Pre-pressing and Pre-heating *via* Hot-Air/Hot-Steam Process for the Production of Binderless Medium-Density Fiberboards

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The hot-air/hot-steam process was used for the first time as a combined pre-pressing and pre-heating system for the production of medium-density fiberboards (MDF) at the pilot scale. Pre-heating systems are designed to pre-heat fiber mats before pressing by hot-presses. Using such techniques, pressing times are reduced significantly and the board properties are influenced positively; both are essential for effective MDF production. In recent years, industry has searched for alternatives to petrochemical binders. Primarily, MDF are bonded by urea-formaldehyde (UF) resins in Europe. To replace UF resins, a laccase-mediator-system (LMS) was used to activate the wood fibers' self-cohesion. It was found that the internal bond strength (IB) and thickness swelling (TS) were noticeably improved by applying the hot-air/hot-steam process before final hot-pressing for both LMS and 10% UF binding systems. Simultaneously, the total pressing time could be reduced by 25% when combining the hot-air/hot-steam process with hot-pressing.

Keywords: Hot-air/hot-steam process; hot-press; pressing time; laccase-mediator-system; MDF

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INTRODUCTION

In the early 1970s, the worldwide first continuous hot-press was installed for the production of particleboards in Germany. Before that time, blended and pre-formed wood particles were exclusively pressed by intermittently working single- or multi-daylight presses. Later on, these systems were largely replaced by continuously working presses, also for the production of fiber boards, yielding higher production capacities. Today, such presses are called Contipress, ContiRoll, or ContiPlus (Dieffenbacher 2015; Siempelkamp 2016).

With the application of the continuous hot-pressing technology, the pre-pressing and pre-heating of fiber fleeces were introduced as previous and separated steps. For the pre-heating of fiber fleeces, different techniques are known. Generally, pre-heating systems results in an enhanced plasticization of wood fibers and simultaneous reduction of pressing time and pressing power, *e.g.*, resulting in fiberboards of higher quality and lower formaldehyde emissions. The oldest and most common system is the steam jet principle, which is used in most factories, producing medium density fiberboards (MDF) to increase productivity and achieve good surface qualities (Deppe and Ernst 1996). After additional moistening of the fleece surfaces and conveyance into the hot-press, the generated steam passes into the inner zone of the fleece, curing the binder quickly (Krug 2010). The most effective heat transport *via* steam is realized by the steam injection process (Dunky and

Niemz 2002). This system may be integrated in the first zone of a hot-press, injecting hot steam (approx. 100 °C) into the fiber fleece while the press is closing and compressing the fleece. It has been reported that over 15% of total pressing time can be saved using the steam injection technique (Dieffenbacher 1998). A disadvantage of the steam injection process is seen in the choice of an adequate binder, as not every binder system is suitable for pre-heating by steam injection, resulting in poor bonding strength (Umemura *et al.* 1996; Umemura 1997).

A modified system, called CoreHeater, was designed to cut the fiber fleece horizontally in the middle by a band saw and heat up the separated parts to approximately 70 °C by hot steam. The following pressing process in a Contipress could be improved by up to 20% (Krug 2010). Nevertheless, the CoreHeater system still causes troubles in daily MDF production because of the complex design and the fact that the system is easily contaminated. Other systems include microwave pre-heating and radio-frequency pre-heating. Both systems work on the principle of oscillation between water particles in the material induced by an electromagnetic alternating field (Frühwald and Gruchot 2002). The wooden materials are uniformly warmed up, but both systems are expensive and require a lot of electric power; thus, only special products like veneer-based products are produced using these pre-heating procedures (Irle and Barbu 2010).

An essential disadvantage of the separated steps of pre-pressing and pre-heating is the possible “spring-back effect”, which means a sudden pressure relief of the fiber fleece between the pre-press and pre-heater units and the hot-press, which causes quality problems for the fiber boards (Irle and Barbu 2010). To avoid spring-back effects, the fiber or particle materials or the density profiles of the panel boards can be modified (Gupta 2007; Thoemen and Ruf 2008; Pelaez-Samaniego *et al.* 2014).

From a machine-technological perspective, several techniques are known to overcome the “spring-back effect” by combining pre-pressing and pre-heating in one unit, including integrated pre-pressing linked with radio-frequency pre-heating (Krug and Mäbert 2012) or a soft-pressing unit working with a steam-air mixture, called ContiTherm (Siempelkamp 2012) or a Continuous Steam System (CSS) (Dieffenbacher 2015). ContiTherm or CSS systems are usually applied for the production of wood insulation boards, hardening boards with raw densities between 80 and 240 kg/m^3 and thicknesses up to 300 mm (Lempfer 2011).

