

**PROPERTIES OF MEDIUM-DENSITY FIBREBOARDS
BONDED WITH DEXTRIN-BASED WOOD ADHESIVE**

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ABSTRACT

This study focuses on manufacturing of medium density fibreboard (MDF) panels bonded with dextrin-based wood adhesive and crosslinked in situ with various weight ratios of synthetic (e.g., polymeric-methane diphenyl-diisocyanate, pMDI) or bio-based (e.g., glyoxal) crosslinkers. The physical and mechanical properties of the panels were evaluated and compared with those from panels without crosslinker (control). Modulus of rupture (MOR) and internal bond (IB) strength of the MDF panels were considerably increased by increasing the crosslinkers' content. While, slight improvements were observed in modulus of elasticity (MOE) of the panels as a function of crosslinker type and content. Addition of crosslinkers clearly reduced the thickness swelling (TS) and water absorption (WA) of the panels, whereas, the panels with pMDI showed superior performances than the control and glyoxal added ones within 4 h and 24 h immersion in water. The results indicate the potential of dextrin as wood panel adhesive along with the use of appropriate crosslinkers.

KEYWORDS: Crosslinker, dextrin adhesive, glyoxal, mechanical properties, pMDI, thickness swelling.

INTRODUCTION

MDF is a wood-based composite product formed by combining wood fibres with a binder, generally synthetic resin, under heat and pressure. Aminoplastic adhesives are the most common adhesives in the production of MDF, which are mainly based on the reaction of formaldehyde with urea or urea-melamine co-condensates. In the past years, the legal requirements for both work environment and final product emissions have been steadily becoming stricter due to the increasing concerns related to the potential harm of formaldehyde emissions to human health (Hemmila et al. 2017, Cheng et al. 2017). Along with the increasing efforts of the industry to be less fossil fuel dependent, this has led to a great demand for developing environmentally friendly wood adhesives from renewable resources such as lignin, tannins, proteins, and starch.

Starch is used within a wide range of products including coating binders, sizing materials, wet-end additive, food, and pastes (Cheng et al. 2017, Tharanathan 2005, Wang et al. 2013, Zhang et al. 2015). The bonding strength, water resistance and storage performance of native starch, however, cannot meet the requirements for serving as an adhesive in wood-based composite products, and thus its molecular structure must be strengthened. A number of studies have been focused on its chemical modification including crosslinking, grafting copolymerization, oxidation and esterification to improve the performances of starch-based adhesives (Cheng et al. 2017, Wang et al. 2013, Zhang et al. 2015, Sun et al. 2018, Li et al. 2014, Imam et al. 2001, Hosseinpourpia et al. 2018). Among other methods, crosslinking is mostly applied to improve the functional properties of starch (Kou and Gao 2018). Sridach et al. (2013) reported that the thermal properties and mechanical strength of tapioca starch adhesive were improved by crosslinking with hexamethoxymethyl-melamine and citric acid. Shear strengths of corn starch adhesive grafted with polyvinyl acetate measured under dry and humid conditions were remarkably increased by epoxy resin crosslinker (Nie et al. 2013). Plywood manufactured with starch adhesive crosslinked with polymeric-methylene diphenyl-diisocyanate (pMDI) also exhibited considerably higher wet bonding strength than plywood glued with unmodified starch (Gu et al. 2010). pMDI is a reactive compound that is commonly used as a sole adhesive in wood panels manufacturing or as a crosslinker to enhance the bonding quality in natural polymers, however, it is very expensive and needs to be used in stabilized form due to its high reactivity towards moisture (Hemmilä et al. 2017).

Glyoxal (OHC-CHO) is a nontoxic, non-volatile, low cost and biodegradable compound that can be obtained from several natural sources such as from the oxidation of lipids or as a by-product of biological processes (Hirayama et al. 1984, Deng et al. 2018). It has been previously used as a crosslinker to improve the mechanical strength and water resistant of bio-based wood adhesives such as lignin-, tannin- and soy protein-based (Ballerini et al. 2005, Lei et al. 2008, Amaral-Labat et al. 2008, El Mansouri et al. 2007). Moisture resistance of starch-coated paper was enhanced after crosslinking with glyoxal (Lin et al. 2017). Crosslinking of starch foam with glyoxal decreased its water absorption and increased its tensile and flexural strengths (Uslu and Polat 2012).

Dextrin is a low molecular weight carbohydrate that can be obtained through the dry roasting of starch in the presence of an acid catalyst (Hemmila et al. 2017). It was previously applied to improve the initial adhesion (tack strength) of phenol formaldehyde resin in wood composite manufacturing (Sahaf et al. 2012). However, the literature about dextrin-based wood adhesives is rather scarce. Therefore, the present study aimed at evaluating the physical and mechanical properties of MDF panels bonded with dextrin-based adhesive crosslinked in situ with synthetic (e.g., pMDI) and bio-based (e.g., glyoxal) crosslinkers. This approach might be

useful in developing novel wood panel adhesives based on a backbone of natural origin (dextrin) and combined with synthetic and bio-based crosslinkers.

