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INFLUENCE OF SURFACE ROUGHNESS OF WOOD FIBRES ON PROPERTIES OF MEDIUM DENSITY FIBREBOARDS

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Abstract:

The properties of medium density fibreboards (MDF) are amongst others influenced by the properties of the wood fibre pulp. One influencing property is supposed to be the surface roughness which is known to be important for the bond quality e.g. when gluing veneer and solid wood.

At the Institute for Wood Technology Dresden (IHD) in cooperation with the ETH Zurich and the BA Saxony examinations on the determination of roughness of wood fibre surfaces as well as the influence of the surface roughness on MDF-properties have been conducted. The aim of the work was to evaluate the surface roughness of fibre pulps made of different wood species and defibrated at different temperatures by the use of Confocal Laser Scanning Microscopy (CLSM). Furthermore the influences of the surface roughness of the wood fibre on the resulting MDF-properties were determined.

The results revealed that the pulping temperature and the wood species have a significant influence on the resulting surface roughness. In comparison to Scots pine [Pinus sylvestris L.] beech [Fagus Sylvatica] wood fibres (defibrated at same temperature) have a smoother surface. The surface roughness of the wood fibre pulps induced by the defibration temperature correlate with the MDF-properties.

Key words: MDF; single fibre; surface roughness; beech; pine; confocal laser scanning microscopy.

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INTRODUCTION

The glueability of wood is mainly described by the adhesion behaviour between adhesive and wood surface which is the sum of chemical and physical inter- and intra-molecular powers as well as mechanical interactions (Zeppenfeld and Grunwald 2005). The extend of those bonding mechanisms are amongst others determined by the roughness of the wood surface as adherent. With an increasing surface roughness the bond surface area and the mechanical bond between wood surface and resin increase as well. On the other hand, a surface roughness above a specific ideal degree has a harmful effect on bonding quality by a decreasing contact area and a declining relation of adhesive and surface area (Aydin und Colakoglu 2005). Especially for gluing of veneer and solid wood the ambivalent influence is extensively described in literature. (Dunky and Niemz 2002, Neese et al. 2004, Avdin and Colakoglu 2005; Zeppenfeld and Grunwald 2005, Bekhta et al. 2012).

The relation between surface roughness and bond quality for bonding of veneer and solid wood is supposed to be at least partially valid for the bonding of wood fibres when producing medium density fibreboards (MDF).

Especially for MDF, other properties, such as deep milling quality, are named to be influenced by the surface roughness of the fibres, as well. MDF panels made of beech wood fibres had a better deep milling performance than MDF made of softwood fibres (Krug et al. 2008). The qualitatively examined surface roughness of beech wood fibres indicated smoother surfaces than those of softwood fibres and is stated to be one important influence regarding the resulting deep milling guality (Krug et al. 2008, Scheiding and Krug 2002).

Besides wood species, investigations regarding the influence of the pulping temperature on the resulting fibre surface roughness have been conducted by the use of atomic force microscopy (AFM). With increasing pulping temperature the maximum values of roughness of the sub-micro structure (measuring distance of 6µm) of the fibre surface increased from around 50 to 250nm. The roughness was stated to be the sum of cellulosic fibrillar network, microscopic disruptions (mainly caused by low-temp refining) and reposition of lignin and hemicellulose (mainly caused by high-temp refining). The increasing roughness is discussed to be the result of an increasing plasticization and granular rearrangement of lignin on the fibre surface with increasing temperature (Groom et al. 2006).

In addition to the sub-micro structure the change of the micro structure, including e.g. defects and pits, in longitudinal and perpendicular direction to the fibre axis as well as the distribution of roughness over particle size (e.g. single fibre, fibre bundle, shives) are of great interest for the evaluation of the over all fibre surface roughness.

The investigated fibre properties are mainly not comparable, especially regarding the qualitative study. Further investigations with AFM are limited regarding the amount of work needed for sample preparation as well as the duration of measurements with longer measuring distances of e.g. $20 - 500 \mu m$.

In this work a method for a suitable and quick fibre sample preparation is displayed and the possible evaluation of surface roughness on those samples with the use of a confocal laser scanning microscope (CLSM) is presented using the example of Scots pine [Pinus sylvestris L.] and beech [Fagus Sylvatica] wood fibres pulped at different temperatures. Furthermore the influence of the determined surface roughness of the different fibre pulps on the thereof produced MDF are displayed and discussed.

MATERIAL AND METHODS

Wood fibres

Six variants of fibre pulp were produced by varying the pulping temperature and the wood species. Logs of Scots pine [Pinus sylvestris L.] and beech [Fagus Sylvatica] from the "Dresdner Heide" were cut, debarked, chipped and sieved. The wood chips of the two wood species were then plasticised in a digester at a temperature of 165, 180 or 192°C, defibrated in a 12inch laboratory refiner (Andritz) and dried in a lab flash drier. The grinding plate gap distance was 150µm and the plate driving speed was 3000rpm. Refiner output of absolutely dry fibre was 60kg/h with a sample size for each variant of 12 - 15kg and the inlet temperature of the flash drier was around 110°C.

