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Influence of hydrothermal modification of fibers on some physical and mechanical properties of medium density fiberboard (MDF)

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Abstract Any treatment of fibers can influence properties of medium density fibreboard (MDF). In this research, the effects of hydrothermal treatment on physical and mechanical properties of MDF were studied. Industrial fibers were hydrothermally treated in a stainless steel reactor at 120, 150 and 180 ◦C for 0, 30 and 90 min as holding time. Test boards were made based on 0.7 g/cm³ target density, with 10 mm thickness under a pressure of 30 bar and at a temperature of $170\textdegree C$ and a press time of 10 min. The boards were tested for internal bonding (IB), moduli of elasticity (MOE) and rupture (MOR), thickness swelling and water absorption.

Results showed that the water absorption was not affected by the hydrothermal treatment; while the thickness swelling was improved and the boards became dimensionally stable. The MOE was slightly reduced due to the hydrothermal treatment. The MOR and the IB were significantly decreased by the hydrothermal treatment.

Einfluss einer hydrothermischen Behandlung von Fasern auf physikalische und mechanische Eigenschaften mitteldichter Faserplatten (MDF)

Zusammenfassung Die Eigenschaften mitteldichter Faserplatten (MDF) können durch eine Behandlung der Fasern beeinflusst werden. In dieser Studie wird der Einfluss einer hydrothermischen Behandlung auf die physikalischen und mechanischen Eigenschaften von MDF untersucht. Industri-

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ell hergestellte Fasern wurden in einem Edelstahlbehälter bei 120, 150 und 180 ◦C jeweils 0, 30 und 90 Minuten lang hydrothermisch behandelt. 10 mm dicke Versuchsplatten mit einer Solldichte von $0.7 \frac{g}{cm^3}$ wurden bei einem Druck von 30 bar, einer Temperatur von 170 ◦C und einer Presszeit von 10 min hergestellt. Die Querzugfestigkeit (IB), der E-Modul und die Biegefestigkeit sowie die Dickenquellung und die Wasseraufnahme der Platten wurden geprüft.

Die Ergebnisse zeigten, dass die hydrothermische Behandlung keinen Einfluss auf die Wasseraufnahme hatte. Die Dickenquellung wurde verbessert und die Platten wurden formstabiler. Durch die Behandlung nahm der E-Modul leicht ab und die Biegefestigkeit sowie die Querzugfestigkeit verringerten sich signifikant.

1 Introduction

Many research efforts have been accomplished in the field of thermal treatment of wood. Several types of treatment have been tested in oxygen and oxygen free atmospheres (Militz 2000 , Vernois 2000 , Syränen et al. 2000 , Rapp and Sailer 2001, Mohebby and Sanaei 2005). Many reports on the influences of heat treatment on wood properties have been published. Most of the publications indicated that thermal treatment improves dimensional stability of wood, reduces swelling and enhances fungal resistance of the wood (Tjeerdsma et al. 2000).

Along with the improved properties, some unwanted effects arise in wood; such as reduction in strength (Bengtsson et al. 2002) and modulus of elasticity as well as modulus of rupture (Yildiz et al. 2002). Interest in research on the application of thermal modification of wood-based composites has been arisen since the last decade. It was reported that thermal treatment improves the dimensional stability in particleboards (Boonstra et al. 2006, Ohlmeyer and Lukowsky 2004) and OSB (Paul et al. 2006); however, it reduces modulus of elasticity and modulus of rupture in OSB (Paul et al. 2006).

Increasing demands of enhanced wood products for severe exterior applications have encouraged technologists to modify wood properties. Interest in application of MDF for different purposes is rising on the market every year. For this reason, the current research was undertaken to study the hydrothermal treatment of industrially produced fibers as well as its influences on some physical and mechanical properties of medium density fibreboards.

2 Experimental

Industrially produced fibres were prepared and air dried to achieve equal moisture content. For treatment, a stainless steel reactor was filled with water up to 3/4 of its capacity and heated up to 100 ◦C. Afterwards, the fibres were placed in the cylinder and treated at temperatures of 120, 150 and $180 °C$ for 0, 30 and 90 min of holding time.

The treated fibres were oven dried and test boards were made with dimensions of $10 \times 350 \times 350$ mm³ and with 10% of urea formaldehyde resin (UF). The target density of the boards was adjusted to 0.7 g/cm^3 . The boards were manufactured under a press pressure of 30 bar at 175 ◦C for 5 min. Six boards were considered for the applied treatments as replicates. The boards were conditioned at room temperature and a relative humidity of $65 \pm 1\%$ for two weeks. Water absorption, thickness swelling, modulus of elasticity (MOE), modulus of rupture (MOR) and internal bonding (IB) were tested according to EN 317, EN 310 and EN 319, respectively. Anti-swelling-efficiency was calculated based on the following equation.

$$
ASE = (S_{\text{unt}} - S_{\text{tr}})/S_{\text{unt}} \times 100
$$
 (1)

where: ASE is anti-swelling-efficiency (%), S_{unt} and S_{tr} are thickness swelling of the untreated and treated samples (%), respectively.

