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EFFECTS OF FIRE RETARDANTS ON MECHANICAL PROPERTIES AND WATER RESISTANCE OF *PINUS MASSONIANA* PARTICLEBOARD

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ABSTRACT

Pinus massoniana Lamb. wood particleboards processed by inorganic and organic fire retardants of two densities were prepared by isocyanate and MUF resin. Variations of internal bonding strength (IB), modulus of rupture (MOR), modulus of elasticity (MOE) and thickness swelling rate (TS) of particleboard were observed. Results demonstrated decreasing of IB from 0.81 MPa to 0.42 MPa and 0.36 MPa, MOR from 17.3 MPa to 12.5 MPa and 12.3 MPa, MOE from 1840 MPa to 1328 MPa and 1117 MPa, and increasing of TS from 5.2% to 15.1% and 11.2%, respectively, for the treated MUF particleboards of density 0.65 $g \cdot cm^{-3}$. Similarly, decreasing of IB from 0.93 MPa to 0.66 MPa and 0.64 MPa, MOR from 16.2 MPa to 10.6 MPa and 12.1 MPa, MOE from 1246 MPa to 1573 MPa and 1466 MPa, and increasing of TS from 6.7% to 7.1% and 6.0%, respectively, when isocyanate adhesive was used. The similar changes were showed when the density of particleboard was 0.75 $g \cdot cm^{-3}$. Improving density of particleboard appropriately and decrease density difference between the surface to chip layers could make the profile density curve tend to be stable, which could get a relatively high mechanical strength and water resistance. Synergistic effects between isocyanate and fire retardants was confirmed. The particleboard prepared with isocyanate was obviously superior to that prepared with MUF resin in all performances.

KEYWORDS: Fire retardant, *Pinus massoniana*, particleboard, adhesive, mechanical properties, water resistance.

INTRODUCTION

With the increasing intensifying shortage of wood resources in the world, particleboard production with processing residues and logging residues has become a major way to solve the wood supply and demand contradiction (Yang et al. 2003, Wang et al. 2019, Jiang et al. 2020, Gao et al. 2020, Goodman 2020, Xiao et al.2019). Meanwhile, applications of wood-based panel also shift from the furniture industry to the fields of buildings, vehicles, ships and indoor decoration (Ma et al. 2020a, Park et al. 2012, Umemura et al. 2013). Particleboard has occupied a considerable proportion in construction industry in Europe, America and some developed countries. Especially in Sweden, particleboard occupies 80% in the construction industry (Cao 2019, Gao et al. 2020). When particleboard is applied to indoor occasions, such as indoor decoration and furniture manufacturing. If fire disasters occur, wood materials are a hidden danger to accelerate and expand the fire disaster. Therefore, wood-based materials without processing by fire retardants are listed as the flammable materials (Wu et al. 2021, Lu et al. 2020, Yu et al. 2021, Tang et al. 2020a, Son and Kang 2015). Fire retardant treatment plays a crucial role in the current and future development of particleboard industry (Yu et al. 2020, Tang et al. 2020b, Park and Baek 2015, Lazko et al. 2013).

Many scholars in the world have studied performances and development of flame-retardant particleboard, including preparation technology of flame-retardant particleboard, product performances, selection and development of fire retardant, evaluation of fire retardant performances of particleboard, and so on (Kamal et al. 2010, Ma et al. 2020b, Dai et al. 2021, Lu et al. 2021, Dagdag et al. 2021, Huang et al. 2020). Different types of fire retardant, such as boron series and phosphorus series fire retardants, have different influences on physical and mechanical performances of particleboard (Pedieu et al. 2012, Yan et al. 2019). Different doses of fire retardant and different processing techniques also have different influences on performances of flame-retardant particleboard (Kim 2018, Ren et al. 2015). In future, flame-retardant particleboard is to develop low-toxicity fire retardant which has high-efficiency flame resistance and smoke inhibition performances. Adding fire retardant into the particleboard can increase physical and mechanical properties of board or won't cause significant adverse impacts on physical and mechanical properties of board (Wang et al. 2021, Zhou et al. 2021, Liu et al. 2021). Moreover, fire retardant shall be equipped with good chemical stability and won't affect the processing and usability of particleboard. There are many studies concerning influencing factors of flame-retardant particleboard.

