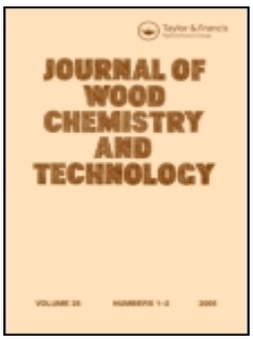

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Effects of Defibration Conditions on Mechanical and Physical Properties of Wood Fiber/High-Density Polyethylene Composites

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Abstract: *Defibration conditions influence wood fiber characteristics and thereby properties of fiber-based materials. In this study, the effects of several defibration conditions on mechanical and physical properties of fiber-based wood-plastic composites (WPCs) are illustrated. Various WPCs were tested containing different thermo-mechanical pulps (TMPs) or groundwood pulp (GWP), whereby material composition (50 wt% wood fibers, 47 wt% polymer, 3 wt% coupling agent) and the production process (internal mixer, injection molder) were kept consistent. The data from the experiment revealed that differing defibration conditions led to statistically significant differences in the tested flexural, tensile, and impact properties as well as in the water absorption of WPC. Overall, the GWP and the TMP which was produced under the mildest defibration conditions performed best in fiber-based WPCs. Therefore, grinders and refiners may be equally suitable to produce pulp for WPC usage. As a side-effect within this study, the reinforcing effect of fiber application on flexural and tensile properties was on an extraordinarily high level.*

Keywords Wood fiber, thermo-mechanical pulp (TMP), groundwood pulp (GWP), wood-plastic composite (WPC), fiber length distribution

Introduction

Wood-plastic composites (WPCs) are usually produced out of wood (e.g., wood flour), plastic, and a coupling agent using extruders for compounding and forming. Recently, there has been a growing interest in applying wood fibers as wooden components in order to reach reinforcing effects. The main challenges for the production of fiber-based WPCs, as the discovery of a suitable production process and material composition, have been under examination in many studies.

Using an internal mixer turned out to be an adequate option to compound fiber-based WPC without feeding problems due to non-free flowing fibers.[1–6] Another approach to

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enable fiber dosing and compounding was to pelletize fibers before processing in a common extruder.[7]

Regarding material composition, an increasing wood fiber content, up to 40–50 wt%, led to better mechanical properties, if it is processed in an internal mixer.[1, 2, 4, 5] The same applied to increasing coupling agent content up to 3 wt%.[1, 4, 5, 8]

It is clear that, for other wood fiber-based materials (e.g., fiberboard and paper), fiber characteristics or defibration conditions influence product properties. Depending on whether the main defibration is carried out by mechanical forces or by chemical lignin removal, pulps are roughly distinguished between mechanical and chemical pulp. Chemical pulp is in general less suitable due to higher prices, whereas subgroups of mechanical pulps were used in many studies for WPC production: groundwood pulp (GWP),[1] refiner mechanical pulp (RMP),[2, 8] thermo-mechanical pulp (TMP),[3–5, 7, 9, 10] and chemi-thermo-mechanical pulp (CTMP).[6, 7, 11, 12] Until now it has not been clear which mechanical pulp or defibration condition leads to the most suitable performance in fiber-based WPC. Evaluating defibration condition effects on WPC properties, by comparing results of several studies, is inadmissible because of differing production processes and material compositions. However, only a few studies have compared fiber-based WPCs differing in defibration conditions and therefore allow a partial evaluation of the fiber type performance.

Migneault et al.[6, 12] tested WPCs containing CTMPs differing in size and discovered that a great fiber length-to-diameter ratio had a beneficial effect on mechanical and physical properties; information on fiber size causing defibration conditions was not provided. Nygård et al.[7] analyzed TMP- and CTMP-based WPCs and determined that CTMP led to slightly higher tensile and impact strengths than TMP, probable due to fewer lignin-covered surfaces of the CTMP; wood fibers were pelletized before compounding. Zhang et al.[4] and Zhang et al.[5] studied WPCs containing TMPs differing in refining conditions (steam pressure: 0.75–1.5 MPa; boiling time: 3–6 min) and found out that defibration conditions influenced tensile properties; the direction of influence was not exerted.

In summary, very little is known about the effect of defibration conditions on fiber-based WPC properties. In particular, there has not been any direct suitability comparison of GWP and TMP as wooden components in WPC. However, further information on the performance of wood fibers in WPC production is of special interest for various pulp- or fiber-producing industries (e.g., paper and fiberboard industry), as they are combined with the chance to place free pulp capacities in a promising new market.