For the production of wood-based panels such as MDF, a ContiTherm or CSS can be used as a combined pre-press and pre-heater before hot-pressing by Contipress, which is required to produce raw densities well above 240 kg/m^3. In both pre-heating systems, the maximum temperature is limited to 100 °C, caused by the chemical-physical condition of the steam-air mixture. Most petrochemical binder systems, like urea formaldehyde (UF) and phenol formaldehyde (PF) resins, completely react at temperatures above 100 °C (Deppe and Ernst 1996; Habenicht 2009). Therefore, the final pressing has to take place at higher temperatures, located in a Contipress. As an alternative to petrochemical binder systems, natural alternatives have been the recent focus of various research activities. For example, binders based on renewable resources such as isolated lignins, tannins, proteins, casein, glutens, or starches have been tested as possible substitutes, especially to replace formaldehyde-containing binder systems (Niemz 1993; Kharazipour 1996; Türk 2014). Commonly, all mentioned natural binder-systems also need temperatures well above 100 °C to react completely for gluing.

In a previous study, Euring *et al*. (2015) described a new hot-air/hot-steam process, which was originally developed as a soft-pressing unit for the production of insulation boards. The difference from the ContiTherm or CSS systems is the higher process temperature. By applying hot air and afterwards hot steam, temperatures well over 100 °C are reached, which are required in the hardening of native wood fiber lignin (Euring and Kharazipour 2012; Euring *et al*. 2015). Especially thermo-mechanical-pulping (TMP) wood fibers have the ability for self-bonding, also called auto-adhesion (Zavarin 1984; Back 1987; Suzuki *et al.* 1998; Unbehaun *et al.* 2000). Caused by the thermo-mechanical treatment of wood, the glass transition temperature (T_g) of native lignin is reached between 100 and 170 °C (Irvine 1985; Nada *et al.* 2002). Mainly the middle-lamella lignin is plasticized and then available for lignin-lignin and lignin-polysaccharides cross-linking reactions during hot pressing exceeding the T_g again (Bouajila *et al.* 2005). Such lignin reactions are effectively supported by laccase-mediator systems (LMS) through oxidation processes (Kües *et al.* 2007; Kudanga *et al.* 2008; Widsten and Kandelbauer 2008; Nyanhongo *et al.* 2010). Wood fibers lignin is initially activated, yielding phenoxy-radicals that positively influence the fiber to fiber bindings while pressing (Felby *et al.* 1997; Widsten 2002). LMS application for the production of wood fiber composites offers many advantages. In addition to the independence from fossil oil resources, the use of LMS causes no extra emissions (either formaldehyde or volatile organic compounds, VOCs) out of the products (Müller *et al.* 2009; Euring *et al.* 2011, 2013, 2015, 2016).

For the production of MDF with LMS, temperatures to exceed T_g again are reached using only hot-pressing systems like daylight or continuous presses, but it is known that natural binding systems require longer pressing times than conventional systems, which means lower productivity and higher costs (Dunky and Niemz 2002).

Therefore, in this study, the hot-air/hot-steam process was applied for the first time as a pre-pressing and pre-heating system for pilot-scale MDF production, prior to the use of a hot-press. The aim was to pre-heat the LMS-treated wood fiber fleece completely up to 160 °C using a defined time of hot air and then hot steam, followed by final hot-pressing to ensure an effective lignin polymerization and a presumed reduction of the total pressing time. Various pre-heating and final pressing procedures were chosen, and the physical-mechanical properties of the produced MDF were compared.

EXPERIMENTAL

Materials

Wood fibers (22 kg wood fibers with a moisture content of 10 % per batch)

Pine wood (containing 100% *Pinus sylvestris* wood) was defibrated into fibers by the thermo-mechanical-pulping (TMP) process at STEICO AG, Czarnkow, Poland.

Laccase (100 U laccase/g referred to absolutely dry wood fibers)

Novozym 51003 *Trametes villosa* laccase, recombinantly produced in *Aspergillus oryzae* by Novozymes (Bagsveard, Denmark), was used. Its specific activity was routinely determined by measuring the oxidation of di-ammonium salt of 2,2´-azinobis-3-ethylbenzothiazoline-6-sulfonic acid (ABTS) (Matsumura *et al.* 1986). One unit [U] of laccase is described as the amount needed for the enzymatic conversion of one micromole ABTS per minute. The activity of the laccase stock was approximately 1000 U/g.