Wood fibre separation and fixation

For separating the wood fibres a modified air-jet sieve (referring to Fig. 1) was used with a mesh of 315µm. The outlet of the air jet sieve was connected with a flow-box with high-voltage (40kV) electrodes at the top of the flow-box, for deposition of the single fibres towards the grounded plate at the bottom of the flow-box. Microscope slides were placed on top of the grounded plate with double-faced tape for the fixation of the single fibres towards the slide. Vacuum was applied at the outlet of the flow-box by an exhauster.

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Schematic illustration of fibre separation by air-jet sieving, fibre deposition by electrostatic and fixation on microscope slide by doubled-faced tape.

Air flow starts at the inlet of the rotating air-nozzle at the lower end of the air-jet sieve. With the air flow in the air-jet sieve, the fibre sample distributes well above the sieve and single fibres pass the sieve with the air flow towards the flow box. The single fibres are charged with a negative potential by the high-voltage electrodes and tend towards the grounded plate. Fibres hitting the microscope slide are bonded to the double-faced tape and are fixed onto the microscope slide. One microscope slide carries a few hundred well distributed, flat and horizontal aligned single fibres that are fixed and ready for scanning. Therewith the preparation of hundreds of samples of one variant takes less then 15 minutes.

Confocal Laser Scanning Microscopy

Of each variant 45 fibres were scanned (referring to Fig. 2) with a confocal laser scanning microscope (CLSM – Keyence VK-8710) with a magnification of 100.



3D Image of the topography of a single pine wood fibre defibrated at 180°C.

The resolution of the scans is 140nm for the x and y-directions and 50nm for the z-direction. With the included software surface profiles with 20µm distance were measured at each fibre (referring to Fig. 3). Each profile was height corrected and the arithmetical mean deviation of the roughness profile (Ra) was calculated according to ISO 4887:2010.



Example of surface profile measurement of visibly smoothest part of single pine wood fibre defibrated at 180°C.

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Measurements on each single fibre were done on both the visibly smoothest part and an expectable rough part, which mostly was a pit. The 6 variants with 2 measurements (smoothest and roughest surface area of fibre) and number of measurements n = 45 for each series made an over all number of measurements of 540. The 12 series (n = 45 for each series) were tested for

- log-normal distribution,
- Analysis of Variance.

Manufacture and testing of medium density fibreboard

The manufacture of the lab-scale medium density fibreboards (MDF) were conducted at the laboratory of the Department Materials of the Institute for Wood Technology Dresden (IHD). The single-layered panels had a format of 440mmx460mm, thickness of 8mm (7mm after sanding) and a raw-density of 750kg/m³. The fibres were resinated with an UF-resin (LL 4550 from Momentiv) and a hydrophobic agent (Hydrowax 138 from Sasol). The applied content was 10% for the UF-resin and 1% for the hydrophobic agent based on oven-dry mass of wood fibre. The resinated fibres were formed to mats and then placed in the press between cold pressing plates. The pressing temperature was 220°C and press-time-factor was 15s/mm.

The fibreboards were sanded and cut into specimen according to the standards and stored in standard climatic conditions (20°C and 65% relative humidity) until they achieved equilibrium moisture content. The following properties (referring to table 1) were tested.

Table 1

Conducted tests for determination of properties of lab-scale medium density fibreboards.

Test	Unit	Standard	Number of specimen n
Modulus of rupture MOR	N/mm²	DIN EN 310:	6
Modulus of elasticity MOE	N/mm²	DIN EN 310:	6
Internal bond IB	N/mm²	DIN EN 319:1993/ DIN 323:1993	10
Thickness swelling after 24 h water storage	%	DIN 317:1993	10

RESULTS AND DISCUSSION

Surface roughness

The surface roughness values were tested positive for log-normal distribution by testing for Kolmogorov-Smirnov and applying Quantil-Quantil-Plots. Further Analysis (e.g. ANOVA) were done for the logarithmised values.





Mean Values of the arithmetical mean roughness Ra according to surface area, wood species and pulping temperature.

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The mean values of the Ra log-normal distributions of the 12 series are shown in Fig. 4. The Ra mean values of the rough surface areas with pits are more than twice as high as the corresponding values for the smooth surface area. The Ra mean values of the beech variants are all lower than those for the corresponding pine values. The analysis of variance confirms a significant lower surface roughness for beech fibers in comparison to fibers made of Scots pine. This approves the stated differences by Scheiding and Krug (2002) and Krug *et al.* (2008). Pine wood lignin consists to 86% of Guajacyl lignin (G-lignin) which has less methoxyl groups at the aromatic ring and therewith is cross-linked to a higher degree than Syringyl lignin (S-lignin). Beech wood lignin consists only to 56% of G-lignin and to 40% of S-lignin. This explains on the one hand the lower softening point for beech wood, as discussed already. On the other hand the lower degree of cross-linkages may explain the lower surface roughness of beech wood fibres compared to pine wood fibres. The separation of the middle-lamellae during the pulping process for beech wood should be less of a rupture, since the lignin has less cross-links and therewith could contribute to a smoother surface roughness.

