

Canola Meal Adhesive for the Production of Wood Fiber Insulation Boards Using Hot-Air/Hot-Steam-Process

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Abstract

Canola meal as a by-product from the vegetable oil production provides a protein-rich material which is available in large quantities but with limited areas for application. The objective of this study was to investigate the possibility of utilizing canola meal adhesive for the production of wood fiber insulation boards (WFI) using the hot-air/hot-steam-process. WFI with two different thicknesses (40/60 mm) and different densities (110/140/160/180 kg/m³) were manufactured. The testing focused on their physical-mechanical properties such as internal bond strength (IB), compressive strength (CS) and short-term water absorption (ST-WA) measured according to European standards. For a better understanding of the material and curing dynamics, the canola meal was analyzed on its protein content, lignin and pentosane content as well as its extractives content using hot water, cold water and successive extraction. Using a canola meal based adhesive resulted in promising results for IB and CS up to density of 140 kg/m³. Nonetheless, there is place for improvement for the ST-WA.

Keywords: Adhesive, Canola Meal, Hot-Air/Hot-Steam-Process, Physical Mechanical Properties, Protein, Wood Fiber Insulation Board

1. Introduction

Insulation materials made from wood fibers can provide a resource-saving alternative to materials such as mineral fibers, expanded glass, foam glass, calcium silicate, perlite, expanded clay, natural pumice, rock wool, polystyrene or sheep's wool (Eichhorn, 2017). The material wood is in itself a hygroscopic material (Lundgren, 1958). Once the moisture content of wood exceeds 20% of total weight, its insulating properties are lost (Informationsdienst Holz, 2007). The usage should therefore be limited on areas where no permanent higher moisture content is to be expected. In order to reduce the water absorption, polymeric diphenylmethane diisocyanate (pMDI) is currently mainly used as a binder for the industrial production of wood fiber insulation boards (WFI), in particular, through the ability of forming strong and durable covalent bonds with wood components (Zhou & Frazier, 2001; Frihart, 2013).

However, pMDI and other polyurethanes (PU) on the market are based on fossil fuels (Tsuomis, 1991; Türk, 2014). In addition, pMDI is considered problematic in terms of processing since it adheres to almost all surfaces and thus, a release agent is required during processing. Furthermore, pMDI is classified as harmful to health: vapors, gases, aerosols and dust particles can cause respiratory problems, skin contact can lead to allergic reactions and bronchial hypersensitivity (Bundeministerium für Gesundheit, 2004). Besides the sensitizing effect according to TRGS 401 there is also the possibility of contact eczema (Bundesanstalt für Arbeitsschutz und Arbeitsmedizin, 2009). For this reason, both industry and science are searching for equivalent, alternative binders.

One possible opportunity is to use plant proteins derived from canola. There are several studies using modified and unmodified protein isolates (Li et al., 2012; 2017; Hale, 2013; Wang et al., 2014; Bandara et al., 2017) or canola meal together with phenol-formaldehyde-resin (Yang et al., 2010; 2011; 2012; 2014) as adhesive for wood or wood composites. The advantage of using canola meal is to avoid expensive, low-yield and chemical-rich protein isolation (Elstner & Stein 1982). Therefore, this study focusses on the usage of solubilized

canola meal as adhesive for the production of WFI for the first time by means of using the innovative hot-air/hot-steam-curing-technology developed by Euring and Kharazipour (2013).

As the third largest oil seed crop (McVetty & Duncan, 2015), canola seeds provides 40% oils and 60% seed meal with a protein content between 35-40 % (Raymer, 2002; Booth & Gunstone, 2004). The protein composition consists of 50% cruciferin, 20-40% napin and up to 8% oleosin (Von der Haar et al., 2014). While napin constitutes a water-soluble albumin with a secondary structure, cruciferin is a more complex salt-soluble globulin with a quaternary structure (Fahs & Louarn, 2013; Von der Haar et al., 2014). The annual (2019) worldwide production of canola seeds or rapeseed is 70.5 Mio. t (FAOSTAT 2020). This equivalents to a production of 45.7 Mio. t. of seed meal (60%) as by-product and is therefore available in large quantities. According to a study from van Zeist et al. (2012) the costs amounts to 213 \$/t for canola meal.