Results were statistically analyzed based on a full factorial design and Duncan test was used to compare means.

3 Results

3.1 Water absorption

Analysis of variance (ANOVA) of the data showed insignificant effects of the treatment factors (temperature, treatment time and their interactions) on the water absorption of the **Table 1** Analysis of variance for applied treatments **Tabelle 1** Varianzanalyse

**: Significant at 99%; *: Significant at 95%; ns: Not significant

Fig. 1 Main effect of temperature on water absorption after soaking in water for 24 h

Abb. 1 Einfluss der Temperatur auf die Wasseraufnahme nach 24 stündiger Wasserlagerung

Fig. 2 Main effect of treatment time on water absorption after soaking in water for 24 h

Abb. 2 Einfluss der Behandlungsdauer auf die Wasseraufnahme nach 24-stündiger Wasserlagerung

test specimens after 2 and 24 hours of soaking in the water (Table 1).

As it is shown in Figs. 1 and 2, negligible reduction occurred in the water absorption of the treated boards. The reduction is not statistically significant. It means that elevation of the treatment temperature and extending the treatment did not affect the water absorption.

3.2 Thickness swelling

It was revealed that the thickness swelling of the specimens was significantly affected by the temperature and the holding time at a confidence level of 99% with no interactions between (Table 1).

Results indicated a reduction in the thickness swelling of the hydrothermally treated boards. It was revealed that elevation of the temperature decreased the thickness swelling of the boards up to 35% (Fig. 3) and extending the treatment time reduced it up to 22% (Fig. 4). In general, it was revealed that the swelling was affected by the hydrothermal treatment and the hydrophobicity of the treated

Fig. 3 Main effect of temperature on thickness swelling after soaking in water for 24 h

Abb. 3 Einfluss der Temperatur auf die Dickenquellung nach 24 stündiger Wasserlagerung

Fig. 4 Main effect of treatment time on thickness swelling after soaking in water for 24 h

Abb. 4 Einfluss der Behandlungsdauer auf die Dickenquellung nach 24-stündiger Wasserlagerung

boards was improved in comparison with the untreated ones.

3.3 Modulus of elasticity (MOE)

As it is shown in Table 1, the treatment temperature and the holding time as well as their interactions affected the modulus of elasticity at a confidence level of 95%.

Results revealed that the MOE was reduced due to the hydrothermal modification of the fibers. The elevation of the temperature to $120\degree C$ and higher decreased the MOE by approximately 20% (Fig. 5). Beside that, the treatment of the fibers for more than 30 min diminished the modulus of the elasticity by approximately 15% in the treated boards (Fig. 6).

3.4 Modulus of rupture (MOR)

According to the results of ANOVA, the modulus of rupture (MOR) was also influenced significantly by the temperature

Fig. 5 Main effect of temperature on MOE **Abb. 5** Einfluss der Temperatur auf den E-Modul

Fig. 6 Main effect of treatment time on MOE **Abb. 6** Einfluss der Behandlungsdauer auf den E-Modul

Fig. 7 Main effect of temperature on MOR **Abb. 7** Einfluss der Temperatur auf die Biegefestigkeit

Fig. 8 Main effect of treatment time on MOR **Abb. 8** Einfluss der Behandlungsdauer auf die Biegefestigkeit

and the treating time as well as their interactions at confidence levels of 99% and 95%, respectively (Table 1).

Results showed significant loss in the MOR due to the hydrothermal treatment. Increase of the temperature to 120 ◦C and higher reduced the modulus of rupture by about 18– 28% in the boards (Fig. 7). In addition to the treatment temperature, any increase of the treatment time to 30 min or more affected the fibers and reduced the MOR in the boards by about 14–20% (Fig. 8).

3.5 Internal bonding (IB)

ANOVA showed that the internal bonding (IB) of the boards was significantly influenced by the treatment temperature as well as its interaction with the holding time at a confidence level of 99% (Table 1). However, no significant influence was determined for the holding time.

Results showed significant effect of the temperature on the internal bonding of the boards. As it is shown in Fig. 9, when the fibers are treated at 120 ℃ and higher, the IB was

Fig. 9 Main effect of temperature on internal bonding (IB) **Abb. 9** Einfluss der Temperatur auf die Querzugfestigkeit

Fig. 10 Main effect of treatment time on internal bonding (IB) **Abb. 10** Einfluss der Behandlungsdauer auf die Querzugfestigkeit

decreased by about 20–44%. Meanwhile, any increase of treatment time could not affect the internal bonding in the treated boards (Fig. 10).

