontal solid line, Fig. 3b). At each adhesive usage tested, the MOE at the high density level was always higher than that at the low density level (Fig. 3b).

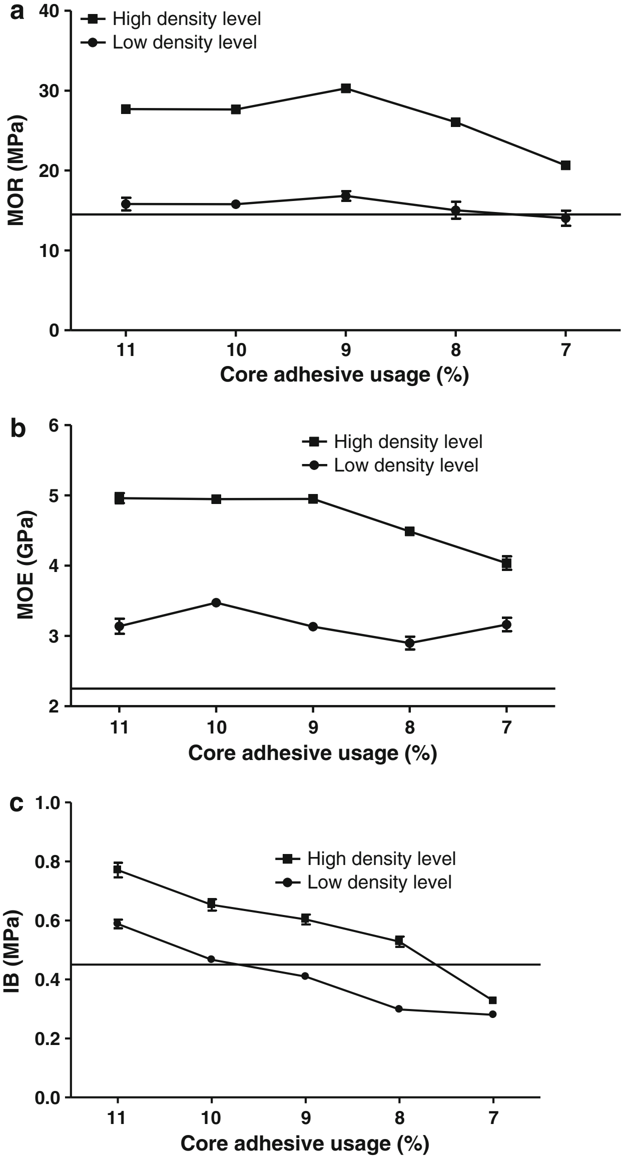

Fig. 3 **a** Effect of the core adhesive usage on the MOR at a high density level and a low density level. [surface adhesive usage (dry basis on wood particles), 15 wt%; hot-press temperature 170 °C; hot-press time, 270 s. Data are the means of eight replications, and the *error bar* represents one standard error of the mean]. **b** Effect of the core adhesive usage on the MOE at a high density level and a low density level. (Processing parameters and statistical details are the same as those in **a**). **c** Effect of the core adhesive usage on the MOE at a high density level and a low density level. (Processing parameters and statistical details are the same as those in **a**)

The effect of the core adhesive usage on the IB of the particleboards made at two different density levels is shown in Fig. 3c. At the high density level (0.80 g/cm^3), the IB decreased significantly when the core adhesive usage was reduced from 11 to 10 wt%, but remained statistically the same when the adhesive usage was reduced from 10 to 9 wt%. Further reduction of the adhesive usage from 9 to 8 wt% or from 8 to 7 wt% significantly decreased the IB. The IB at all adhesive usages except 7 wt% met the minimum industrial requirement (horizontal solid line, Fig. 3c). At the low density level (0.70 g/cm^3), the IB significantly decreased when the adhesive usage was reduced from 11 to 8 wt% and then remained the same when the adhesive usage was further reduced from 8 to 7 wt%. The IB met the minimum industrial requirement when the adhesive usage was 10 wt% or higher (horizontal solid line, Fig. 3c). At the same adhesive usage, the IB at the high density level was significantly higher than that at the low density level.