Canola meal consists about 46.5% of carbon from photosynthesis (Peterson & Hustrulid, 1997). Wooden biomass stores about 50% of total weight in carbon (Lamlom & Savidge, 2003). Burning or feeding canola meal would end up in neutral carbon food print, whilst using wood fibers and canola meal for insulation purpose, the amount of carbon would remain stored within its whole utilization period.

2. Material and Methods

The native canola expeller used in this study was obtained by Kleeschulte GmbH & Co. KG (Büren, Germany). The used TMP wood fibers with a moisture content of 10-12% were obtained by GUTEX Holzfaserplattenwerk H. Henselmann GmbH & Co. KG (Waldshut-Tiengen, Germany) which constitutes a mixture of 80% norway spruce (*Picea abies*) and 20% silver fir (*Abies alba*).

2.1 Canola Meal Adhesive Preparation

In the first step, the canola meal adhesive was prepared. Therefore, the canola expeller was crushed to about <5 mm sized pieces and subsequently sieved. The green-yellowish meal fractions <400 µm were used for adhesive production, whereas bigger fractions were discarded containing mostly hull fragments and crude fiber. To evaluate the canola meal characteristics, nitrogen analysis according to Kjeldahl was carried out as well as lignin analysis according to TAPPI standard (1985), hot and cold water extractions, successive extraction as well as pentosane content determination. The adhesive was produced by mixing 17.5% canola meal together with 17.5% urea, 50% water and 15% sodium hydroxide (1M) solution. After stirring for about 20 minutes the relatively high viscous dispersion with total solids content of 35% and pH value of 12.5 was ready to use. In order to enhance the hydrophobicity of the produced boards, 2% (based on absolute dry wood fibers) of a paraffin emulsion (HydroWax Syntec Blue, Sasol Germany GmbH, Hamburg, Germany) was added to the canola meal dispersion.

2.2 Manufacture of Wood Fiber Insulation Boards (WFI)

In the second step, the adhesive mixture was added to the fibers on a resin load of 15% (solids) based on absolute dry wood fibers. By means of an atomizer spray nozzle, the adhesive was blended with wood fibers at a blending unit of the institutes own pilot plant for MDF production. Then, the treated fibers were loosened and dried via flash tube dryer at 50°C to a moisture content of 16-17%. The fibers were then compressed to 40 mm, or 60 mm, respectively, and were then transferred to the hot-air/hot-steam unit. The curing of WFI was done like described in Ostendorf et al. (2020) with temperatures of 160-170°C. As reference, WFI were manufactured with 4% pMDI using same treatment. Following the industrial technology, a further pMDI reference was produced using merely hot-steam injection for about 60s according to Ostendorf et al. (2020). The attempt of curing canola adhesive bonded fibers with solely hot-steam injection failed due to insufficient hardening of the boards.

For each WFI variant, three boards were manufactured and conditioned to equilibrium moisture content at 23°C and 50% RH. Afterwards the edges of the WFI were trimmed and test sample were cut-to-size according to the EN standards. The samples were analyzed conferring to the following physical-mechanical properties: internal bond (IB) strength test (EN 1607 2013), compressive strength (CS) test (EN 826 2013) and short term (24h) water absorption (ST-WA) during partial immersion (EN 1609 2013). Furthermore, samples of cured WFI were analyzed using cold and hot water extraction to evaluate changes in pH and extractives content when treating fibers with canola meal adhesive.

3. Results and Discussion

3.1 Canola Meal Characteristics

The results for the canola meal analysis are shown in table (1). The protein content for meal fractions <400 µm according to the Kjeldahl method is 36.7%. In comparison, the original expeller shows a protein content of 30.6% (Landwirtschaftliche Untersuchungs- und Forschungsanstalt Nordrhein-Westfalen, 2018). Thus, by

grinding and sieving, it is possible to increase protein content by removing unwanted components such as residues of hull fragments. This can be confirmed by the very low measured lignin content (0.7%) of the meal. In contrast, a study of Briones et al. (2010) determined a lignin content of up to 16% for canola meal. This distinct difference in lignin content indicates, that most part of hull fragments were removed by sieving canola meal, considering, that the hulls cover 95% of canola lignin content (Carré et al., 2016).