The aim of this paper was to verify the effects of different defibration conditions on mechanical and physical properties of fiber-based WPC. In order to reach this goal, four types of WPC were tested, containing one out of three different TMPs or GWP. The fiber length distribution within each pulp was determined applying a newly developed measuring device.

Materials

Wood Fibers

Three types of TMP and one GWP were used for specimen preparation. The TMPs were produced at the Institut für Holztechnologie Dresden gGmbH (IHD) (Dresden, Germany) using a laboratory refiner, without adding any additives or resin. Wood specie (Scots pine - *Pinus sylvestris*) and grinding gap distance (0.15 mm) were kept consistent for all TMPs. In order to obtain different types of TMP, boiling time (t_B), boiling temperature (T_B), and boiling pressure (p_B) were varied within TMP A (t_B: 8 min, T_B: 200°C, p_B: 1.6 MPa), TMP B (t_B: 4 min, T_B: 170°C, p_B: 0.8 MPa) and TMP C (t_B: 1 min, T_B: 143°C, p_B: 0.4 MPa).

The GWP was kindly supplied by the Papierfabrik Utzenstorf AG (Utzenstorf, Switzerland). A mixture of logs, dominated by spruce (*Picea abies*) and fir (*Abies alba*), was processed in an industrial grinding plant, which was designed for paper specifications.

Before specimen preparation, all fibers were oven-dried at 105°C until a constant weight was obtained.

Polymers

High-density polyethylene (HDPE), called Hostalen GC 7260 (LyondellBasell Industries), was used for composite preparation. It had a density of 0.960 g/cm^3, a melt flow rate of 8 g/10 min (190°C; 2.16 kg), and was provided by UltraPolymers Deutschland GmbH (Augsburg, Germany).

Additives

Maleic anhydride modified HDPE (MA-HDPE), known as Bondyram 5108 (Polyram Ram-On Industries L.P., Ram- On, Israel), was used as the coupling agent for composite preparation. It had a density of 0.950 g/cm^3, a melt flow rate of 8 g/10 min (190°C; 2.16 kg), and was kindly supplied by Velox GmbH (Hamburg, Germany).

Methods

Fiber Characterization

The fiber characterization was done by applying a fiber analyzing system, which is currently under development at the institutions of the authors with significant contributions from the Cognitive Systems Laboratory (KOGS) (Hamburg University, Hamburg, Germany) and Fagus-GreCon Greten GmbH & Co. KG (GreCon) (Alfeld, Germany). This measuring system can be classified as a dry image analysis system: fiber separation is arranged in an air-borne state and image is captured as soon as the fibers are placed onto a glass slap. Subsequent to image recording, the length of each captured fiber was determined by a fiber tracing software.[13–16] Further technical details of this fully automatic measuring system were published by Benthien et al.[15–16]

Each fiber characterization run was carried out on approximately 0.5 g fibers while three repetitions were made. Each detected and length measured fiber was assigned into one of 655 length classes by a special analysis tool. As a final output of this tool, the number of fibers and the mean value of the squared fiber length (l^2) were provided for each length class. Based on this data, the double length-weighted relative frequency was calculated and plotted as a line chart for fiber characterization. The double length-weighted fiber length (L_w) was calculated in accordance with Robertson et al.[17] ($L_w = \sum n \cdot l^3 / \sum n \cdot l^2$) for each fiber type (TMP A, TMP B, TMP C, GWP). Moreover, fiber length and color was haptically and visually evaluated as usually practiced in the industry, due to the lack of adequate measuring systems for fiber quality control,[15] where TMP is produced for wood-based panel manufacturing (e.g., medium-density fiberboard).

Compounding and Injection Molding

For all produced WPC specimens, the composition of raw materials was kept consistent: 50 wt% wood fibers, 47 wt% polymer (HDPE), and 3 wt% additive (MA-HDPE). To obtain different WPCs, the type of wood fibers was changed. The prepared WPCs were noted according to the designation of wood fibers contained: WPC A (TMP A), WPC B (TMP B), WPC C (TMP C), and WPC GWP (GWP). In addition to the four types of WPC, pure HDPE specimens were produced as a reference.

Wood fibers, polymer, and additive were compounded at 160°C and 50 rotations per minute in a laboratory internal mixer named Haake Rheomix OS (Thermo Electron Corporation (Thermo Fischer Scientific), Karlsruhe, Germany). Wood fibers were not added before the registered torque indicated that the melt of polymer and additive had reached a constant state. The compounds were removed from the internal mixer 15 min after adding the wood fibers, cooled to ambient temperature, and manually broken into pieces (diameter about 1 cm), which were suitable for the injection molding machine.

