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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 × 10 × 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).

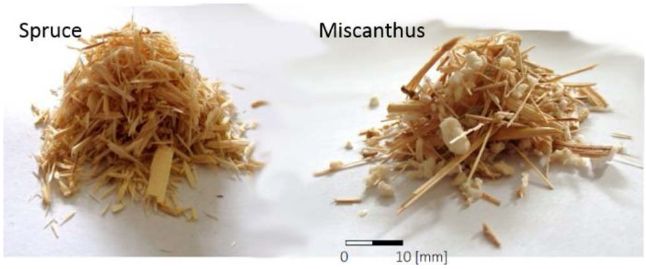

Fig. 1. Spruce and *Miscanthus* particles as used for particleboards.

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 × 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.

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 × W × T = 290 × 50 × 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 × 50 × 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 × 50 × 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

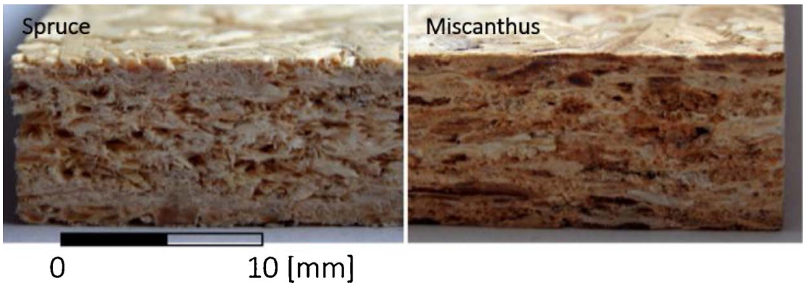

Fig. 2. Cross sectional views of the produced spruce and *Miscanthus* particleboards.

subsequent detection of sugars at 560 nm (Sinner et al., 1975; Sinner and Puls 1978). Data were processed using dionex® chromeleon software (Thermo Fischer Scientific Inc., Sunnyvale, United States). The detected glucose was taken as an equivalent for the cellulose content, since this long-chain polysaccharide is made up of glucose monomer units (Gibson, 2012). The sum of detected mannose, galactose, arabinose, rhamnose and xylose was taken equal to the hemicellulose content, while the insoluble substrate that remained after hydrolysis was considered to be the lignin content (Weiss et al., 2013).

3. Results and discussion

Compared to spruce, *Miscanthus* particles contained 7% less cellulose, and also 9% less lignin (Table 1), which was in coherence with literature values (Kim et al., 2015). Literature also reports that *Miscanthus* may contain up to 4% ash (Kim et al., 2015), and up to 24% extractives (Kim et al., 2012), the latter including mostly fatty acids, sterols, and other aromatic compounds (Brosse et al., 2012). This complies with our data, as the sum of cellulose, hemicellulose and lignin measured for the *Miscanthus* particles was 73% wt, with the remaining 27% wt being the contents of ash and extractives. The high extractive content potentially reduces water absorption and thickness swelling, while IB could be lowered (Nemli et al., 2006). The lower lignin content may lead to higher water absorption (Achyuthan et al., 2010). Other authors (Nasser, 2012; Nemli et al., 2003) suggested that the reduced cellulose amount in *Miscanthus* is hampering the mechanical properties of produced particleboards. Hemicellulose, lignin and cellulose contents need to be considered with respect to seasonal fluctuations taking place in *Miscanthus*. Arnoult et al. (2015) found higher amount of cellulose, hemicellulose and lignin in *Miscanthus* harvested in winter season, compared to those harvested in autumn. The practical meaning is that particleboards produced from winter-harvested *Miscanthus* may show different mechanical properties.

3.1. Microstructural analysis

Miscanthus as a representative of the *Poaceae* family is structurally different from wood. *Miscanthus* stalk sections in transversal (Fig. 3D) as well as longitudinal (Fig. 3A–C) directions are shown. The outer ring of the stalk contains the vascular bundles, which are embedded in parenchyma tissue, and surrounded by the epidermis (ep). The inner region of the stalk cross section (core) is mainly built of soft parenchyma cells, also with inserted vascular bundles (Kaack et al., 2003; Xue et al., 2015).