Effects of surface adhesive usage on the MOR and MOE of particleboard panels are shown in Fig. 4a. The MOR significantly decreased when the adhesive usage was reduced from 15 to 13 wt% and then remained the same when the adhesive usage was further reduced from 13 to 12 wt%. The MOR significantly decreased again when the adhesive usage was further reduced from 12 to 11 wt%. The MOR at all surface adhesive usages tested exceeded the minimum industrial requirement (horizontal solid line, Fig. 4a). The MOE did not significantly change when the surface adhesive usage was reduced from 15 to 14 wt%, and then significantly decreased when the adhesive usage was further reduced from 14 to 13 wt%. The MOE remained the same when the adhesive usage was reduced from 13 to 12 wt% and then significantly decreased when the adhesive usage was further reduced from 12 to 11 wt% (Fig. 4a). The MOE at all adhesive usages tested met the minimum industrial requirement (horizontal dashed line, Fig. 4a).

The effect of the surface adhesive usage on the IB of particleboard panels is shown in Fig. 4b. The IB significantly decreased when the adhesive usage was reduced from 15 to 13 wt%. The IB at 13 wt% was somehow significantly lower than that at 12 wt%. The IB remained the same when the adhesive usage was reduced from 12 to 11 wt%. The IB met the minimum industrial requirement (horizontal dashed line, Fig. 4b) when the adhesive usage was at 14 wt% or higher.

The effects of the moisture content of the surface wood particles on the MOR and MOE are shown in Fig. 5a. The MOR significantly increased when the moisture content was raised from 7.1 to 8.6 wt% and then remained the same when the moisture content was further raised from 8.6 to 9.0 wt%. However, the MOR significantly increased when the moisture content was raised from 9.0 to 9.4 wt%. The MOR at all moisture contents tested met the minimum industrial requirement (horizontal solid line, Fig. 5a). The MOE did not significantly change when the moisture content was raised from 7.1 to 9.0 wt% and then significantly increased when the moisture content was further raised from 9.0 to 9.4 wt%. All MOE at the moisture

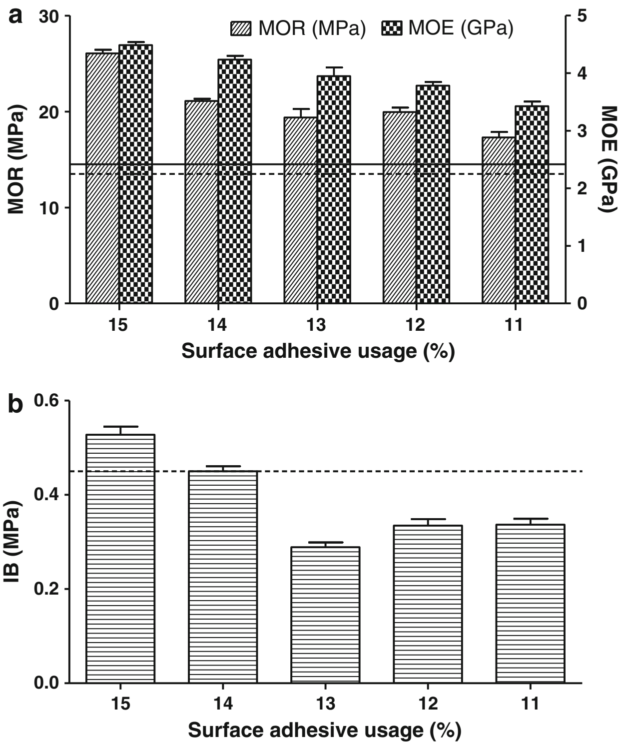

Fig. 4 **a** Effects of the surface adhesive usage on the MOR and MOE of particleboard. [core adhesive usage (dry basis on wood particles), 8 wt%; hot-press temperature, 170 °C; hot-press time, 270 s; the targeted particleboard density, 0.80 g/cm^3. Data are the means of eight replications, and the *error bar* represents one standard error of the mean]. **b** Effect of surface adhesive usage on the IB of particleboard. (Processing parameters and statistical details are the same as those in **a**)