All specimens were injection-molded using a Haake MiniJet II (Thermo Electron Corporation (Thermo Fischer Scientific), Karlsruhe, Germany). The molding conditions, temperature of the pre-heating cylinder (T_{PC}), temperature of the mold (T_M), first pressure (p_1, lasting 15 seconds), and second pressure (p_2, lasting 15 seconds), were varied within WPC compounds (T_{PC}: 200°C, T_M: 100°C, p_1: 60 MPa, p_2: 30 MPa) and pure HDPE (T_{PC}: 160°C, T_M: 90°C, p_1: 50 MPa, p_2: 30 MPa).

Existing burrs on specimen edges were carefully removed using fine sand paper (grid P180). Specimen's surface layer was not further processed.

Determination of Specimen Properties

Specimens were conditioned (seven days) and tested at 20°C and 65% relative humidity. Dumbbell-shaped specimens ($75 \times 10 \times 2$ mm^3) were used for tensile tests in accordance with DIN EN ISO 527–2 (2012). Rod-shaped specimens ($80 \times 10 \times 4$ mm^3) were used for all other tests.

Mechanical Properties. Flexural strength and flexural modulus of elasticity were determined by testing 10 specimens per formulation according to DIN EN ISO 178 (2011) using 64 mm support span and 2 mm/min test speed.

Tensile strength and tensile modulus of elasticity were determined by testing 10 specimens (type: 1BA) per formulation according to DIN EN ISO 527–2 (2012) using 25 mm measuring length and 1 mm/min test speed.

Charpy impact strength was determined by testing 10 unnotched specimens per formulation according to DIN EN ISO 179-1 (2010) using edgewise impact direction and 4 J - pendulum.

Physical Properties. Density was determined by testing 30 specimens per WPC formulation and 20 specimens of pure HDPE according to DIN EN 323 (1993).

Water absorption and thickness swelling were determined after 5 h of immersion in boiling water by testing five specimens per WPC formulation according to E DIN EN 15534-1 (2012). In addition, water absorption and thickness swelling were determined by testing five specimens per WPC formulation under the same conditions but after 1 h of immersion in boiling water.

Statistical Analysis

In order to evaluate the significance of differences among mean values of HDPE, WPC A, WPC B, WPC C, and WPC GWP a single factor analysis of variance (ANOVA) and a Tukey HSD test were conducted using the analysis tool of SAS JMP. The null hypothesis (no effect) was accepted if the p-value exceeded the $\alpha = 0.05$ significance level.

Results and Discussion

Fiber Characterization

The results of the haptical and visual fiber quality evaluation corresponded with expectations due to the defibration conditions: fibers, obtained from a boiling process high in temperature and long in duration, were categorized as "short and dark" (TMP A), while fibers, obtained from a short duration and low temperature boiling process, were categorized as "long and bright" (TMP C). Fiber length and color of TMP B were categorized as "medium," so in between TMP A and TMP C. The GWP were categorized to be finer than TMP A and brighter than TMP C. The result of the haptical and visual fiber length evaluation can be traced looking at Figure 1.

The results of the fiber characterization are given in Figure 2. The fiber size distribution is quite similar for all TMPs. Nevertheless, differences enabled a differentiation between the three TMP types. Focusing first on the plotted double length-weighted relative frequency of the TMPs, the highest frequency of fibers with a length between 0.5 and 3 mm was found for TMP A. The lowest frequency of short fibers was found for TMP C. The order of TMPs was reversed looking at fibers with a length of more than 3 mm. In this case, the highest frequency of fibers was found for TMP C, followed by TMP B and TMP A, whereas, in the case of longer fibers, the frequency of fibers with a length greater than 3 mm increased with decreasing boiling temperature and boiling time. The double length-weighted fiber length of the TMPs was found to be 2.6 mm (TMP A), 2.9 mm (TMP B), and 3.3 mm (TMP C). Consequently, this indicated that the fiber length decreased with increasing boiling temperature and boiling time. The fiber size distribution of the GWP was found to be different in comparison to those of the investigated TMPs. The highest frequency was found for fibers with a length of about 1 mm. The frequency of longer fibers was much lower than for any of the TMPs. The double length-weighted fiber length of the GWP was 1.7 mm. The number of detected fibers within three repetitions was 710,000 (TMP A), 720,000 (TMP B), 610,000 (TMP C), and 1.1 million (GWP).

Fiber differences in the present study resulted very likely from varied defibration conditions, namely boiling temperature and boiling time within the TMPs and defibration machinery between the TMPs and GWP.