Fig. 4 is showing ruptured surfaces of a *Miscanthus*-made as well as a spruce-made particleboards, after IB testing. At the low magnification structural differences between *Miscanthus* and spruce particles are barely visible (Fig. 4A and D). At higher resolution, however, the *Miscanthus* fracture surfaces clearly reveal collapsed parenchyma cells (Fig. 4B), while spruce shows more smooth fracture surfaces without visible cell collapse (Fig. 4E). The collapsed parenchyma cells in *Miscanthus* suggests a direct link to weaker mechanical properties. Hashim et al. (2011) who have manufactured particleboards from oil palm biomass, found compressed cell structures, with some cells showing fractured surfaces. However, this type of cell compaction is probably related to some properties of the used biomass as it was not much present in our material and might be also observation site dependent.

3.2. Mechanical properties

MOR of the spruce-made particleboards bonded at the higher adhesive dosage (MDI-6%) was significantly higher (ANOVA $p < 0.05$) than the *Miscanthus*-made ones. With the lower adhesive dosage (MDI-4%), MOR of the spruce-made and *Miscanthus*-made particleboards were not significantly different (ANOVA $p > 0.05$). Likewise, no effect (ANOVA $p > 0.05$) of the increased adhesive dosage on the MOR was found, neither for the *Miscanthus*-made, nor for the spruce-made particleboards (Fig. 5).

MOE shows similar trends as MOR. Average MOE at both resin dosage levels of the spruce-made particleboards was higher compared to *Miscanthus*, however, differences were not significant ($p > 0.05$). MOR and MOE of the *Miscanthus*-made particleboards were similar to data reported by Salvadó et al. (2003). In contrast, our *Miscanthus* MOR and MOE values were two times higher than the low-density *Miscanthus*-made particleboards tested by Balducci et al. (2008), and by Dix et al. (2009). Interestingly, MOE and MOR of our *Miscanthus*-made particleboards were similar to the medium density urea-formaldehyde-bonded *Miscanthus* particleboards presented by Balducci et al. (2008). MOR of our particleboards was similar to boards produced with other plant materials. Bending properties of boards produced from cotton stalks (Guler and Ozen, 2004), sunflower stalks (Khristova et al., 1996) or cotton, kenaf and reed mixed with poplar (Philippou and Karastergiou, 2001) were at similar levels.

Assessing the viability of *Miscanthus* particleboards with respect to

Table 1
Chemical composition of used raw materials for particleboard production, the values in brackets are obtained from literature, n.d. – no data.

	Spruce particles		Spruce wood (Stelte et al., 2011)		*Miscanthus* particles		*Miscanthus* Stalks (Kim et al., 2012; Gunnarsson et al., 2014)	
Cellulose [%]	45.4		43		38		36–38	
Mannose [%]	12	Σ20.9	n.d	Σ23	0.5	Σ21.1	0.1–0.6	Σ18–26
Galactose [%]	2.1		n.d		0.9		0.2–4.4	
Arabinose [%]	1.1		n.d		2.7		0.2–3	
Rhamnose [%]	0.2		n.d		0.2		n.d.	
Xylose [%]	5.5		n.d		16.8		19.6	
lignin [%]	28.2		25		17		13–18	