In this study, key attentions were paid to changes of fire retardant particleboard performances under different adhesives and densities.

MATERIALS AND METHODS

Materials

Isocyanate adhesive resin with solid content 100% was obtained from Costar Polymer Co., Ltd, China. Melamine-urea-formaldehyde (MUF) resin with solid content 52% and viscosity 144 mPa·s was prepared in the lab. *Pinus massoniana* Lamb. wood particles with moisture content 5% were also prepared in the lab. The inorganic flame retardant (FR-A) was mixed with 60% ammonium sulfate, 10% diammonium hydrogen phosphate, 10% borax and 20% boric acid. The organic flame retardant (FR-B) was mixed with 26% dicyandiamide, 25% phosphoric acid, 20% boric acid and 35% water. Chemical reagents used were all in analysis grade.

Treatments of *Pinus massoniana* wood particles with fire retardants

Pinus massoniana Lamb. wood particles were dried in an oven at 60°C ± 5°C until reaching constant weights and then cooled down at the room temperature, weighted and then put in vacuum chamber. The fire retardant with the amount of 12% on dry wood particles was poured into the chamber under vacuum conditions. Next, the wood particles were taken out at the room temperature for about one week and then dried in an oven at 90°C ± 5°C until the moisture content at 5% ~ 9%.

Preparation of particleboard and testing

Three identical monolayer particleboards of 350 × 350 × 10 mm size were prepared in the lab. The adhesive solids load was 10% on dry wood particles. The panels were pressed with a three-stage hot pressing cycle, with a pressure cycle of 33 $kg·cm^{-2}$ at 140°C for 8 min (2 + 4 + 2). Two kinds densities for board for 0.65 $g·cm^{-3}$ and 0.75 $g·cm^{-3}$ were set up.

Testing of mechanical properties and water resistance

The particleboards were conditioned in the lab at 20 ± 2°C and relative humidity of 65 ± 5% for 24 h. After sanding the surface, the boards were cut to 50 × 50 mm for the tests of dry internal bonding (IB) strength, elastic modulus (MOE), modulus of rupture (MOR) and the thickness swelling (TS) rate of water absorption and a size of 250 × 50 mm for bending strength test. At least three samples were tested for each test according to GB/T 17657-2013.

Testing of profile density

The density profile of the board was tested by their thickness scan at a rate of 0.4 $mm·s^{-1}$ using a DAX 6000 profile densitometer (GreCon, Alfeld, Germany). Based on the X-ray scanning system, the density profile reflects the density change through the board thickness.

RESULTS AND DISCUSION

IB of particleboard analysis

Internal bonding strength is an important index to evaluate performances of particleboard. The IB results of particleboards which are prepared by MUF resin and isocyanate under different target densities and different fire retardants are shown in Fig. 1. It could be seen from Fig. 1a that when MUF resin was used as the adhesive, the IB of unprocessed particleboard, particleboard processed by inorganic fire retardant (FR-A) and particleboard processed by organic fire retardant (FR-B) under the densities of 0.65 $g\cdot cm^{-3}$ and 0.75 $g\cdot cm^{-3}$ were 0.81 MPa and 1.17 MPa, 0.42 MPa and 0.51 MPa as well as 0.36 MPa and 0.57 MPa, respectively. It also could be seen from Fig. 1b that when isocyanate was used as the adhesive, the IB of unprocessed particleboard, FR-A and FR-B under the densities of 0.65 $g\cdot cm^{-3}$ and 0.75 $g\cdot cm^{-3}$ were 0.93 MPa and 1.27 MPa, 0.66 MPa and 0.79 MPa as well as 0.64 MPa and 0.81 MPa, respectively.