Visual Specimen Characterization

Visual specimen evaluation indicated that the chosen production process and the material composition used were suitable to manufacture accurate fiber-based WPCs, while two conspicuous features were noticed: (1) the specimen color depends on the used wood fiber type; the darker the fibers, the darker the specimens; (2) a slight fiber gradient seemed to occur between the specimen ends; in particular for the dumbbell-shaped specimens, the quantity of visible fibers or fiber bundles increased from the end, which was closer to the molding opening to the end, which was closer to the molding bottom (Figure 3).

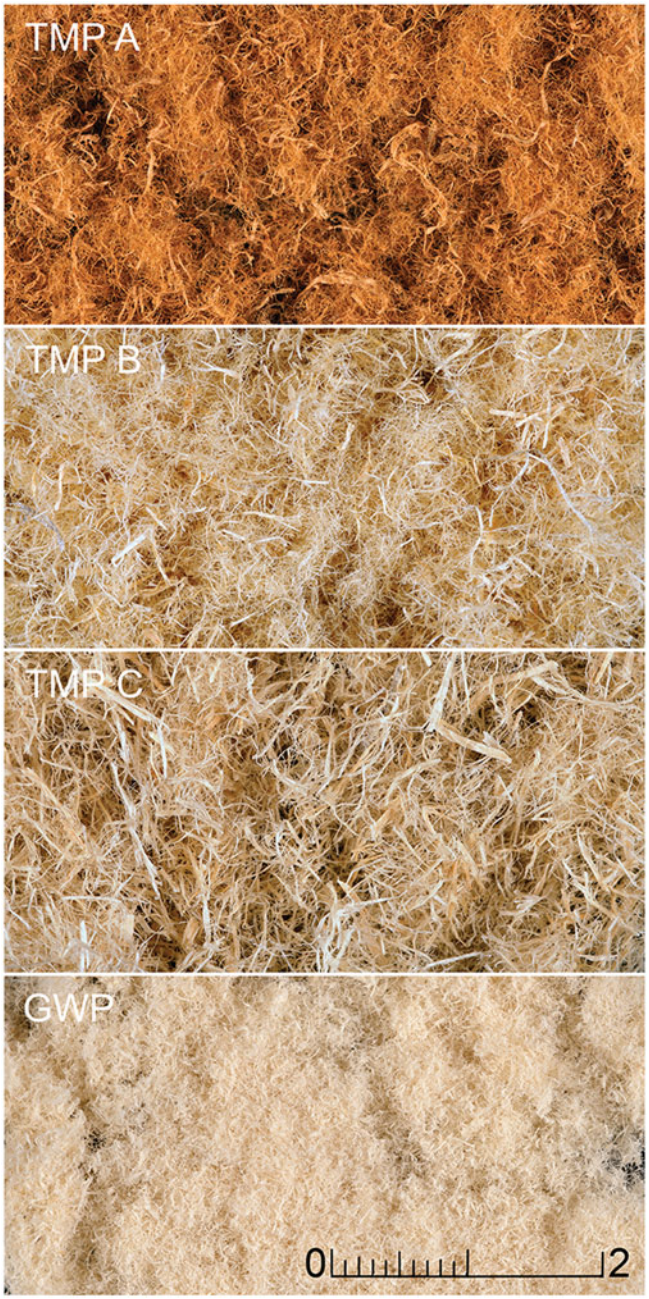

Figure 1. Fiber material (color figure available online).

Determination of Specimen Properties

Mechanical Properties. An overview of the achieved mechanical properties of all specimens is given in Table 1. Statistically significant differences occurred among the WPC results in the tested flexural, tensile, and impact properties. Since the WPCs only differed