MATERIAL AND METHODS

Wood fibres (*Pinus sylvestris*) obtained from thermo-mechanical-pulping (TMP) were kindly provided by Steico AG, Czarnkow, Poland. Polymeric-methylene diphenyl-diisocyanate (pMDI) adhesive (I-Bond PBEM 4352) was purchased from Huntsman (Everbeg, Belgium). Glyoxal (~40% solid content) was kindly provided by BASF SE (Ludwigshafen, Germany). Short-branch pea dextrin was supplied by Emsland-Stärke GmbH (Emlichheim, Germany).

Fibreboard production

MDF panels were manufactured by standardized procedures that simulated industrial production in the laboratory, as described previously (Hosseinpourpia et al. 2017). Dextrin-based adhesive was prepared by dissolving the short-branch pea dextrin (140 g) in distilled water (200 ml) at 60°C for 1h. The dry fibres (approximately 5% moisture content) were first resinated with a load ($\text{wt}_{\text{dry resin}}/\text{wt}_{\text{dry fibre}}$) of 15% dextrin-based adhesive for 5 min, and then were further blended for 5 min with glyoxal and pMDI crosslinkers to establish various weight ratios of dextrin:glyoxal (OH:CHO of 1:0.1, 1:0.3 and 1:0.6) and of dextrin:pMDI (OH:NCO of 1:0.1, 1:0.3 and 1:0.6), as presented in Tab. 1.

Tab. 1: Weight ratio of glyoxal and pMDI crosslinkers to dextrin-based wood adhesive and mean densities of the MDF panels (\pm standard deviation).

Sample code	Weight ratios of the crosslinkers		Density (kg·m ⁻³)
	OH _{dextrin} :CHO _{glyoxal}	OH _{dextrin} :NCO _{pMDI}	
Dex	-	-	650 ± 26
Dex-Gly1	1:0.1	-	644 ± 32
Dex-Gly2	1:0.3	-	661 ± 19
Dex-Gly3	1:0.6	-	641 ± 27
Dex-pM1	-	1:0.1	663 ± 16
Dex-pM2	-	1:0.3	652 ± 25
Dex-pM3	-	1:0.6	657 ± 22

Thereafter, a fibre mat was formed by hand (450 × 450 mm) and cold pre-pressed. Hot-pressing was performed at 200°C using 11.7 mm stops in a single daylight hot press. The pressing time was set to 40 s·mm⁻¹. After hot-pressing, the MDF panels were cooled to room temperature, cut to 400 × 400 mm dimensions and sanded to a thickness of 10.0 mm using a band sander. Tab. 1 also presents the densities of the final panels. The panels were then cut into various test pieces according to the respective standards for evaluating physical and mechanical properties, as described below. Prior to testing, all samples were conditioned in a climate chamber at 65% RH and 20°C for 14 days.

Testing of the MDF panel properties

Three MDF panels were used to evaluate the physical and mechanical properties for each gluing system. Bending test was performed to determine the MOR and MOE of the MDF panels (4 samples per board, n = 12) according to EN 310 (1993) using a Zwick 010 testing machine

(Zwick GmbH & Co. KG, Germany). Rectangular samples measuring 400 x 50 cm were tested using a span of 200 mm and a cross-head speed of 1.8 mm·min⁻¹. The portion between 20 and 40% maximum load was considered for measuring the MOE.

The IB strength test or tensile strength perpendicular to the surface of panels was conducted following EN 319 (1993). Five samples measuring 5 × 5 cm per MDF panel (n= 15) were effectively bonded with a hot-melt glue and the tension test were performed using the Zwick machine. A loading speed 8 mm·min⁻¹ was used for testing.

The water related properties of the MDF panels were determined using 5 × 5 cm samples immersed in water. The TS and WA were assessed after 2 and 24 h of water soaking, respectively. The TS (%) was evaluated by the difference between the final and initial thickness, according to EN 317 (1993) using 5 samples per panel (n= 15).

Statistical analysis

One-way analysis of variance (ANOVA) was performed by means of IBM SPSS Statistics24. The statistical differences between the values were evaluated by Tukey's honestly significant difference at an error probability of $\alpha = 0.05$ (Adamopoulos et al. 2015, Hosseinpourpia et al. 2019).

RESULTS AND DISCUSSION

Fig. 1 illustrates the MOR (Fig. 1a) and MOE (Fig. 1b) for the fibreboards bonded with the dextrin-based adhesive as a function of the synthetic (pMDI) and bio-based (glyoxal) crosslinkers' content.