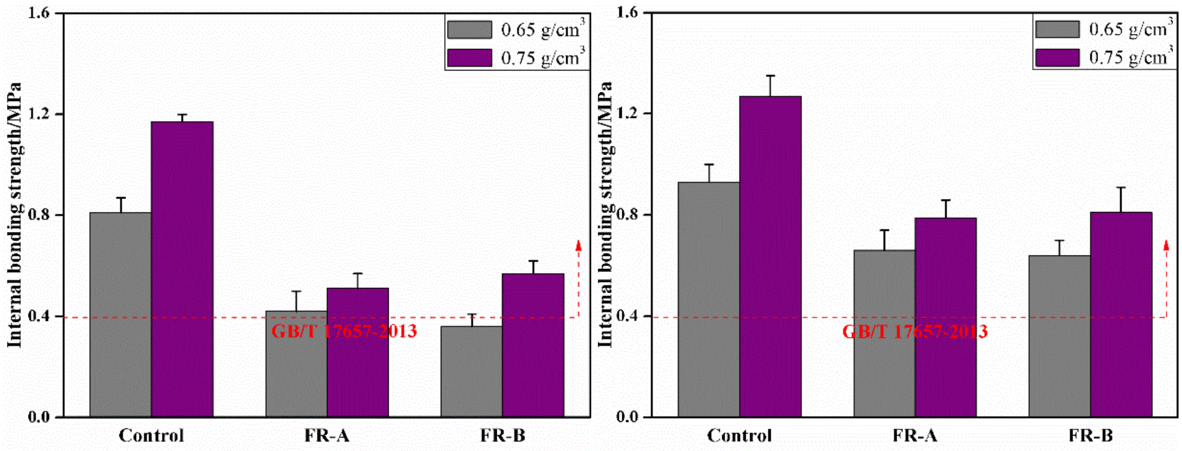

Fig. 1: The internal bonding strength particleboard prepared with MUF resin (a), isocyanate resin (b).

The results reflected that fire retardant could decrease the IB of particleboard significantly. The possible reasons were introduced as follows: *(1)* It involved flow, diffusion, permeation and curing of adhesive on wood surface (Tian et al. 2021). After fire retardant processing for wood particles, fire retardant might be left on particleboard surface to influence wetting, permeation and solidification of adhesives, thus influencing the bonding strength. Different types of fire retardants and adhesives had different influences on bonding strength. *(2)* pH was influenced curing speed and curing quality of MUF resin adhesive (Wu et al. 2019). FR-A and FR-B both had high contents of phosphoric acid and boric acid to make some MUF resin cured in advance and lower the bonding strength. However, isocyanate had high reaction activity and fire retardant exerted relatively small influences on bonding strength. *(3)* FR-A and FR-B both had high hygroscopicity, thus increasing water absorption of processed wood particles. When they were mixed with adhesives, on one hand, it was easy to cause excessive infiltration of adhesives into wood pores, which caused glue missing and thereby decreased the bonding strength. On the other hand, diffusion of steam might decrease cross linking degree of adhesives in the process of hot pressing, which further led to damages of adhesive interfaces and decrease bonding strength.

Besides, the IB of particleboard whether it was processed by fire retardant or not increased continuously with the increase of density. During preparation of particleboard, the compression volume varies, stress decreases gradually from surface to internal surface and a great density gradient was formed on the thickness profile, which were caused by inhomogeneity of pavement as well as uneven heating on surface layer and core layer of particleboard. The particleboard surface undertook the maximum stress and also had the maximum density. The core layer undertook the minimum stress (Li et al. 2014). The wood had plasticity and core layer had the minimum density led to the rebounding of particleboard thickness after pressure removal. Therefore, the IB was determined by the weakest part inside the particleboard. Density of core layer influenced the IB of particleboard significantly, while the core-surface density gradient determined the IB of particleboard directly. Increasing the density could decrease porosity inside the particleboard. Increasing compactness led to the reduction of core-surface density gradient, thus increasing the IB accordingly.

