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Research paper

Utilizing *Miscanthus* stalks as raw material for particleboards

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ABSTRACT

Miscanthus x giganteus stalks were studied as a possible replacement for wood in particleboards. Produced particles from *Miscanthus* contained 38% of cellulose, and 17% of lignin, while spruce had 45% cellulose, and 28% lignin. The amount of hemicelluloses was the same for both, spruce and *Miscanthus* (21%). *Miscanthus*-made particleboards were produced at two levels of methylene diphenyl diioscyanate resination, i.e. 4% and 6%. Modulus of rupture (MOR), modulus of elasticity (MOE), internal bonding strength (IB), thickness swelling and water absorption were measured. Mechanical properties of the *Miscanthus-made* particleboards were overall reduced: compared to spruce, MOR and MOE were down by 30%, while IB was lowered by 60%. Microscopic analysis of fracture surfaces of the *Miscanthus*-made particleboards after IB testing showed collapsed cells regions in the soft parenchyma, with no obvious adhesive failures. In contrast, spruce-made particleboards revealed much smoother fracture surfaces with structural failures running through cell walls and possibly also through gluelines. The collapsed parenchyma cell regions suggest a direct link to the reduced mechanical properties. Further, compared to spruce the *Miscanthus*-made particleboards have shown higher thickness swelling, but lower water absorption. For *Miscanthus*, no effects of higher MDI adhesive dosages on MOE, MOR and IB were observed. To further improve properties of *Miscanthus*-made particleboards, at sorting-out of parenchyma tissue components to the highest degree possible is recommended, prior to hot-pressing.

1. Introduction

Due to its worldwide abundancy, wood has been for more than 80 years the prime raw material to produce particleboards. In Europe, over 28 million m^3 of particleboard panels are produced per anno (EPF, 2014). Considering the high production volumes, along with evidenced restrictions of natural resources (Giljum et al., 2009), a shortage in wood supply is potentially becoming a critical future matter. Strategies addressing this challenge may be especially considered by countries having a low forest area. Here, an increasing variety of lignocellulosic resources could be of strategic importance, including biomass residues obtained from abundantly growing agricultural plants. While plant seeds are utilized as food and feed, and stem parts, leaves, or root peels are converted to fine chemicals or biogas (Mast et al., 2014), lesser utilized plant parts could be also used in panel production. Utilization of agricultural residues for panel production to be used in furniture, or packaging, would certainly have economic benefits. Utilization of agricultural residues for commodity products also lowers environmental burdens by improving resource efficiency of the agricultural value-chain (Börjesson and Tufvesson, 2011; Geldermann et al., 2016).

Past research addressing particleboard production using plants residues include e.g. rice straw (Gerardi et al., 1998; Li et al., 2010; Yasin et al., 2010), wheat straw (Mo et al., 2003), sunflower stalks (Bektas, 2005; Guler et al., 2006; Khristova et al., 1996; Mati-Baouche et al., 2014; Klímek et al., 2016), reed canary grass (Trischler and Sandberg, 2014), date palms (Amirou et al., 2013), oil palms (Hashim et al., 2011), opium poppy husks (Küçüktüvek et al., 2017), topinambour and cup-plant stalks (Klímek et al., 2016, and cotton stalks (Guler and Ozen, 2004). Balducci et al. (2008) and Dix et al. (2009) introduced residues of several central European agricultural plants as raw materials for low-density particleboards, and Selinger and Wimmer (2015) have shown light-weight sandwich particleboards made with shives and fibers from hemp. While various agricultural residues are recognized as being viable in the production of the particle-based panels, research concerning the utilization of *Miscanthus* x *giganteus* is limited. Balducci et al. (2008) and Dix et al. (2009) have introduced a lightweight *Miscanthus* particleboard, showing moderate mechanical performance due to the lower density. *Miscanthus* was also utilized to produce fiberboard panels by

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Salvadó et al. (2003). *Miscanthus* as a plant genus comprising a perennial, woody, rhizomatous, a bamboo-like grass, is native to tropical and subtropical regions of Asia and Southeast Africa. The plant has a usual height between 1.5 m and 4 m, with stem diameters between 1 and 2 cm. Species such as *M. floridulus* and *M. lutarioparius* may even reach heights up to 6–7 m. Due to the tolerance of varying ecological conditions, *Miscanthus* has been getting also popular in colder European climates (Monti et al., 2015; Parajuli et al., 2015). Today, *Miscanthus* is a widely used energy crop (Ameline et al., 2015), and a resource for fine chemicals (Arnoult et al., 2015; Kim et al., 2015). With a cultivation area in Europe of 38,300 ha (Iqbal and Lewandowski, 2016), the thick-stemmed nodal woody *Miscanthus* (Xue et al., 2015), with a dry mass yield of up to 40 t/ha (Lewandowski et al., 2003; Monti et al., 2015), could be a highly attractive resource in particleboard production. We therefore hypothesize that *Miscanthus* is a resource suitable for particleboards showing acceptable material performance. The following research tasks are pursued: (1) Designing *Miscanthus*-made particleboards suitable for general purposes according to EN 312. (2) Property comparison between *Miscanthus*-made and spruce-made particleboard. (3) Assessing the effect of different adhesive amounts on bending properties, internal bonding as well as thickness swelling, and finally (4) understand property differences between *Miscanthus*-made and spruce-made particleboards at the micro-structural level.