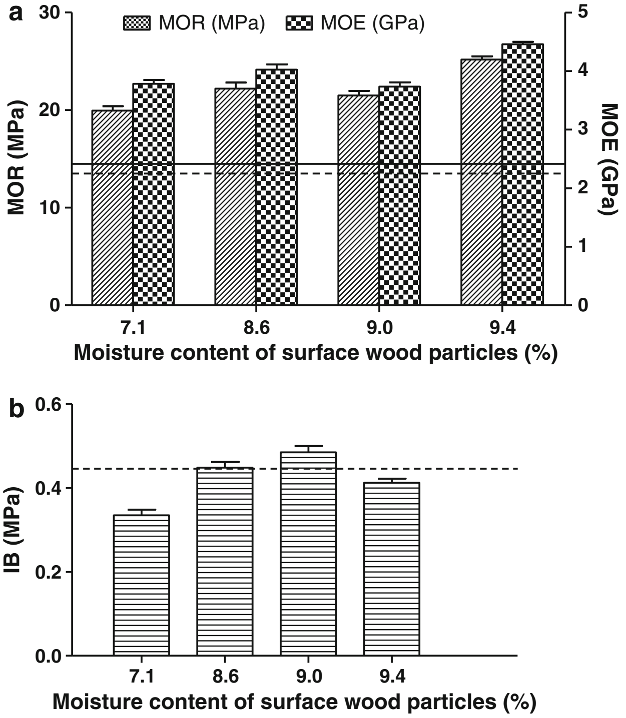

Fig. 5 **a** Effects of the moisture content of the surface wood particles on the MOR and MOE of particleboard. [surface adhesive usage (dry basis on wood particles), 10 wt%; core adhesive usage (dry basis on wood particles), 8 wt%; hot-press temperature, 170 °C; hot-press time, 270 s; the targeted particleboard density, 0.80 g/cm^3. Data are the means of eight replications, and the *error bar* represents one standard error of the mean]. **b** Effect of the moisture content of the surface wood particles on the IB of particleboard. (Processing parameters and statistical details are the same as those in **a**)

contents tested met the minimum industrial requirement (horizontal solid line, Fig. 5a).

The effect of the moisture content of the surface wood particles on the IB is showed in Fig. 5b. The IB significantly increased when the moisture content was increased from 7.1 to 8.6 wt% and then remained statistically the same when the moisture content was further raised from 8.6 to 9.0 wt%. However, the IB was reduced significantly when the moisture content was further increased from 9.0 to 9.4 wt%. The IB met the minimum industrial requirement when the moisture content was in the range of 8.6 to 9.0 wt%.

The effects of the adhesive composition on the MOR and MOE are shown in Fig. 6a. Adhesive I consisted of SF, PEI, MA and NaOH with the SF/PEI/MA/NaOH weight ratio of 7/1.0/0.32/0.1. Adhesive II consisted of SF, PEI and MA with the SF/PEI/MA weight ratio of 7/1.0/0.32. Adhesive III consisted of SF, PEI, succinic acid and NaOH with the SF/PEI/succinic acid/NaOH weight ratio of 7/1.0/0.32/0.1. The MOR of adhesive I was significantly higher than that of adhesive II and adhesive III. The MOR of the adhesive II and the adhesive III was statistically the same. The average MOR of all adhesives met the minimum industrial requirement (horizontal solid line, Fig. 6a). The MOE of adhesive I was significantly higher than that of adhesive II and adhesive III. Adhesive II and adhesive III had the same MOE values. All MOE met the minimum industrial requirement (horizontal solid line, Fig. 6a).