Mediator (10 mM/g referred to absolutely dry wood fibers)

4-Hydroxy-benzoic-acid (HBA) was obtained from Alfa Aesar (Ward Hill, USA) with a purity of 99%.

Buffer (pH 6)

McIlvain-buffer (pH 6.0) containing 0.2 M dipotassium hydrogen phosphate (K_2HPO_4) and 0.1 M citric acid ($C_6H_8O_7$) (AppliChem; Darmstadt, Germany) was used as a reaction buffer for the LMS (also for inactivated LMS and laccase treatments) in all experiments.

Urea-formaldehyde resin (10% UF/22 kg wood fibers)

Commercial UF-resin K 465 with a solid content of 66% and a pH value of 7.5 from BASF (Ludwigshafen, Germany) was used for reference MDF.

Methods

Temperature gradient measurements

Temperature measurements were taken over a 12 s/mm time span (total pressing time was 120 s) during the production of 10-mm-thick MDF, using different methods to find out more about the thermal behavior within the pre-pressed fiber fleeces:

a) hot-air/hot-steam + hot press
b) hot-air/hot-steam + cold press
c) hot-press
d) hot-air + hot-press
e) hot-steam + hot-press

For a better comparison between treatments, the hot-air was exclusively adjusted to a temperature of 140 °C and applied with a flow rate of 15 m/s for 5 s/mm, which was found to be the optimum in this study. The hot-steam was set to 100 °C and applied for 1 s/mm. Hot-pressing took place at 190 °C and – depending on the method – at different pressing times, varying from 6 to 12 s/mm. The method `hot-air/hot-steam + cold press´ was performed with a cold press-platen (room temperature) to analyze the effect on the properties, after the MDF was only pre-heated by the hot-air/hot-steam process.

Measurements were taken from the middle of the boards using a Greisinger GMH 3250 precision quick response thermometer (Regenstauf, Germany).

Production of medium density fiberboard

The MDF were produced at the Institute's own pilot plant (BINOS, Springe, Germany) using the newly integrated hot-air/hot-steam process. The hot-air/hot-steam unit with integrated pre-press (Fig. 1) was self-constructed using Leister Technologies (Aachen, Germany) for hot-air and MG Dampftechnik (Bedburg, Germany) for hot-steam, followed by a hot-press from Siempelkamp (Krefeld, Germany).

Twenty-two kilograms of wood fibers were used for each MDF production. The wood fibers were first blended at room temperature with 10 L of binder solution, containing 100 U laccase/g referred to absolutely dry wood fibers with or without 10 mM HBA/g referred to absolutely dry wood fibers, heat-inactivated LMS (cooked for 10 min), or 10% urea-formaldehyde resin. The horizontal blender system contained three injectors for spraying binders onto fibers. The blended fibers were conveyed to a tube dryer to reduce the fiber moisture content to approximately 8% with a temperature of 100 °C and pre-formed by a form conveyer. The handling procedure of blending, drying, and fleece generating took approximately 30 min.

The pre-formed fiber fleeces were immediately pre-pressed from approximately 400 to 100 mm and then treated with hot-air/hot-steam or without hot-air/hot-steam before final pressing in the hot-press (see parameters above). The target board thickness was 10 mm, with a raw density of 750 kg/m^3.

As this unit represents a new technology, the scheme is shown in Fig. 1.

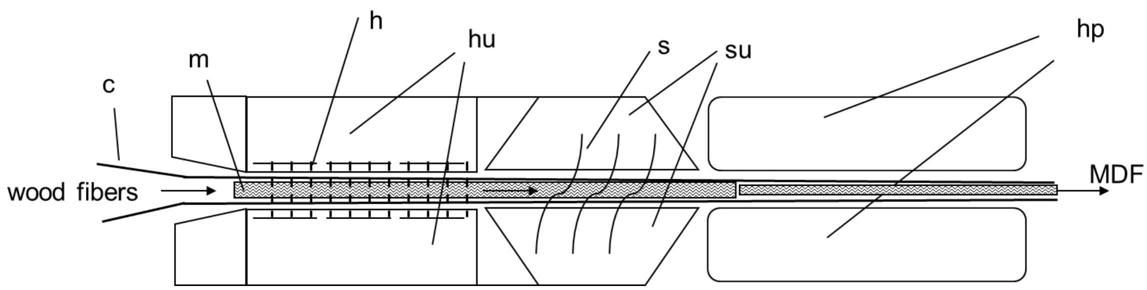

Fig. 1. Scheme of the new hot-air/hot-steam process. Wood fibers were treated with LMS, UF, laccase, or inactivated LMS and then transferred into the hot-air/hot-steam unit with integrated pre-press, followed by hot-press. Components and materials: c = conveyor, m = fibermat, h = hot air, hu = hot-air subunit, s = hot steam, su = hot-steam subunit, hp = hot-press