2. Materials and methods

Miscanthus stalks (*Miscanthus x giganteus*) were obtained from a cultivation site in Northern Germany. Stalks were approximately 1.7 m long; cross-sectional diameters were between 15 and 30 mm. As a control, recently felled (fresh) spruce wood (*Picea abies* L. *[Karst.]*) without bark was also used. Raw materials were chipped in a Klöckner 400/120 H2W (Klöckner Maschinenfabrik, Lauenburg, Germany) chipper, using a cutting speed of 725 rpm, and a feeding speed of 1 m/s. The obtained chips at approximate dimensions of $20 \times 10 \times 5\ mm^3$ were subsequently milled to particles in a Condux-Werk HS 350 (Condux Maschinenbau GmbH & Co. KG, Hanau – Wolfgang, Germany) hammer mill. Particles were screened in a cascadic-vertical Allgaier D7336 (Allgaier-Werke GmbH, Uhingen, Germany) screener. The sieve cascade system with mesh size openings of 5.0 mm, 3.15 mm, 1.24 mm and 0.60 mm was used to sort particles to different fractions. Particles sized > 1.24 mm, and < 5 mm, were taken and manually mixed at a weight ratio 50:50. Particles mixtures were oven-dried at 74 °C for 4 days, reaching a final moisture content between 5%–7% d.w. (Fig. 1).

2.1. Preparation of panels

Particleboards with a targeted density of 600 kg/m^3, and a constant thickness of 11 mm, were produced with spruce and with *Miscanthus* particles (Fig. 2), respectively, using methylene diphenyl diioscyanate (MDI) as the adhesive (Huntsman I-BOND® PM4390, Huntsman GmbH, Hamburg, Germany). Two levels of adhesive dosage were used, i.e. at amounts of 4% (MDI4), and 6% (MDI6), respectively. MDI was applied to the particles in a drum blender for 5 min, using a pneumatic spraying nozzle. Prior to pre-pressing the resinated particles were manually distributed in a wooden forming box ($550 \times 550\ mm^2$). Then, the formed mats were hot-pressed at 200 °C and at 3.2 MPa press pressure, for 100 s. The final panel thickness was checked at several randomly selected spots. Final panel thickness was 11 ± 0.1 mm, both species at two resin dosage levels, resulting in four different particleboard types with three replicates each.

Fig. 1. Spruce and *Miscanthus* particles as used for particleboards.

2.2. Material properties and data evaluation

Mechanical testing was carried out on a Zwick® 1474 universal testing machine using testXpert II software (Zwick GmbH & Co. KG, Ulm, Germany). Three point bending tests (EN 310) were employed to determine Modulus of rupture (MOR) as well as Modulus of elasticity (MOE), with the samples ($L \times W \times T = 290 \times 50 \times 12$ mm) submitted to a loading rate of 7 mm/min until failure. Internal bonding (IB) strength was measured according to EN 319. Prior to testing the samples were sanded and glued between stainless steel blocks. The blocks were positioned in gimbal-mounted holders, and pre-loaded in tension at 5 N. Subsequently, a loading rate of 1 mm/min was applied until failure.

Thickness swelling was determined according to EN 317. Conditioned samples sized $12 \times 50 \times 50\ mm^2$ were fully immersed in 20 °C distilled water. Thickness swelling was measured at two time intervals, after 2 h as well as 24 h. As soon immersion time had elapsed, the test samples were taken out from the water and excess water removed with paper tissues. Thickness swelling was measured manually using a thickness gauge, positioned in the center of the samples. Vertical density profiles (VDP) were determined using the x-ray density scanning device GreCon RG44® (GreCon, Germany). Five samples per particleboard type, $12 \times 50 \times 50\ mm^2$ in dimension, were scanned. The obtained data were analyzed using Statistica v.12 (StatSoftinc., Tulsa, United States) software. Normality of the data was checked with the Shapiro-Wilk test. Analysis of variance (ANOVA) with Scheffé post-hoc test was employed, with the level of significance set at 5%.

2.3. Scanning electron microscopy

Surface topography of the particleboards was investigated using the scanning electron microscope TESCANVEGA3 (Tescan Brno, s.r.o., Brno, Czech Republic). Morphology of the *Miscanthus* stalks was studied as well as the particle–particle interactions for both particleboard types, all captured with a secondary electron detector. Specimens obtained from the ruptured regions of the IB samples were gold-coated in a vacuum sputter coater. The SEM accelerating voltage was set at 16.7 kV. The regions of the fractured particleboard surfaces were captured.

2.4. Chemical analysis

For the chemical analysis one sample of 200 mg per material type was prepared. These samples were then pre-hydrolyzed with 2 ml of a 72% H_2SO_4 (30 °C, 1 h). The reaction mixture was diluted with 56 ml ultra-pure water, and post-hydrolysis was performed in an autoclave at 120 °C, and 1.2 MPa pressure for 30 min. For the high-performance liquid chromatography borate analysis, wood sugars were separated in a 5.6 mm column, 115 mm long (Omnifit®, Diba Industries, Inc., Danbury, North America) filled with strong anion exchange resin 114 MCL gel CA08F (Mitsubishi Chemical Corporation, Tokyo, Japan) at 60 °C. The mobile phase (0.7 ml/min) consisted of solution A, 0.3 M potassium borate buffer with pH 9.2, and solution B, 0.9 M potassium borate buffer with pH 9.5. After sample injection chromatographic separation started with 90% (A) and 10% (B), with the run lasting 35 min. Data acquisition was ceased after 50 min. For quantification a post-column derivatization of monosaccharides with Cu-bichinconinate (0.35 ml/min) was applied. The reaction was performed at 105 °C in a 30 m crocheted Teflon coil of 0.3 mm inner diameter. This enabled the