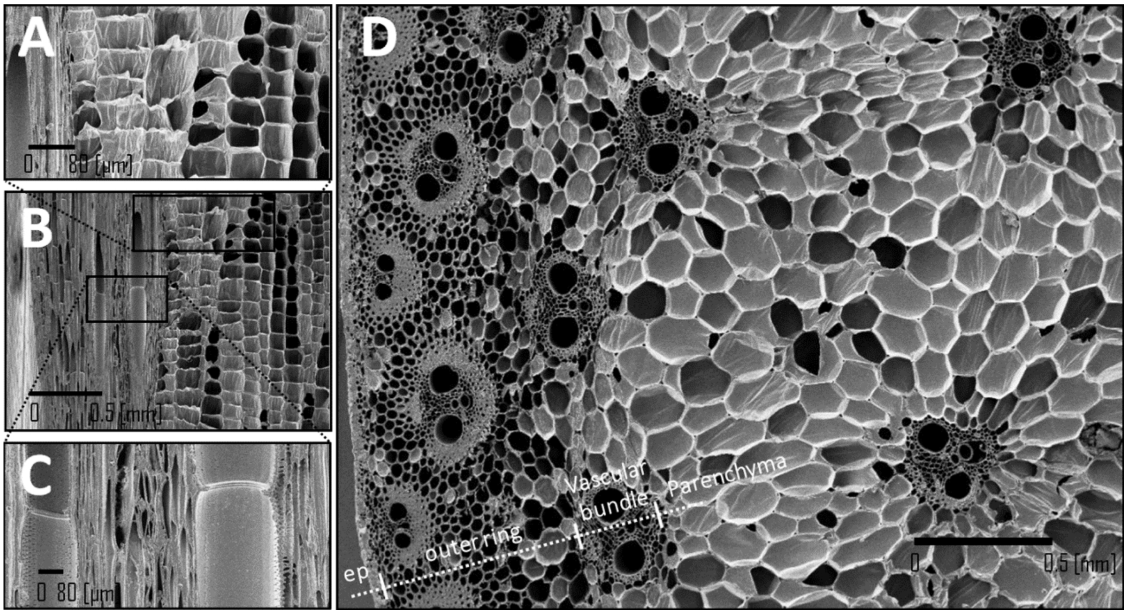

Fig. 3. Microscopic structure of *Miscanthus* stalks. The longitudinal (A–C) and transverse cross-section (D). ep – epidermis.

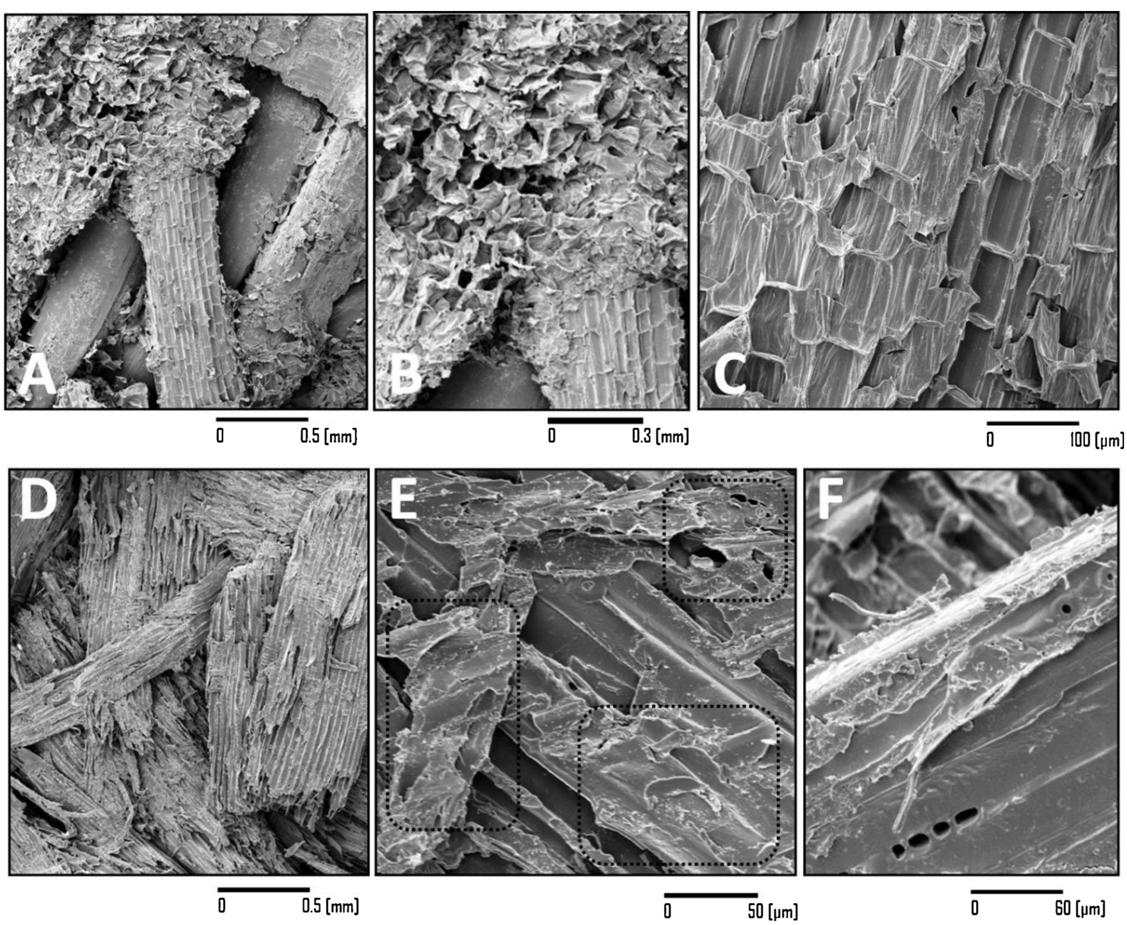

Fig. 4. SEM of the ruptured specimen's surfaces. A–C – *Miscanthus* particleboard, D–F – Spruce particleboard. C – collapsed/ruptured parenchyma cells.