Each treatment was replicated six times. The testing of physical-technological properties was conducted for the internal bond strength (IB) of the boards in accordance with EN 319 (1993). The thickness swelling (TS) test was performed according to EN 317 (1993).

Pressing time optimization

To optimize the total pressing time, different MDFs were produced using the LMS according to the method described above. The final hot-pressing time was reduced systematically from 6 to 1 s/mm after the pre-heating hot-air/hot-steam process. Afterward, the IB and TS of MDF were tested considering EN 319 (1993) and EN 317 (1993).

RESULTS AND DISCUSSION

Temperature Gradient Measurements

It is known that high temperatures are required for MDF production, especially with natural binder systems such as LMS (Kües *et al.* 2007; Euring *et al.* 2015). MDF temperatures were measured during different pre-heating and pressing treatments lasting all together 12 s/mm (Fig. 2). The highest temperature measured was approximately 160 °C after 6 s/mm (5 s/mm hot-air + 1 s/mm hot-steam pre-heating) and remained stable until final hot-pressing was done. In comparison, fiber fleeces pre-heated with only hot air were heated up to approximately 140 °C after 4 s/mm and did not increase further. Using only hot-steam as pre-heating, the fiber fleeces were warmed up faster than without pre-heating, but the temperatures were slightly above of those that were only pressed by hot-pressing. The temperature decreased dramatically during pressing with the cold press.

These results reveal the high potential of the hot-air/hot-steam process to pre-heat the wood fiber fleeces effectively. As in the previous study by Euring *et al.* (2015), the sudden increase of the temperature from approximately 145 °C to over 160 °C at the moment the fiber fleece is heated with hot air and then with hot steam also occurs during MDF production.

This essential chemical-physical reaction will help to ensure the complete plasticization of LMS-activated wood fiber surface lignins (Euring *et al.* 2015). Recent analytical studies about lignin interactions will follow shortly. In addition, the hot-air/hot-steam process can be applied for any kind of binder systems in MDF production.

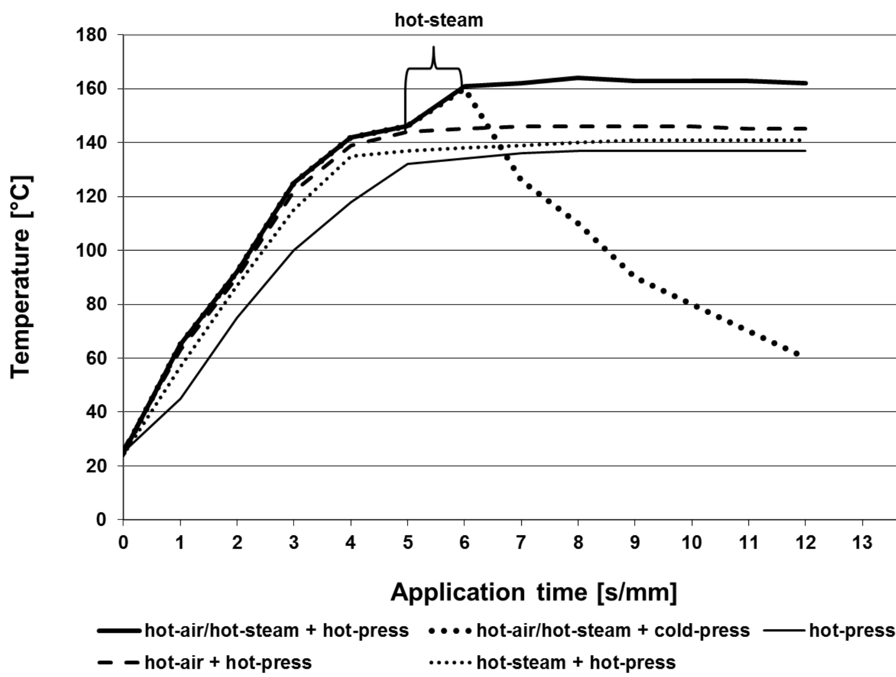

Fig. 2. Temperature gradient, measured in the middle of LMS-bonded MDF during various treatments