4 Discussion

According to the results, the water absorption was not affected by the hydrothermal treatment. It is likely that the water absorption had no general influence on the dimensional stability of the MDF. Similar result was also reported in OSB made from heat treated chips by Paul et al. (2006). The thickness swelling represents the dimensional stability in the boards. The current study showed its improvement by the hydrothermal treatment. The reduction of the thickness swelling could be related to the chemical modification of the fiber cell walls during the treatment. Concerning the reports, the hemicelluloses became degraded due to the thermal elevation and changed to soluble extractives (Garrote et al. 1999, Tjeerdsma and Militz 2005). As the hemicelluloses are very hydrophilic compounds in the cell wall, their removal could affect the dimensional stability in the boards. The hemicelluloses are heteropolymers with branched structures and they have higher tendencies for the water absorption. Any removal of the hemicelluloses could affect significantly the swelling of the boards.

Increase of the crystalline regions in the cellulose microfibrils is another reason for the reduction of the thickness swelling (Wallenberger and Weston 2004, Yildiz and Gümüşkaya 2006). The moisture absorption could be suppressed due to growth in the cellulose crystallinty and reduction of the amorphous regions in the cellulose microfibrils, bcause less hydroxyl groups are free in the cellulose microfibrils after the hydrothermal treatment. Cross linkings between the cell wall polymers, especially lignin, and esterification between the cellulose microfibrils are other reasons for reduction of the swelling (Tjeerdsma and Militz 2005, Boonstra and Tjeerdsma 2006).

Loss of MOE in thermally modified wood was reported by various authors (Yildiz et al. 2002, Bengtsson et al. 2002) and also heat treated composites (Sundqvist et al. 2006, Ohlmeyer and Lukowsky 2004, Paul et al. 2006). The MOE and MOR losses in the treated boards could be related to formation of soluble acidic chemicals, such as formic acid and acetic acid, from the hemicelluloses degradation (Garrote et al. 2001, Sundqvist et al. 2006). Those acids accelerate depolymerization of the cellulose by breaking down the long chain cellulose to shorter chains. Depolymerization and shortening of the cellulose polymer could affect the MOE and MOR in wood (Winandy and Rowell 2005). It is known that acidic condition at elevated temperature can degrade the wood by hydrolysis (Tjeerdsma and Militz 2005) and affects wood strengths (Winandy and Rowell 2005).

The internal bonding strength (IB) indicates homogeneity of the adhesion between the fibers, which are bonded by adhesives. Therefore, it indicates whether the applied adhesive is adoptable to the modified fibers or not and it shows also whether the adhesive is capable to wet perfectly the hydrothermally treated fibers or not. On the other hand, the IB represents single fiber strengths against applied load during the test. If the fibers are not perfectly wet from the adhesive or their strengths are reduced due to the treatment, the IB is susceptible for reduction.

Any loss of the IB strength here might be related to the loss of adhesion between the fibers due to loss of wettability. As reports indicated, the fibers become hydrophobic after the heat treatment and their wetting capability becomes less due to altered chemistry (Hakkou et al. 2005, Tjeerdsma and Militz 2005). The UF resin, which was used for the current study, is a polar adhesive and it initially needs to wet the fibers for bonding and it is also able to bond materials with the same polarities (Pizzi 1994, Vick and Rowell 1990, Frihart 2005). However, its wetting capability became influenced due to loss of fibers' wettability and also binding the cell wall polymers, which are chemically modified to new compounds, became problematic. The loss of IB in particleboard was also reported by Boonstra et al. (2006).

As another reason, it could be suggested that the single fiber strengths became less due to the hydrothermal treatment. Since, it was observed during the treatment, the treated fibers look brittle.

5 Conclusion

Insignificant water absorption was determined in the medium density fiberboards made from hydrothermally modified fibers. However, the thickness swelling of the boards was significantly reduced due to the treatment. The hydrothermal modification of the fibers cause the fiberboards become dimensionally stable. The moduli of elasticity were significantly changed due to the hydrothermal modificationandthey were decreased proportional to increased temperature and the holding time. The internal bonding of the boards was drastically reduced due to elevation of the treatment temperature; while, it was not affected by the extended holing time. Internal bonding related to single fiber strengths as well as their wettability related to adhesive type. Further researches should be done to study wettability of the fibers versus to different formulation of the adhesives as well as single fiber strengths. As the treated boards become hydrophobic, it would be recommended to study the mechanical properties at varying relative humidities, because the current results has ascribed to the mechanical properties of the boards at dry condition.