It was observed that the MOR in panels containing glyoxal and pMDI increased with the increase of crosslinkers' weight ratio. Addition of crosslinkers increased significantly MOR as compared with the controls (Dex), with the exception of the lower glyoxal content (Dex-Gly1). At each respective weight ratio, pMDI enhanced the MOR of the panels in a greater extent than glyoxal. According to EN 622-5 (2009), the minimum requirements for MOR of MDF panels for indoor applications is 22 N·mm⁻². All boards were above this limit except for those bonded with dextrin alone (Dex), and with dextrin and lower amount of glyoxal (Dex-Gly1). The MOE of the panels, in contrast, was less affected by the addition of crosslinkers, as shown in Fig. 1b.

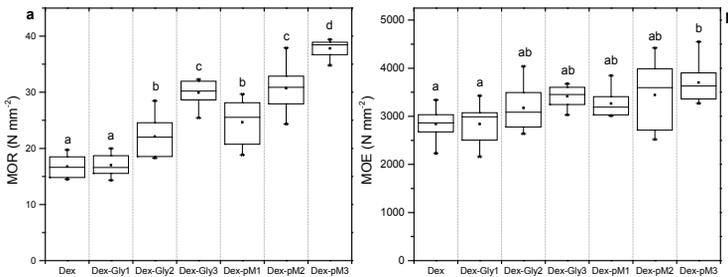


Fig. 1: (a) MOR and (b) MOE of the MDF panels. Box-plots with whiskers from the minimum to the maximum; the box represents the 25%, 50% and 75% quartile; the mean value of each data set is depicted as quadrate inside the box. The indicators were statistically tested with ANOVA and Tukey's HSD test. Values labelled with different letter are statistically different at an error probability of $\alpha=0.05$, $n=12$.

When referring to EN 622-5 (2009), all tested panels independently from the crosslinker type and content surpassed the minimum specification MOE value of $2500 \text{ N}\cdot\text{mm}^{-2}$ recommended for MDF panels for indoor applications. The MDF panels bonded with sole dextrin (Dex) displayed a MOE value of $2800 \text{ N}\cdot\text{mm}^{-2}$. The highest MOE values of 3415 and $3700 \text{ N}\cdot\text{mm}^{-2}$ were obtained from panels bonded with dextrin-based adhesive crosslinked with the highest amount of glyoxal (Dex-Gly3) and pMDI (Dex-pM3), and were respectively 36.6% and 48% higher than the standard value. Only panels crosslinked with the highest pMDI weight ratio (Dex-pM3) exhibited a statistically significant increment in MOE value than the controls (ANOVA, $\alpha=0.05$).

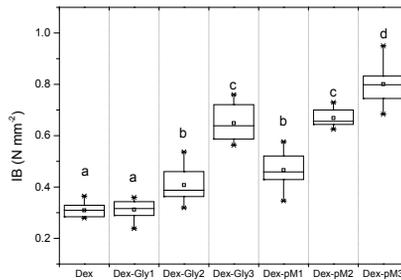


Fig. 2: IB strength of the MDF panels. Box-plots with whiskers from the minimum to the maximum; the box represents the 25%, 50% and 75% quartile; the mean value of each data set is depicted as quadrate inside the box. The indicators were statistically tested with ANOVA and Tukey's HSD test. Values labelled with different letter are statistically different at an error probability.

The addition of crosslinkers in the dextrin-based glue systems had exactly the same effect on the IB strength of the MDF panels like on their MOR. In detail, the IB strength significantly increased with the increase of crosslinkers' weight ratios (Fig. 2), except for those made of dextrin adhesive and low glyoxal content (Dex-Gly1).

The IB values of the panels crosslinked with highest glyoxal weight ratio (Dex-Gly3) and of the boards crosslinked with intermediate and highest content of pMDI (Dex-pM1 and Dex-pM2) exceeded the minimum specified standard value of $0.6 \text{ N}\cdot\text{mm}^{-2}$ for IB strength, which is recommended by EN 622-5 (2009) for MDF panels designed for indoor applications. The linear correlation observed between MOR and IB strength of the MDF panels as a function of crosslinkers' addition with increasing weight ratios, indicated the close interrelation between these two parameters (Fig. 3).

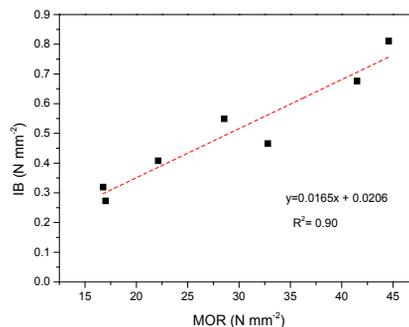


Fig. 3: Linear relationship between IB strength and MOR of the MDF panels.