standards, it was found that the average MOR and MOE comply with the EN 312 P1 class requirements. MOR of both panel types have shown suitability for general purposes used in dry conditions. P1 class according to EN312 standard does not mention a minimum MOE. The average MOEs found for both particleboard-types met the higher class P2 requirement.

Fig. 6 shows that *Miscanthus* particleboards had reduced IB values, compared to regular spruce-made particleboards. This is most likely due to the specific anatomical structure of *Miscanthus,* which is fundamentally different from spruce. The presence of rather soft *Miscanthus* pith particles in particleboards might have resulted in a weaker particle bonding situation. Similar IB values for *Miscanthus*-made and

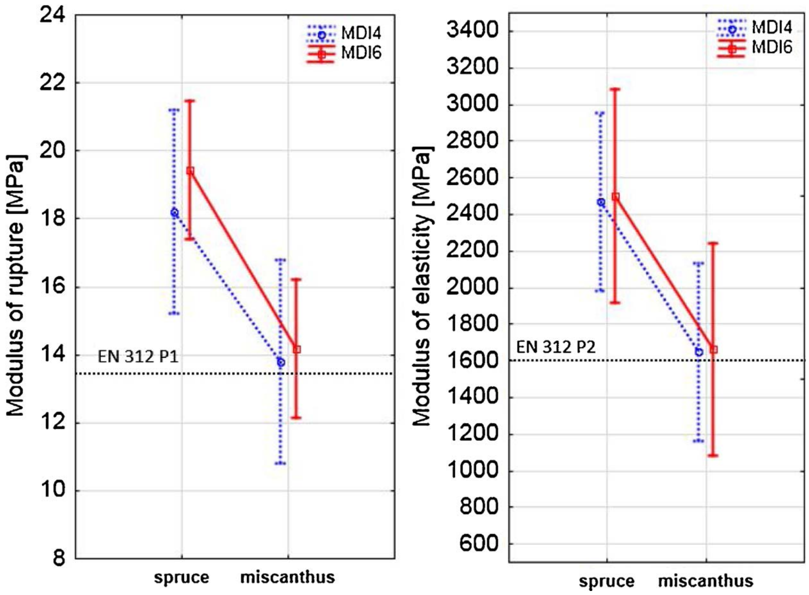

Fig. 5. Modulus of rupture (MOR) and modulus of elasticity (MOE) of the spruce and *Miscanthus* particleboards; MDI4–particleboards are bonded with 4% weight amount of the MDI resin; MDI 6 – particleboards are bonded with 6% amount of the MDI resin.

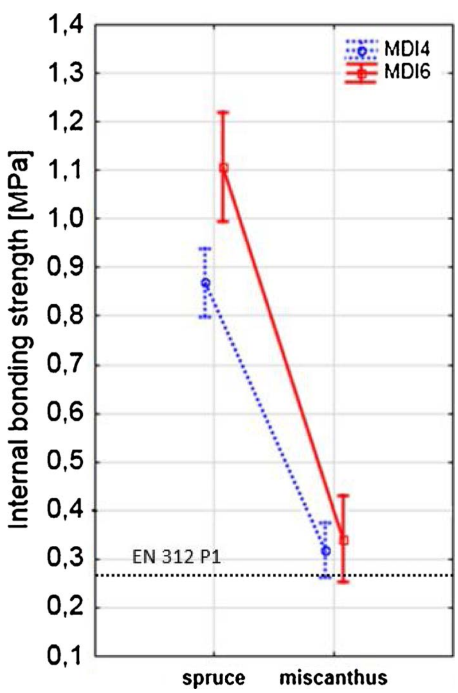

Fig. 6. Internal bonding strength (IB) of the spruce and *Miscanthus* particleboards; MDI4–particleboards bonded with 4% MDI; MDI 6 – particleboards bonded with 6% MDI.