A pentosane content of 2.8% is measured for canola meal. A study by Clandinin and Rao (1970) found a pentosane content of 8.3% for canola meals, showing a reduction by milling and sieving canola expeller. By extracting canola meal with petrol ether, 13.1% of lipids and fatty acids are removed which is mostly congruent with the value for expeller (13.9%, Landwirtschaftliche Untersuchungs- und Forschungsanstalt Nordrhein-Westfalen, 2018). Within this successive extraction method, 22.2% extractives are determined. In contrast, by extracting with merely cold or hot water respectively, extractives of 23.0% or 27.8% are detected. The pH measurement for the liquid phase of the mentioned extractions show pH values of 5.7 (cold) and 5.3 (hot), therefore in the acid range. The question occurs, if the measured components besides protein have an influence of the gluing properties on the canola meal adhesive. The strong alkaline treatment with NaOH might lead to a decomposition of the components as stated by Yang et al. (2014) which might end up in losing additional adhesive effects (e.g. holocelluloses).

When canola meal is mixed together with water, urea and NaOH, it is necessary to generate a sort of fluid and sprayable solution. Therefore, this study focused on the usage of <400 μm fractions due to fact, that water uptake increases with decreasing particle sizes (Ranjbar, 2013). Using smaller particle sizes, for instance <200 μm , would consequently end up in an unsuitable not-sprayable slurry.

Table 1. Results for canola meal analysis

| | | Canola meal (<400 μm) |
|-----------------------------|-----------------|-----------------------------------|
| Moisture content (%) | | 8.5 |
| Pentosane (%) | | 2.3 |
| Lignin (%) | | 0.7 |
| Nitrogen (N) content (%) | | 5.9 |
| Protein (N6.25) content (%) | | 36.7 |
| Hot water-extraction | pH | 5.3 |
| | Extractives (%) | 27.8 |
| Cold water-extraction | pH | 5.7 |
| | Extractives (%) | 23.0 |
| Successive extraction (%) | Petrol ether | 13.1 |
| | Diethyl ether | 0.4 |
| | Acetone-water | 4.5 |
| | Ethanol-water | 4.2 |
| | | Σ 22.2 |

3.2 Analysis of Wood Fiber

The results of the analysis of WFI specimen treated with alkaline canola meal adhesive and native wood fibers are shown in figure (1). The pH changes from acid into neutral range (6.94) for cured WFI in comparison to native wood fibers. It is anticipated, that canola proteins are solubilized in alkaline environment (Li et al., 2011; Hale, 2013). Furthermore, the proteins starts to unfold and their contact area increased (Hale, 2013; Li et al., 2017). The authors assume that, when it comes to contact with acidic wood fibers, the proteins precipitate on fiber surfaces. By subsequently compressing and high temperature treatment within hot-air/hot-steam-process, the protein cures by denaturation and interaction with wood fibers. Since cruciferin has a higher molecular weight than napin and therefore a higher unfolding-length, it is considered to have a higher adhesion strength (Fahs & Louarn 2013).

The content of extractives is more than tripled by the extractive-rich adhesive; also considering additional 2% hydrophobic agent (solids content = 55%) within the adhesive formulation. With a pH-neutral ranged panel

product, an interaction with pH-sensitive construction materials like iron, where the corrosion rate increases with $\text{pH} > 9$ (Kaesche, 1970), is less critical.

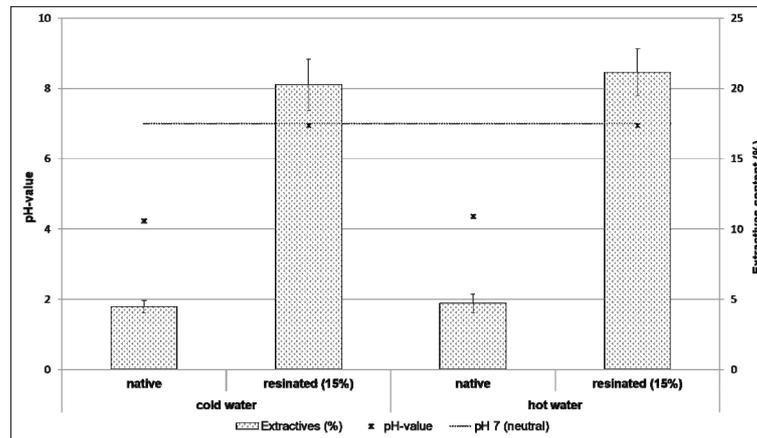


Figure 1. pH-value and extractives content of native wood fibers and cured WFI using 15% canola meal adhesive after cold and hot water extraction ($n=3$)

3.3 Physical-Mechanical Properties

The results for the physical-mechanical properties of the WFI are summarized in table (2). The general observations are a decrease in IB and CS strength as well as decreased ST-WA with reducing densities, which constitutes a well-known correlation between density and physical-mechanical properties (Niemz & Wagenführ, 2018). Moreover, there are no indicative changes in physical-mechanical properties between two different panel thicknesses of 40 and 60 mm.