MOR of particleboard analysis

Modulus of rupture (MOR) is an important performance that determines whether the particleboard can be used as a structural component. MOR results of particleboard prepared with MUF resin and isocyanate under different densities and different fire retardants processing are shown in Fig. 2. It could be seen from Fig. 2a that when MUF resin was used as the adhesive, the MOR of unprocessed particleboard, particleboard processed by FR-A and FR-B under 0.65 $g\cdot cm^{-3}$ and 0.75 $g\cdot cm^{-3}$ were 17.3 MPa and 24.6 MPa, 12.5 MPa and 14.9 MPa as well as 12.3 MPa and 13.1 MPa, respectively. It also could be seen from Fig.2b that when isocyanate was used as the adhesive, MOR of unprocessed particleboard, particleboard processed by FR-A and FR-B under 0.65 $g\cdot cm^{-3}$ and 0.75 $g\cdot cm^{-3}$ are 16.2 MPa and 22.3 MPa, 10.6 MPa and 13.4 MPa as well as 12.1 MPa and 16.3MPa, respectively.

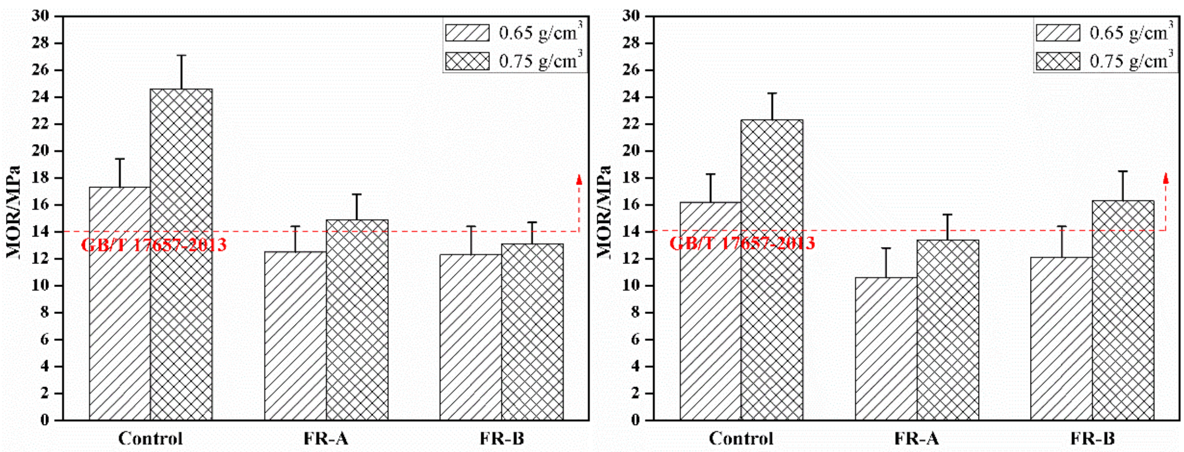

Fig. 2: The MOR of particleboard prepared with MUF resin (a), isocyanate resin (b).

At deformation of particleboard caused by bending stress, the failure often initiated from pores. Reducing pores also increased the area of undertaking the acting force and thereby increased MOR. Therefore, increasing density of particleboard increased the compactness and there were more wood in unit volume to undertake compressive stresses.

The surface density of particleboard was also an important factor that influenced MOR and the highest bending stress was on the surface layer. The surface-core density gradient of particleboard also influenced MOR greatly. MOR was negatively related with density gradient.

Given the same density, MOR of particleboard processed by FR-A and FR-B were significantly lower than that of unprocessed particleboard. This was because adding fire retardant influences bonding of particleboards, thus causing uneven density distribution and decreasing MOR.

Influences of fire retardant that using isocyanate as adhesive on MOR of particleboard were smaller than those which using MUF resin as adhesive. This was because the network chain in cured MUF resin molecules was relatively short, the crosslinking density was relatively high and the steric hindrance was relatively high. These were disadvantageous for the generation of strain when there was a great steric hindrance. Moreover, the cured MUF resin had significant brittleness for melamine triazine rings' strong rigidity (Liang et al. 2020). It was also another reason that causes great fluctuations of MOR.