wood-made particleboards are reported by Balducci et al. (2008). Particleboards made with alternative resources often deliver lower IB values, compared to wood-based particleboards. IB values turned out to be also lower for particleboards manufactured with cotton stalks (Guler and Ozen, 2004), vine stalks (Yeniocak et al., 2014), tree leaves (Aghakhani et al., 2013), hazelnut husks (Çöpür et al., 2007), or when made from rice husks (Suleiman et al., 2013). It was surprising that the higher MDI dosage did not improve IB in the *Miscanthus*-made particleboards, while the IB for the spruce-made particleboards was increased significantly ($p < 0.05$) with the higher adhesive dosage. As a summary, IB of *Miscanthus*-made particleboards was significantly reduced over conventional spruce-made particleboard, even the obtained IB values were still above the threshold value of 0.28 MPa, as defined in EN 312 for general purpose particleboards in dry conditions.

Thickness swelling after water immersion of 2 h (TS 2 h) for both MDI-glued *Miscanthus*-made particleboard types was reduced by up to 30%, compared to spruce-made particleboards. While the higher MDI dosage reduced thickness swelling of spruce particleboards, there was no such effect for the *Miscanthus* type. Results for 24h-thickness swelling turned out to be similar. Here, *Miscanthus*-made particleboards performed with a significantly lower 24h-swelling compared to spruce. The higher adhesive amount was more expressed in TS 24 than in the TS 2 h data. The higher MDI-dosed particleboards had overall a significantly reduced TS 24. The spruce-made particleboards bonded with the higher MDI dosage shows about the same 24h-thickness swelling than the *Miscanthus* particleboards bonded with the lower MDI amount. Compared to the spruce type, water absorption also turned out to be higher for both *Miscanthus* particleboard types (Fig. 7).

A remarkable outcome here was the thickness swelling of the *Miscanthus* particleboards, which was below the one found for spruce particleboards, while water uptake being significantly higher. It can be assumed that this is due to the presence of *Miscanthus* pith particles in the boards. The *Miscanthus* pith is almost entirely composed of parenchyma cells, with have soft and spongy structures, responsible in the living plant for storing and transporting nutrients. The dry pith particles in *Miscanthus*-made particleboards are therefore absorbing water without swelling, due to the spongy nature of this tissue. Water absorption and thickness swelling of the produced particleboards were within the range of results shown by Balducci et al. (2008) for low density *Miscanthus* particleboards. In general, adding water-repellents such as paraffin (Papadopoulos, 2006), or phenolic resin as adhesive (Khristova et al., 1996; Pizzi and Mittal, 2003), surface finishing with e.g. veneer overlays (Král et al., 2013; Nemli et al., 2005) would increase water repellency of the panels.

3.3. Density profile

The vertical density profiles (Fig. 8) of the *Miscanthus*-made particleboards did not differ from the profiles measured for the spruce particleboards. The usual U- shape of vertical density profiles (Wong, 1999; Wong et al., 1998), with density peaks near to the surface layers, were observed for both particleboard types. Likewise, the average densities of the panels made with the two raw materials *Miscanthus* and spruce were not significantly different. Average density was 635 kg/m^3 for spruce, and 628 kg/m^3 for the *Miscanthus*-made particleboards. This finding suggests that neither the density profile, nor the mean density were responsible for the differences in the physical and mechanical performance, as found for the spruce-made and the *Miscanthus*-made