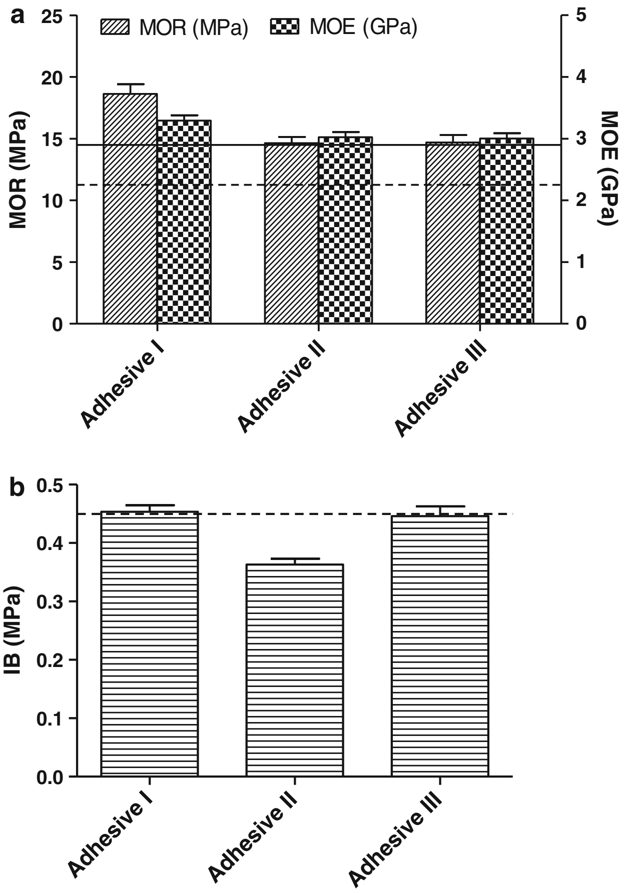

Fig. 6 **a** Effects of the adhesive composition on the MOR and MOE. (surface adhesive usage, 10 wt%; core adhesive usage, 8 wt%; hot-press temperature, 170 °C; hot-press time, 270 s; the targeted particleboard density, 0.80 g/cm^3. Data are the means of eight replications, and the *error bar* represents one standard error of the mean. Adhesive I: SF/PEI/MA/NaOH; Adhesive II: SF/PEI/MA; Adhesive III: SF/PEI/succinic acid/NaOH). **b** Effect of the adhesive composition on the IB. (Processing parameters, sample denotation and statistical details are the same as those in **a**)

The effect of the adhesive composition on the IB is showed in Fig. 6b. Adhesive I and adhesive III had the same IB. The IB of adhesive II was significantly lower than that of adhesive I and adhesive III. Moreover, the IB of adhesive I and adhesive III just met the minimum industrial requirement (horizontal dashed line, Fig. 6b), whereas the IB of adhesive II did not.

The previous publication showed that SF/PEI/MA/NaOH at the weight ratio of 7/1.0/0.32/0.1 was an optimum recipe for making interior type II plywood [7]. We assumed that this recipe was also optimum for making M-2 grade particleboards. The function of the hot-pressing in particleboard production is to provide the necessary heat and pressure for curing the adhesive, and consolidating the discrete particles into a solid board. During the hot-pressing, the heat is transferred from surface to core so the core temperature is generally lower than that of the surface temperature. Full curing of an adhesive can be accomplished by either increasing hot-press temperature at a fixed hot-press time or increasing hot-press time at a fixed hot-press temperature. However, if the hot-press temperature was higher than that needed for full curing of the adhesive at a fixed hot-press time or if the long hot-press time was longer than that needed for full curing of the adhesive, mechanical properties of resultant particleboard panels would either remain the same or decreased due to partial degradation of adhesives and wood particles. Therefore, hot-press temperature and hot-press time should be optimized. At a fixed hot-press time of 300 s, it appeared that the adhesive was already fully cured at 170 °C and further increase in the hot-press temperature did not significantly change the MOR, MOE and IB (Fig. 1a, b). At a fixed hot-press temperature of 170 °C, it appeared that the adhesive was already fully cured at the hot-press time of 270 s. The decreased MOR and MOE at the hot-press time of 370 s over 300 s implied that the hot-press time of 370 s was too long (Fig. 2a).