For correlations with MDF-properties the mean value of smooth and rough surface area was calculated for each variant as referring to Fig. 5. The surface roughness for fibres of Scots pine is at a minimum at a pulping temperature of 180°C. With lower pulping temperatures the surface roughness increases significantly. With pulping temperatures above 180°C the surface roughness increases as well with a lower extend. The surface roughness of wood fibres made of beech increases with increasing pulping temperature.

This difference is probably reasoned by the softening temperature of the softwood components, especially for lignin which is around $170 - 175^{\circ}$ C, regarding to the conditions in the digester, whereas hardwood lignin has a softening temperature below 165° C (Asplund 1940). Pulping below the softening point might lead to an increased number of cracks and therewith to a greater roughness.



Fig. 5 Mean Values of the arithmetical mean roughness Ra according to wood species and pulping temperature.

These results are not according to the results of Groom *et al.* (2006) and the discrepancy might be explained by the different magnifications and measuring distances. The high resolution of an AFM is capable of measuring the effect of increasing granular lignin within the sub-micro level by increasing pulping temperature, but therefore micro cracks resulting with lower pulping temperatures and rough surface areas including pits are assumed to be out of the measuring distance of a typical AFM measure.

Regarding the results, the surface roughness of wood fibre pulp might be differentiated into the surface roughness below softening temperature due to mechanically induced micro cracks and the surface roughness above softening temperature due to hydrothermal changes of the surface structure. Both are determinable with the use of CLSM, though the hydrothermal roughness is not as significant determinable as with the use of an AFM due to a lower resolution. The lowest surface roughness for wood fibre pulp is assumed to be produced with pulping temperatures right above the softening temperature of the pulped wood species.

Influence of fibre surface roughness on MDF properties

The correlations of the in Fig. 5 displayed arithmetical mean deviation of roughness profile Ra of the different wood fibre pulps and the properties of the thereof produced MDF are shown in Fig. 6.

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Comparable correlations for both wood species are given for MOR and IB. In both cases the value of the MDF-property decreases with increasing Ra. In this matter the nature of roughness is not critical. Cyr *et al.* (2008) described an increasing loss of resin due to cracks, lumen and pits and a corresponding decrease of strength properties of the MDF. They examined amounts of resin penetrating into the lumen through cracks and pits as well as through openings with a size of a few nanometres. It can be assumed that the resin loss increases with an increasing roughness of the fibre surface and lead to decreasing strength properties.

Since the beech wood variants were all defibrated above the softening temperature, no mechanically induced roughness for beech wood fibres were determined. This leads to contrary correlations between the MDF-properties (MOE and Thickness swelling) and surface roughness in accordance to the wood species. The origin of the surface roughness is critical for these MDF-properties. With increasing pulping temperature the wood fibre becomes more hydrophobic due to splitting off of hemicellulose and stiffer due to condensation reactions of the lignin, so that the hydrothermal induced roughness accompanied with a change in chemical constitution leads to increasing MOE values and decreasing thickness swelling. The mechanically induced surface roughness with a resin loss of greater extend has more impact on the properties than the change in chemical constitution and leads to decreasing MOE values and increasing thickness swelling.



Correlation between MDF - properties and arithmetical mean deviation of the roughness profile Ra according to wood species. (left above) Modulus of rupture MOR, (right above) Modulus of elasticity MOE (left below) Internal bond IB, (right below) Thickness swelling.

CONCLUSION

With the introduced apparatus for fibre separation and fixation it is possible to prepare a few hundreds fibre specimen of one variant in less then 15min. With the chosen parameters for scanning, measuring and evaluation of Ra the measurement of one specimen takes about 4 minutes, so that the needed over all time

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for measurements is quite low compared to existing methods. The investigation of surface roughness with the use of a confocal laser scanning microscope was successful for the determination of differences in the micro level regarding the influence of pulping temperature, wood species and the considered surface areas. The surface roughness of wood fibre pulp can be differentiated into the surface roughness below softening temperature due to mainly mechanically induced micro cracks and the surface roughness above softening temperature due to hydrothermal changes of the surface structure. There is a significant difference in the fibre surface roughness between the two investigated wood species with smoother surfaces for beech compared to pine wood.

All measurements have been done on single wood fibres, though the amount of single wood fibres in the typical conglomerate of fibre pulp for MDF production is usually only around 10 – 30 mass - %. Of great interest for further investigations, is the determination of the distribution of the mean surface roughness regarding the inhomogeneous mixture of fines, single fibres, fibre bundles and shives. With this information it would be possible to evaluate a surface roughness distribution for the complete fibre pulp surface and could be more useful for the understanding of the gluing behaviour of fibre pulps.

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