Physical-Technological Properties

The internal bond strength and thickness swelling tests showed hot-air/hot-steam treatment with subsequent hot-pressing improved the MDF board properties for both LMS and 10% UF binder systems (Table 1). Hot-air/hot-steam treatment before hot-pressing increased IB from 0.75 to 1.06 N/mm² for LMS and from 0.91 to 1.21 N/mm² for the UF system compared with only hot-pressing. All LMS-bonded MDF reached the required IB of 0.6 N/mm^2 using all methods with final hot-pressing (EN 622-5 (2010)). However, only with the hot-air/hot-steam plus hot press method did LMS-bonded MDF reach the required TS of 15% (EN 622-5 (2010)).

Cold-pressing after pre-heating resulted in low IB and high TS. These results show that the procedure to produce MDF only using the hot-air/hot-steam process is not effective enough, which is in contrast to the application for insulation boards with lower raw densities (Euring *et al.* 2015). For producing MDF, a final hot-pressing step at high temperature and pressing power is necessary to fulfill the required norms (Back 1987; Dunky and Niemz 2002; Irle and Barbu 2010).

Table 1. Internal Bond Strengths (IB) and Thickness Swellings (TS) of MDFs after various treatments

Sample	IB (N/mm^2)	TS after 24 h (%)	Hot-pressing time (s/mm)
LMS (hot-air/hot-steam + hot-press)	1.06 (±0.04)	15 (±2)	6
LMS (hot-air/hot-steam + cold-press)	0.49 (±0.05)	57 (±4)	6*
LMS (hot-press)	0.75 (±0.08)	18 (±3)	12
LMS (hot-air + hot-press)	0.72 (±0.06)	21 (±3)	7
LMS (hot-steam + hot-press)	0.77 (±0.07)	23 (±2)	11
10% UF (hot-air/hot-steam + hot-press)	1.21 (±0.09)	7 (±0)	6
10% UF (hot-air/hot-steam + cold-press)	0.61 (±0.03)	22 (±2)	6*
10% UF (hot-press)	0.91 (±0.08)	13 (±1)	12
10% UF (hot-air + hot-press)	0.78 (±0.04)	14 (±1)	7
10% UF (hot-steam + hot-press)	0.84 (±0.04)	15 (±2)	11
Laccase (hot-air/hot-steam + hot-press)	0.58 (±0.02)	52 (±5)	6
Laccase (hot-air/hot-steam + cold-press)	0.33 (±0.03)	67 (±7)	6*
Laccase (hot-press)	0.45 (±0.03)	70 (±6)	12
Laccase (hot-air + hot-press)	0.38 (±0.04)	65 (±5)	7
Laccase (hot-steam + hot-press)	0.47 (±0.04)	61 (±6)	11
inact. LMS (hot-air/hot-steam + hot-press)	0.22 (±0.02)	77 (±8)	6
inact. LMS (hot-air/hot-steam + cold-press)	0.11 (±0.02)	98 (±10)	6*
inact. LMS (hot-press)	0.17 (±0.03)	82 (±9)	12
inact. LMS (hot-air + hot-press)	0.15 (±0.02)	88 (±12)	7
inact. LMS (hot-steam + hot-press)	0.16 (±0.02)	81 (±11)	11

*Pressing with cold-press at room temperature
Note: requirements for 10-mm-thick MDF (EN 622-5 (2010)): min. 0.6 N/mm^2 IB and max. 15% TS

It becomes clear that hot-air or hot-steam treatment alone before hot-pressing could not improve the IB and TS properties, neither for LMS-treated MDF nor for 10% UF-bonded MDF. Therefore, hot air and hot steam have to be combined to operate together as an effective pre-heating system. The high temperatures during hot-air and hot-steam treatment lead to better bonding as the obvious result of an effective lignin-lignin crosslinking, even by using UF resin. Evaluating the IB and TS of laccase- and inactivated LMS-bonded MDF, the positive effect of hot-air/hot-steam pre-heating before hot-pressing can also be seen, apparently supported by the self-bonding mechanism of lignin (Bouajila *et al.* 2005; Zhang *et al.* 2015). Nevertheless, the values are not acceptable for the EN standard.