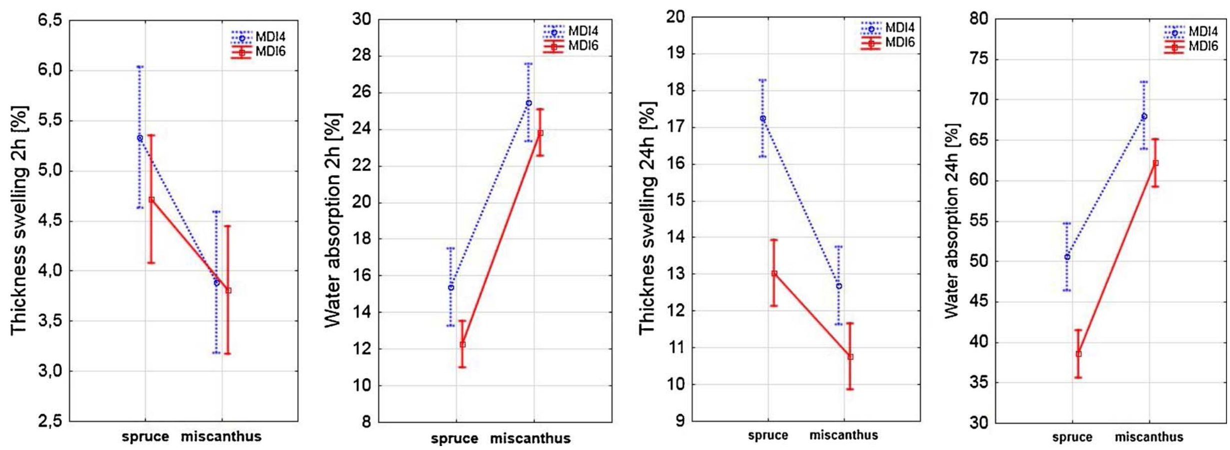

Fig. 7. Thickness swelling and water absorption after 2 h and 24 h, for spruce and *Miscanthus* made particleboards; MDI4–particleboards bonded with 4% MDI resin; MDI 6 – particleboards bonded with 6% MDI resin.

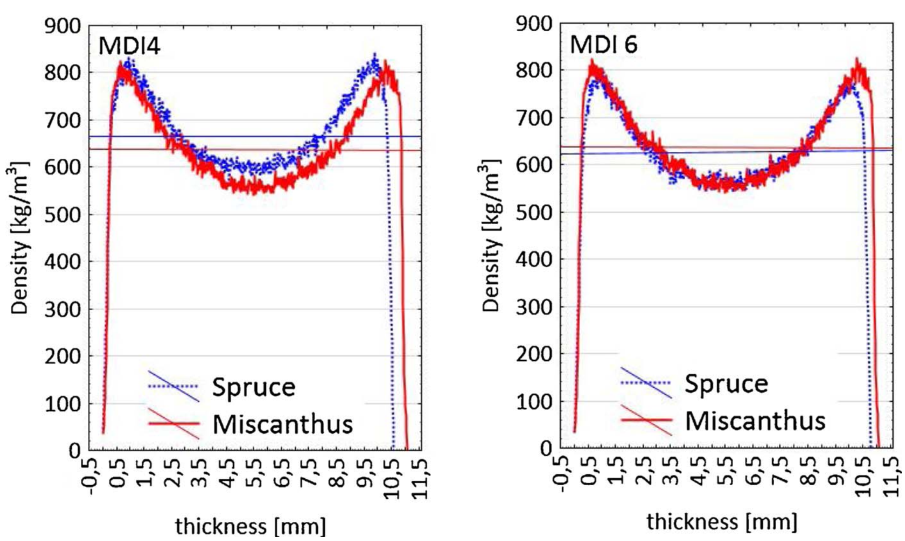

Fig. 8. Vertical density profiles of the spruce and *Miscanthus* particleboards, MDI 4–particleboards bonded with 4% MDI resin; MDI 6 – particleboards bonded with 6% MDI resin.

particleboards. This also suggests that the compaction ratio, which is the ratio of panel density to density of the raw materials, is more or less identical for both material-types. With the compaction ratio being standard, it is anticipated that the panel springback will be also low and similar across the tested material types.

4. Conclusions

In this research, wood for particleboards was substituted by particles obtained from *Miscanthus* stalks. Despite the fact that the mechanical properties observed for *Miscanthus*-made particleboards were lower than those obtained for the spruce-made particleboards, the *Miscanthus*-made particleboards still met the requirements for general use particleboards in dry conditions, as defined in EN 312. The microscopic evaluation has shown that the soft parenchyma tissues are triggering mechanical failures, which are compromising the mechanical properties of *Miscanthus*-made particleboards. This finding is seen as important in the further development of particleboards containing *Miscanthus* as the biomass resource. Based on the observed structural-mechanical failures, it is recommend to sort out parenchyma tissue components to the highest degree possible, prior to hot-pressing. This could significantly improve their physical and mechanical properties. It is concluded that the involved parenchyma tissues are most-likely responsible for higher water uptake rates of the *Miscanthus*-made particleboards, while thickness swelling was in fact lower than with the spruce-made particleboards. Future research may include optimization trials of material mixes, e.g. Miscanthus and wood. A research focus could be also on compaction ratios of particleboards made with different raw materials, addressing possible consequences on panel springback, and understand relationships to various properties.

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