MOE of particleboard analysis

Modulus of elasticity (MOE) is an important index that measures performances of particleboard and it refers to the stress-strain relationship in the limit of proportionality. It refers to performance index of rigidity. During the use of particleboard, it may develop great deformation if the rigidity is not sufficient and it also influences the usability of the particleboard. Therefore, particleboard must meet some stiffness requirements. MOE test results of particleboard prepared with MUF resin and isocyanate under different densities and different fire retardants are shown in Fig. 3.

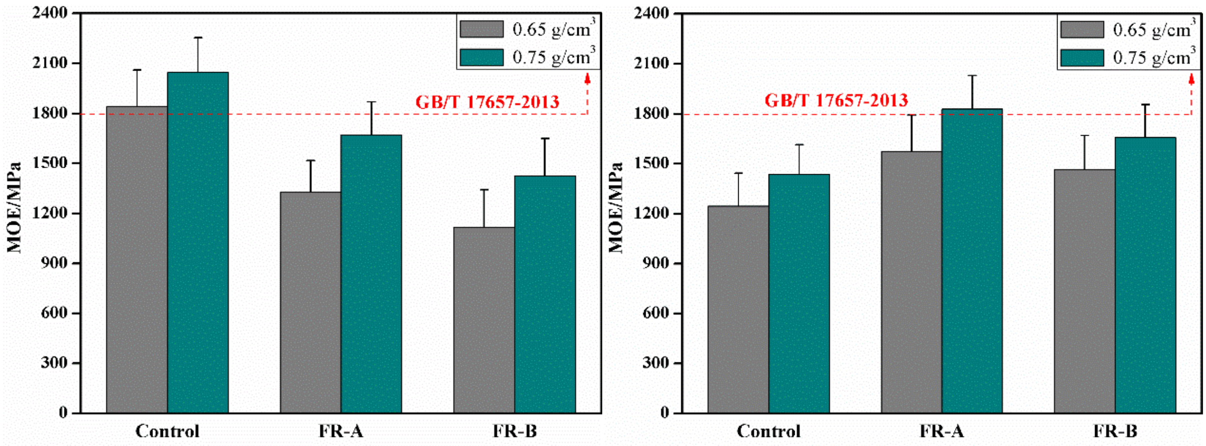

Fig. 3: The MOE of particleboard prepared with MUF resin (a), isocyanate resin (b).

It could be seen from Fig. 3a that when MUF resin was used as the adhesive, MOE results of unprocessed particleboard, particleboard processed by FR-A and FR-B under 0.65 $g{\cdot}cm^{-3}$ and 0.75 $g{\cdot}cm^{-3}$ were 1840 MPa and 2046 MPa, 1328 MPa and 1671 MPa as well as 1117 MPa and 1427 MPa, respectively. Generally speaking, MOE of particleboard was positively related with MOR and influencing factors of MOR have the same influences on MOE. For particleboard prepared with MUF resin, fire retardant had similar influences on MOE and MOR. In other words, fire retardant processing can decrease MOE of particleboard significantly and increasing density of particleboard was beneficial to improve MOE.

It can also be seen from Fig. 3b that when isocyanate was used as the adhesive, MOE results of unprocessed particleboard, particleboard processed by FR-A and FR-B under 0.65 g·cm^{-3} and 0.75 g·cm^{-3} are 1246 MPa and 1437 MPa, 1573 MPa and 1829 MPa as well as 1466 MPa and 1658 MPa, respectively. For particleboard prepared with isocyanate, fire retardant processing increases MOE of particleboard. Similarly, increasing density of particleboard was also beneficial to improve MOE.