Although, the addition of glyoxal significantly enhanced the mechanical strength of the MDF panels bonded with dextrin adhesive, it was inferior to that with pMDI at comparable contents. This might be due to the higher mobility of pMDI on the surface of the fibres that previously bonded with dextrin-based adhesive as compared to glyoxal (Younesi-Kordkheili and Pizzi 2018, Mai et al. 2017). pMDI has been used as a wood adhesive for many years and is known for its outstanding bonding strength in the wood-based panels (Papadopoulos et al. 2002). Therefore, the isocyanate groups in pMDI react with the hydroxyl groups both in the wood fibres and the dextrin-based adhesive, and thus provide a stronger mechanical strength as compared to glyoxal. Moreover, the two aldehyde groups in glyoxal are very close to each other, which reduce the mobility of the glyoxal crosslinked products (Rojas and Azevedo, 2011), and may resulted in low mechanical strength of glyoxal crosslinked panels. Younesi-Kordkheili et al. (2018) most recently reported that the addition of pMDI improved the mechanical strength of wood-based panels bonded with lignin-urea-formaldehyde resin.

The TS and WA of the MDF panels bonded with different dextrin-based glue systems are presented in Fig. 4a and Fig. 4b.

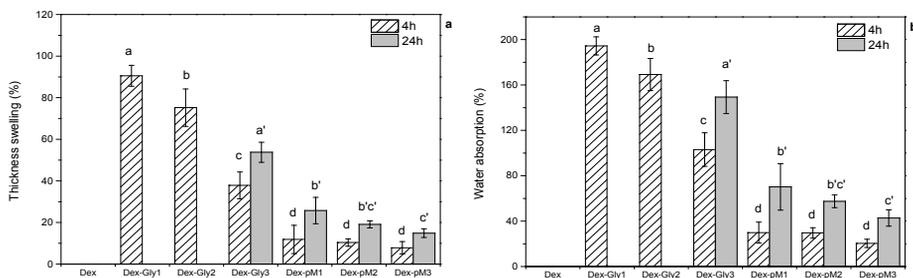


Fig. 4: TS (a) and WA (b) of MDF panels after 4 h and 24 h measuring time. Mean values \pm standard deviations. The indicators were statistically tested with ANOVA and Tukey's HSD test. Values labelled with different letter are statistically different at an error probability of $\alpha=0.05$, $n=15$.

The control samples (Dex) deteriorated after short-term (4h) immersion in water, which should be due to the high sensitivity of dextrin-based adhesive towards water. The addition of glyoxal enhanced the short-term water resistance of the samples, although only the samples containing the highest concentration of glyoxal (Dex-Gly3) were able to be measured after 24 h. The poor water resistance of the panels containing low concentration of glyoxal might be related to the replacement of hemiacetal bonds with hydrogen bonds in the presence of water (Rojas and Azevedo 2011). Norström et al. (2015) also quoted that glyoxal had no considerable effect on the water resistance of xylan adhesive.

The TS and WA of the fibreboards were improved by increasing the amount of pMDI. According to EN 622-5 (2009), the maximum thickness swelling within 24 h of MDF panels for indoor applications is 20%. The MDF boards crosslinked with intermediate and highest content of pMDI (Dex-pM1 and Dex-pM2) fulfilled the requirement. As described previously, the isocyanate groups in pMDI are highly unsaturated and can react with a number of active hydrogen compounds such as alcohol or amine groups (Sonnenschein 2015). The presence of plenty active hydroxyl groups in fibres' surface and dextrin structure, and also in moisture of fibre and glue system will lead the reaction with isocyanate, which will result in the formation of polyurethane and polyurea linkage, respectively. Allophanate and/or biuret bonds will generate

in the reaction system due to the presence of excess amount of primary amino-groups at elevated temperature (Sonnenschein 2015, Chen and Yan 2018). The above mentioned chemical reactions are seem to be more stable as compared to the hemiacetal bonds that created between the aldehyde (-CHO) groups in glyoxal and the hydroxyl groups.

CONCLUSIONS

The potential of using dextrin-based adhesive crosslinked in situ with glyoxal and pMDI for manufacturing MDF panels was evaluated. By increasing the crosslinkers' content, MOR and IB strength of the panels were considerably increased. This confirms a good bonding quality when using crosslinkers than dextrin alone. MOE values of the MDF panels were slightly improved by adding the synthetic and bio-based crosslinkers, and all panels reached values above the standard requirements for indoor uses. TS and WA of the panels obviously reduced by increasing crosslinkers' content, while the panels with intermediate and high amount of pMDI exceeded the minimum requirement of the standard. Overall, the MDF panels crosslinked with pMDI exhibited superior physical and mechanical properties compared to the ones glyoxal-crosslinked and the controls. Nevertheless, using a high amount of glyoxal crosslinker (Dex-Gly3) could generate environmentally friendly panels with good mechanical performances, although their water-related properties still need to be improved.

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