The mechanical properties of the particleboard are highly dependent upon the board density. Increase in the density would increase the contact of the wood particles and tightly consolidate the particle mat, thus increasing the strength. This explanation is consistent with our results that at any given core adhesive usage, the particleboard panels with the density of 0.80 g/cm^3 always had higher MOR, MOE and IB than those with the density of 0.70 g/cm^3, respectively (Fig. 3a–c).

In the preparation of particleboard panels, the adhesive was sprayed as fine droplets on surfaces of wood particles. The adhesive coverage on wood particles had to be sufficiently high to form good adhesive bonding. It was believed that not all surfaces could be covered by the adhesive. Therefore, the increase in the adhesive usage would typically increase the strengths (MOR, MOE and IB) of the resulting particleboard panels. The IB was particularly dependent upon the core adhesive usage because failure of all test specimens occurred in the core layer during the IB test, which explained why the IB almost decreased linearly when the core adhesive usage was reduced from 11 to 7 wt% at both high and low density levels (Fig. 3c). Stress first occurred on face layers of test specimens during the bending test. Therefore, the MOR and MOE were more closely related to the strength of face layers than that of the core layer unless the core layer was too weak to support the face layers during the bending test. The MOR at both high and low density levels and MOE at the high density level did not significantly decrease until when the core adhesive usage was reduced from 9 to 7 wt% (Fig. 3a, b). It is still poorly understood on why the MOE at the low density level fluctuated when the core adhesive usage was in the range of 11 to 7 wt% (Fig. 3b). That MOR and MOE gradually decreased when the surface adhesive usage was reduced from 15 to 11 wt% and was consistent with the explanation that the MOR and MOE were close related to the surface adhesive usage (Fig. 4a).

The surface adhesive usage was not supposed to have a great impact on the IB because all test specimens failed at the core layer, which was not consistent with the results shown in Fig. 4b where a reduction of the surface adhesive usage significantly decreased the IB. One possible explanation was that reducing surface adhesive usage also reduced the moisture content of the surface layer and thus reduced the heat transfer from the surface layer to the core layer, which in return slowed down the cure of the adhesive in the core layer. The results from Fig. 5b indeed indicated that the IB increased significantly when the moisture content of the surface layers was increased from 7.1 to 8.6% (Fig. 5b). The high moisture of face layers is not always helpful for improving the IB though. During the hot-pressing, water was turned into stream, resulting in the build-up of internal pressure. The stream would come out and disrupt the adhesive bonds when the hot-press was open, thus reducing the IB, which explained why the IB decreased when the moisture content of surface wood particles was raised from 9.0 to 9.4%. The cell walls of wood particles were compressed during hot-pressing and tended to bounce back when the hot-press was open. This phenomenon is known as springback in the wood composite industry. The internal pressure from the steam build-up also contributes to the springback. The MOR, MOE and IB are compromised results of adhesive strength and springback.

The effects of some adhesive components on the mechanical properties of the resulting particleboard panels were also investigated. Results shown in Fig. 6a and b further confirmed that NaOH was an essential component of this SF–PEI–MA adhesive. That the adhesive I had a higher MOR and MOE than adhesive III appeared to indicate that the double bond of MA played in important role in the adhesive, whereas that the IB was the same for adhesive I and adhesive III suggested that the double bond of MA did not play a significant role. Further investigation is warranted for a better understanding of the exact roles of each component in the adhesive.

Conclusions

This study demonstrated that the formaldehyde-free, environmental friendly SF-PEI–MA–NaOH adhesive could be used for making M-2 grade particleboard panels. The following variables and conditions were most desirable for making particleboard: hot-press temperature, 170 °C; hot-press time, 270 s; the adhesive usage of surface particles, 10 wt%; the adhesive usage of the core particles, 8 wt%; and the target particleboard density, 0.80 g/cm^3.

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