Combining with Figs. 2 and 3b, fire retardant treatment lowered MOR of particleboard prepared with isocyanate, but the reduction amplitude was significantly lower than that of particleboard prepared with MUF resin. Fire retardant treatment decreased the MOE of particleboard prepared with MUF resin, but increased the MOE of particleboard prepared with isocyanate. This might be because there was some synergistic effect between fire retardants and isocyanate (Li et al 2014, Xu et al. 2020).

Water resistance of particleboard analysis

Test results of the TS of particleboard prepared with MUF resin and isocyanate under different densities and different fire retardants are shown in Fig. 4. It could be seen from Fig. 4a that when MUF resin was used as adhesive, the TS of unprocessed particleboard, particleboard processed by FR-A and FR-B under 0.65 g·cm^{-3} and 0.75 g·cm^{-3} are 5.2% and 5.8%, 15.1% and 16.6% as well as 11.2% and 14.3%, respectively. It could be seen from Fig. 4b that when isocyanate was used as adhesive, the TS of unprocessed particleboard, FR-A and FR-B under 0.65 g·cm^{-3} and 0.75 g·cm^{-3} are 6.7% and 7.3%, 7.1% and 7.6% as well as 6.0% and 6.8%, respectively.

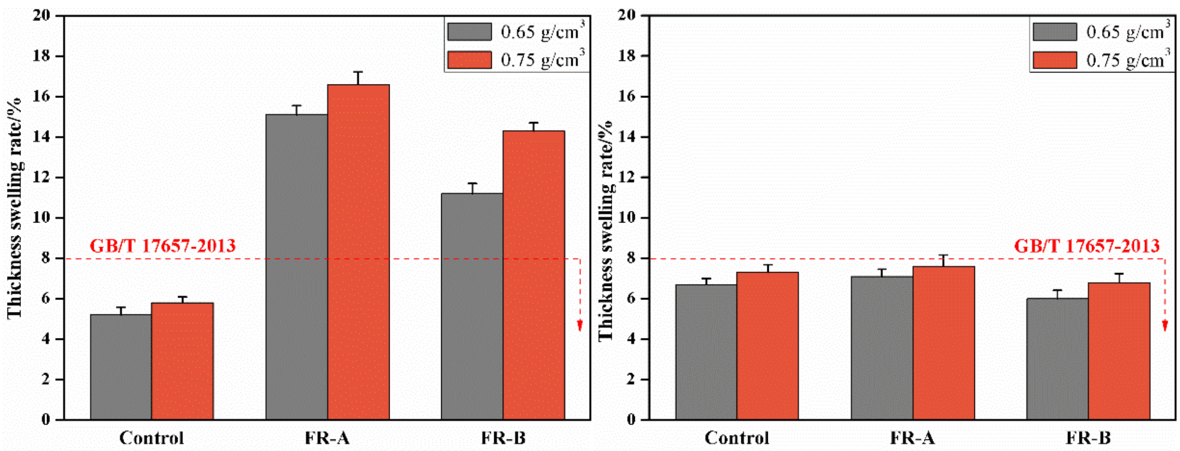

Fig. 4: The thickness swelling rate of particleboard prepared with MUF resin (a), isocyanate resin (b).

Inorganic fire retardants mainly have hygroscopicity and it can absorb and maintain water from surrounding water. This is more serious under conditions with relatively high humidity (Wu et al 2021, Yu et al. 2020, 2021). Inorganic fire retardant was composed of diammonium hydrogen phosphate, ammonium sulfate, borax and boric acid. Ammonium sulfate and diammonium hydrogen phosphate could absorb water quickly when the relative humidity increased 90% to make the moisture content of wood exceed 50% (Cao 2019). Borax and boric acid also could absorb water under a low relatively low humidity. Influenced by molecular bonds, these hydrophilic groups in fire retardant absorbed water into surrounding areas of fire retardant inside the particleboard. With the increase of absorbed water, the particleboard absorbed water and swelled. Moreover, the solubility of fire retardants increased after moisture absorption and the attraction force between fire retardant and water molecules increased. As a result, the TS of particleboard was further increased. Major ingredient of FR-B was the GUP which was produced by reaction of phosphoric acid and dicyandiamide. GUP had weak hydration and relatively low hygroscopicity. Therefore, fire retardant processing influenced water absorption capability of particleboard significantly, especially on FR-A. It was important to note that influences of FR-A and FR-B on the TS of particleboard prepared with isocyanate were significantly smaller than those on particleboard prepared with MUF resin. This further demonstrated that there was some synergistic effect between fire retardants and isocyanate, and it was also confirmed to the conclusion of Li et al (2014).