Pressing Time Optimization

For the MDF industry, the intention of a pre-heating system is to increase the effectiveness and productivity of board production (Irle and Barbu 2010). Generally, pre-heating causes a significant reduction of the pressing time in hot-presses. For that reason, in this study, the hot-pressing time was reduced from 6 to 1 s/mm and the IB and TS were tested. As binder systems, the LMS was chosen to show the effects of using a natural binder system, and UF resin as conventional binding system. It was found that the final hot-pressing time could be reduced from 6 to 3 s/mm without changing the IB and TS, which remained stable at approximately 1.0 N/mm^2 (LMS) and 1.2 N/mm^2 (UF), 15% (LMS) and 10 % (UF), respectively (Fig. 3).

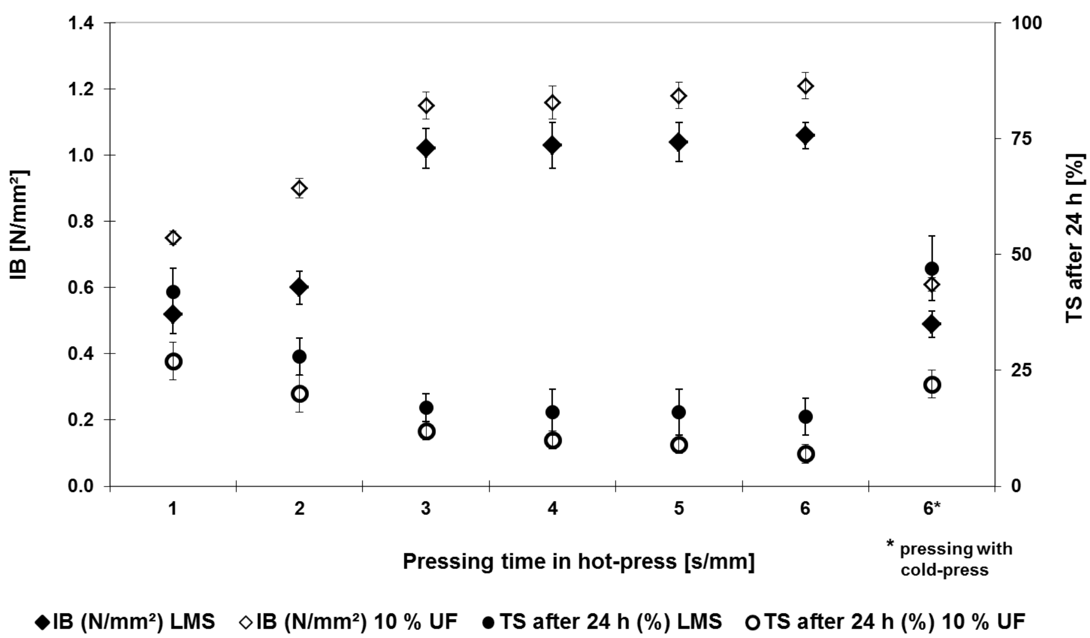

Fig. 3. IB and TS of LMS- and 10 % UF-bonded MDF, produced with various final hot-pressing times after pre-heating *via* hot-air/hot-steam steam process. Requirements for 10-mm-thick MDF (EN 622-5 (2010)): min. 0.6 N/mm^2 IB and max. 15% TS.

For comparison, the values for 6 s/mm cold-pressed MDF after pre-heating are also represented, showing too low IB and too high TS in both cases. The reduction of the pressing time from 12 to 9 s/mm, including pre-heating *via* hot-air/hot-steam process and final pressing *via* hot-press, implies a 25% time savings for the total pressing process. The physical-mechanical properties remain stable, confirming the high potential of the hot-air/hot-steam process for the production of MDF, especially using the natural binding system LMS and also conventional UF resin. The temperatures during pre-heating are high enough for an effective polymerization of the generated lignin fragments through LMS actions and self-bonding mechanisms for a final hot-pressing within short times.

CONCLUSIONS

1. The hot-air/hot-steam process is an effective pre-pressing and pre-heating system for MDF production that is able to reduce the total pressing time by 25%.
2. For the binding of wood fibers, the LMS is a suitable natural-based alternative to UF-resins, which are commonly applied for MDF production at present. Because of the high temperature during pre-heating, the hot-air/hot-steam process helps to polymerize the LMS-activated lignin more efficiently than with hot-pressing alone.
3. The physical-mechanical properties of both LMS-bonded MDF and UF resin-bonded MDF could be improved using the hot-air/hot-steam process.
4. The next steps for this research will be the adaption of the hot-air/hot-steam process at the industrial scale. The pre-heating system will also be tested for the production of particleboards (PB) and oriented strand boards (OSB).

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