The results show that the canola meal bonded WFI are comparable to the pMDI-references cured via hot-steam and to the GUTEX industrial products regarding IB and CS. Exceptions are results for CS at lowest density of 110 kg/m^3 (44.9 kPa for 40 mm and 43.6 kPa for 60 mm). The denaturation temperature of canola protein is about 150°C (Hale, 2013). Hale (2013) also found that, when press temperature increasingly distinct from denaturation temperature, higher adhesion strength occurs due to enhanced interface reaction between wood and proteins. Therefore, the innovative hot-air/hot-steam-process is the proper way to cure natural based adhesives for the production of WFI (Euring et al., 2015; Kirsch et al., 2018; Ostendorf et al., 2020).

In contrast, the pMDI reference cured via hot-air/hot-steam, achieved best results over all conducted tests with the exception of the variants with density of 180 kg/m^3 and board thicknesses of 40 and 60 mm. Here, the canola meal bonded boards show impressive 328.5 kPa of 60 mm thick boards. The enhancement of panel quality by using hot-air/hot-steam-process and pMDI as binder is well documented in several studies (Euring & Kharazipour, 2013; Euring et al., 2015; Kirsch et al., 2018; Ostendorf et al., 2020).

The high ST-WA over all densities and thicknesses is problematic, despite the addition of 2% hydrophobic agent. Frihart and Birkeland (2014) found that carbohydrates in soy flour adhesives are responsible for poor water resistance of protein-to-wood-bonding. Since canola meal consist of up to 50% of carbohydrates (Bell 1984), a negative impact on ST-WA has to be considered. Additionally, NaOH as highly hygroscopic substance is able to absorb humidity from the surrounding environment which can be accumulated between wood fibers and therefore affect the water absorption (Hollemann et al., 2017). On the other hand, the addition of NaOH is an important measure to hydrate and properly unfold the canola protein up to tertiary structure, to expose the functional groups for enhanced interaction between wood and protein (Hale, 2013; Li et al., 2017). This can be explained by polar and non-polar groups of the protein chains which are able to interact with polar hydroxyl-groups of wood. Thereby, the non-polar groups are considered as hydrophobic. The more hydrophobic groups are present, the stronger and more water-resistant is the protein-wood-bonding. Unfortunately, canola provides a low hydrophobic protein (Li et al., 2017). Sun (2005) found that soy protein isolate modification using urea leads to higher amount of water resistant bonds. As soy protein is expected to have a similar functionality as canola (Li et al., 2012), urea is used in this study as further modifying agent besides NaOH. Nonetheless, a beneficial effect might be disabled by the presence of carbohydrates, which is presented in the study of Frihart and Birkeland (2014) for soyflour adhesives. This might be due to steric hindrance or the high

viscosity of the flour based adhesive (Frihart & Birkeland, 2014). The measured oil content of the used canola meal of 13.1% might have a beneficial effect on water absorption. Nonetheless, the results show that the occurred protein-wood-bonding, mostly characterized by electrostatic interactions, hydrogen bonds and van der Waals forces (Fahs & Louarn, 2013), are sufficient for good initial mechanical properties (IB and CS). For further investigations, the addition of crosslinking-agents such as formaldehyde or glyoxal should be considered to improve water resistance as well as mechanical properties.