Moreover, density could also influence hygroscopicity of particleboard. Given a high density, the compression ratio was relatively high and the locked internal stress under thermal compression was also high. After water infiltration, stress was released by powers. In other words, a relatively high TS was produced. When the density was relatively low, although the internal stress was also low, the bonding points decreased to some extent and the porosity increased relatively, forming a high TS.

The density profile characteristic of the particleboard

When MUF resin was used as adhesive, particleboards were prepared by three methods, including 0.75 $g\cdot cm^{-3}$ and FR-B (a), 0.65 $g\cdot cm^{-3}$ and FR-B (b) and 0.65 $g\cdot cm^{-3}$ and FR-A (c). They are used for profile density analysis in Fig. 5.

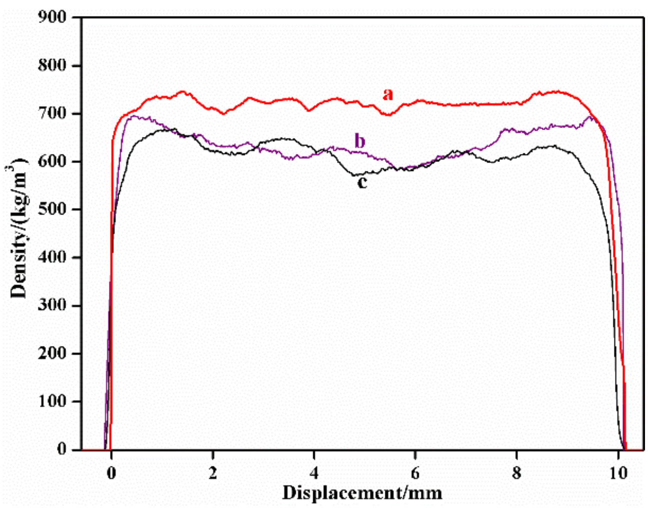

Fig. 5: Density profile of particleboards.

According to comparison of curves b and c, the influence degree of FR-A on density homogeneity of particleboard profile was higher than that of FR-B. According to comparison of curves a and b, increasing target density of particleboard appropriately could decrease the surface-core density gradient and the profile density distribution curve tends to be stable, thus enabling to get a relatively high mechanical strength.

CONCLUSIONS

Pinus massoniana wood particleboard was processed by inorganic fire retardants and organic fire retardants. Then *Pinus massoniana* based particleboards with two densities were prepared by isocyanate and MUF resin. Variations of internal bonding strength, modulus of rupture, modulus of elasticity and thickness swelling rate of particleboard were observed. Results showed that different fire retardants and adhesives influenced performances of particleboard to different extents. *(1)* Fire retardant decreased internal bonding strength, modulus of rupture and thickness swelling rate of particleboard. Inorganic fire retardant influenced performances of particleboard the mostly due to the high hygroscopicity. *(2)* The particleboard prepared with isocyanate was obviously superior to that prepared with MUF resin in all performances. After treated by fire retardants, the internal bonding strength, modulus of rupture, modulus of elasticity and thickness swelling rate of particleboard prepared with MUF resin were affected significantly. The internal bonding strength and modulus of rupture of particleboard prepared with isocyanate decreased, while the modulus of elasticity and water resistance increased significantly, indicating that there were some synergistic effects between isocyanate and fire retardants. *(3)* Improving density of particleboard appropriately and decrease density difference between the surface to chip layers could make the profile density curve tend to be stable, which could get a relatively high mechanical strength and water resistance.

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