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References

- Bengtsson C, Jermer J, Brem F (2002) Bending strength of heattreated spruce and pine timber, The International Research Group on Wood Preservation, IRG Document No. IRG/WP 02-40242
- Boonstra MJ, Tjeerdsma BF (2006) Chemical analysis of heat treated softwoods. Holz Roh- Werkst 64:204–211
- Boonstra MJ, Pizzi A, Zomers F, Ohlmeyer M, Paul W (2006) The effect of a two stage heat treatment process on the properties of particleboard. Holz Roh- Werkst 64(2):157–164
- European Committee for Standardization (CEN), EN 310 (1993) Wood based panels – Determination of modulus of elasticity in bending strength
- European Committee for Standardization (CEN), EN 317 (1993) Particleboards and fiberboards – Determination of swelling in thickness after immersion in water
- European Committee for Standardization (CEN), EN 319 (1993) Particleboards and Fiberboards – Determination of tensile strength perpendicular to the plane of the board
- Frihart CR (2005) Wood adhesion and adhesives. In: Rowell RM (ed) Handbook and wood chemistry and composites. CRC Press, London, pp 215–278
- Garrote G, Dominiguez H, Parajo JC (1999) Hydrothermal processing ´ of lignocellulosic materials. Holz Roh- Werkst 57:191–202
- Garrote G, Dominiguez H, Parajó JC (2001) Study on the deactylation of hemicelluloses during the hydrothermal processing of *Eucalyptus* wood. Holz Roh- Werkst 59:53–59
- Hakkou M, Pétrissans M, Zoulalian A, Gérardin P (2005) Investigation of wood wettability changes during heat treatment on the basis of chemical analysis. Polym Degrad Stabil 89:1–5
- Militz H (2000) Heat Treatment Technologies in Europe: Scientific Background and Technological State-of-Art, Conference on Enhancing the Durability of Lumber and Engineered Wood Products, February 11–13, Kissimmee, Orlando, Forest Products Society, Madison, USA
- Mohebby B, Sanaei I (2005) Influences of the hydro-thermal treatment on physical properties of beech wood (*Fagus orientalis*), The International Research Group on Wood Preservation, IRG Document No. IRG/WP 05-40303
- Ohlmeyer M, Lukowsky D (2004) Wood-based panels produced from thermal-treated materials: Properties and perspectives, Conference on Wood Frame Housing Durability and Disaster Issue, 4–6th Oct., Las Vegas, USA, pp 127–131
- Paul W, Ohlmeyer M, Leithoff H, Boonstra MJ, Pizzi A (2006) Opimising the properties of OSB by a one-step heat pretreatment process. Holz Roh- Werkst 64(3):227–234
- Rapp AO, Sailer M (2001) Oil-heat treatment of wood: Process and properties, COST action 22
- Pizzi A (1994) Advanced wood adhesives technology. Marcel Dekker Inc, New York
- Sundqvist B, Kalrsson O, Westrmark U (2006) Determination of formic-acid and acetic acid concentrations formed during hy-

drothermal treatment of birch wood and its relation to colour, strength and hardness. Wood Sci Technol 40:549–561

- Syränen T, Jämsä S, Viitaniemi P (2000) Heat treatment of wood in Finland – State of the art. In: Proceedings of the Träskydd-, värmebehandlat trä-, egenskaper och användningsormråden, 21st Nov., Stockholm, Sweden
- Tjeerdsma BF, Stevens M, Militz H (2000) Durability aspects of hydrothermaly treated wood. The International Research Group on Wood Preservation, IRG Document No. IRG/WP 00-40160
- Tjeerdsma BF, Militz H (2005) Chemical changes in hydrothermal treated wood: FTIR analysis of combined hydrothermal and dry heat-treated wood. Holz Roh- Werkst 63:102–111
- Vernois M (2000) Heat treatment of wood in France State of the art: In: Proceedings of the Träskydd-, värmebehandlat trä-, egenskaper och användningsormråden, 21st Nov., Stockholm, Sweden
- Vick CB, Rowell RM (1990) Adhesive bonding of acetylated wood. Int J Adhes Adhes 10(4):263–272
- Wallenberger FT, Weston N (2004) Natural fibers, Plastic and Composites. Kluwer Academic Publisher, Massachusetts, p 392
- Winandy JE, Rowell RM (2005) Chemistry of wood strength. In: Rowell RM (ed) Handbook of wood chemistry and wood composites. CRC Press, London, pp 303–347
- Yildiz S, Çolakoğlu G, Yildiz ÜC, Gezer DE, Temiz A (2002) Effects of heat treatment on modulus of elasticity of beech wood. The International Research Group on Wood Preservation, IRG Document No. IRG/WP 02-40222
- Yildiz S, Gümüşkaya E (2006) The effects of thermal modification on crystalline structure of cellulose in soft and hardwood. Build Environ 42:62–67