Table 1. Properties (Internal bond strength (IB), compressive strength (CS) and short-term water absorption (ST-WA) of canola meal bonded WFI in comparison to pMDI bonded WFI and industrially manufactured product. The standard deviation is shown in parentheses

| Density (kg/m ³) | Variant | Thickness (mm) | IB (kPa) <i>n</i> =30 | CS (kPa) <i>n</i> =24 | ST-WA (kg m ⁻²) <i>n</i> =9 |
|---------------------------------|-------------------------|-------------------|-----------------------------|-----------------------------|---|
| 110 | Canola | 40 | 5.3 (±1.0) | 44.9 (±14.2) | 3.3 (±0.9) |
| | | 60 | 5.0 (±1.4) | 43.6 (±9.2) | 1.3 (±0.4) |
| | pMDI HS ^a | 40 | 4.3 (±1.1) | 48.1 (±17.7) | 1.2 (±0.8) |
| | | 60 | 5.9 (±0.8) | 57.0 (±9.6) | 1.3 (±0.4) |
| | pMDI HA/HS ^b | 40 | 5.7 (±2.0) | 49.5 (±19.7) | 0.5 (±0.1) |
| | | 60 | 11.9 (±4.7) | 81.3 (±9.7) | 0.6 (±0.1) |
| | GUTEX ^c | 40/60 | 5 | 50 | ≤2 |
| 140 | Canola | 40 | 7.8 (±1.6) | 74.5 (±17.5) | 3.7 (±0.9) |
| | | 60 | 11.5 (±1.8) | 85.8 (±16.2) | 3.5 (±1.3) |
| | pMDI HS | 40 | 8.4 (±3.1) | 80.2 (±19.1) | 1.0 (±0.2) |
| | | 60 | 13.5 (±4.4) | 124.5 (±15.1) | 2.2 (±0.6) |
| | pMDI HA/HS | 40 | 19.4 (±2.3) | 140.2 (±14.7) | 0.6 (±0.1) |
| | | 60 | 14.3 (±4.8) | 146.9 (±14.6) | 0.9 (±0.1) |
| | GUTEX ^d | 40/60 | 7.5 | 70 | ≤2 |
| 160 | Canola | 40 | 12.7 (±3.7) | 119.2 (±16.0) | 4.6 (±1.2) |
| | | 60 | 15.8 (±3.9) | 128.5 (±16.3) | 4.2 (±1.3) |
| | pMDI HS | 40 | 25.2 (±6.2) | 146.6 (±35.3) | 1.4 (±0.8) |
| | | 60 | 18.3 (±2.7) | 122.7 (±15.8) | 1.7 (±0.5) |
| | pMDI HA/HS | 40 | 25.9 (±2.8) | 192.1 (±21.7) | 0.9 (±0.2) |
| | | 60 | 26.6 (±3.1) | 168.1 (±15.8) | 0.9 (±0.3) |
| | GUTEX ^e | 40/60 | 10 | 100 | ≤1 |
| 180 | Canola | 40 | 24.2 (±4.1) | 240.6 (±55.4) | 4.8 (±1.3) |
| | | 60 | 24.0 (±2.8) | 328.5 (±34.3) | 4.5 (±1.4) |
| | pMDI HS | 40 | 28.6 (±5.8) | 181.1 (±33.2) | 3.4 (±0.2) |
| | | 60 | 27.4 (±2.9) | 207.4 (±39.7) | 1.8 (±1.1) |
| | pMDI HA/HS | 40 | 31.1 (±6.5) | 208.7 (±42.5) | 0.9 (±0.1) |
| | | 60 | 31.5 (±4.3) | 255.0 (±37.9) | 0.9 (±0.1) |
| | GUTEX ^f | 40/60 | 20 | 150 | ≤1 |

^apMDI-reference cured via hot-steam (HS). ^bpMDI-reference cured via hot-air/hot-steam (HA/HS). ^cGUTEX-reference product Thermosafe homogen[®]. ^dGUTEX-reference product Multitherm[®]. ^eGUTEX-reference product Thermowall[®]. ^fGUTEX-reference product Ultratherm[®].

4. Conclusions

This pioneering study shows that it is possible to produce a canola meal based adhesive without expensive protein isolation for the manufacture of WFI. It is possible to increase protein content of canola meal by crushing and sieving to >400 μm fractions. The protein-rich canola meal based adhesive constitutes a suitable alternative to pMDI for the production of wood fiber insulation boards up to a density of 140 kg/m^3 in regard to IB and CS. The curing depends on high temperatures, which can be realized by means of hot-air/hot-steam process. By generating temperatures far beyond 100°C, a proper protein denaturation and thus enhanced mechanical properties are obtained. The alkaline adhesive is buffered by acidic wood fibers. Carbohydrates within the canola meal might provide additional bonding properties but also decreased water resistance. By decreasing the densities, the physical-mechanical properties decrease as well. But still, there is a need for further improvements, especially regarding ST-WA.

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Conflict of interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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