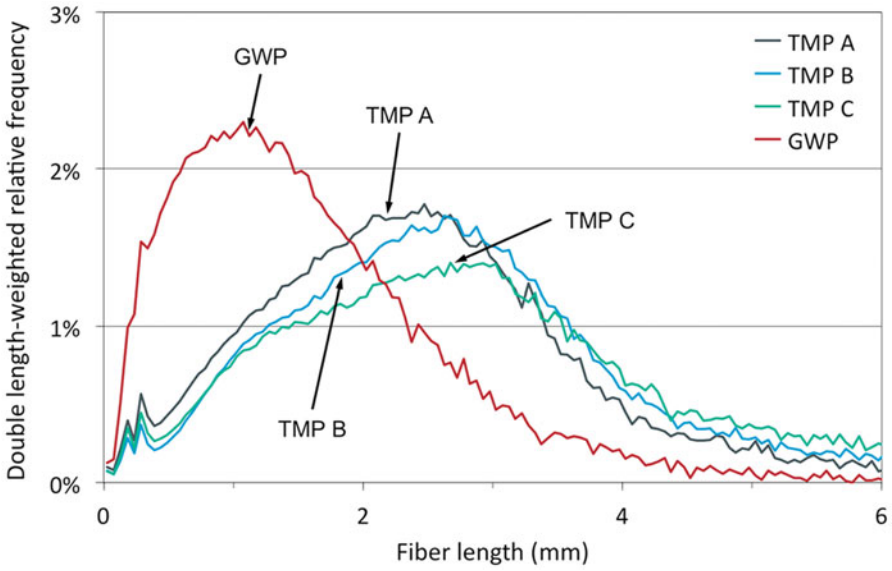

Figure 2. Double length-weighted fiber size distribution (color figure available online).

in defibration conditions of wood fibers, the statistically significant differences were in all probability an outcome of the defibration conditions. The general statement that the wood fiber type or defibration condition can influence mechanical properties of fiber-based WPC is also in accordance with former studies.[4–7, 12]

Within the three WPCs containing types of TMP, WPC C had in the terms of mechanical properties always highest mean values. Since TMP C was characterized as fiber material with highest fiber length, these results are in agreement with former studies,[6, 12] which found a beneficial effect of a great fiber length on mechanical properties of fiber-based WPC.

Regarding all tested WPCs, WPC C and WPC GWP achieved in all mechanical properties the highest and the second highest value in this study (Figure 4 a–d). As a consequence it was assumed that, at least in the area of the tested mechanical properties, TMP C and GWP performed better in WPC than the other fiber types. Since GWP was characterized as fiber material with the lowest fiber length overall, these results could not be explained only by the beneficial effect of great fiber length.

However, fiber material is in addition to fiber size characterized by further properties, which goes along with the defibration conditions. Best mechanical results were achieved in this study using TMP C and GWP, which were produced under mildest boiling conditions or not boiled, respectively. Two possible explanations for the results based on the effect of temperature during defibration are given in the following passages.

As is known for thermally modified timber (TMT), increasing treatment temperatures lead to darker wood color and greater thermal wood degradation in combination with decreasing mechanical properties, among others. In this study, differences in specimen color indicated that wood fiber color and thermal wood degradation, as results of thermal treatment during defibration, were not totally superposed by thermal treatment during compounding and injection molding. Therefore, it can be assumed that better mechanical properties in this study were also caused by less thermal wood degradation during defibration among the fiber types.

Figure 3. Injection molded specimens: WPC A, WPC B, WPC C, HDPE, WPC GWP (from left to right). The boxes show specimen ends from the molding bottom, which are characterized by a slightly higher quantity of visible fibers (fiber bundles) (color figure available online).

As is known for medium-density fiberboard (MDF) production, higher defibration temperatures soften lignin, which has the largest share in the middle lamella between several cells, and thereby allow a defibration with less cell damages and lower mechanical defibration forces. In contrast to higher defibration temperatures, lower temperatures lead to less lignin softening, higher mechanical defibration forces, and more cell damages in the form of ruptures besides the middle lamella. Such ruptures are combined with a higher share of cellulose and hemicelluloses and a lower share of lignin on fiber surfaces. Therefore, it can be assumed that the fiber surfaces of the best performers in this study, TMP C and GWP, had a higher share of cellulose and hemicelluloses and a lower share of lignin. Differences in the surface composition of the fibers could influence the adhesion of HDPE or MA-HDPE. This would be in agreement with the assumption of Nygård et al.[7] that less lignin-covered fiber surfaces lead to better mechanical properties of fiber-based WPC. Nygård et al.[7] explained their assumption in the form that even though lignin is expected to be more compatible with polymer matrix, the presence of such layers will also probably, to

Table 1
Mechanical properties of specimens

	Flexural test						Tensile test			Charpy impact test		
	Strength (MPa)			Modulus of elasticity (GPa)			Modulus of elasticity (GPa)			Strength (kJ/m^2)		
Specimen	MV	SD	HG	MV	SD	HG	MV	SD	HG	MV	SD	HG
HDPE	25.50	0.87	c	1.17	0.04	d	1.20	0.03	d	—	—	—
WPC A	62.27	1.29	b	3.75	0.16	c	4.13	0.23	c	14.95	1.41	b
WPC B	60.58	3.91	b	3.69	0.11	c	4.42	0.15	b	16.13	2.53	a; b
WPC C	65.94	1.88	a	4.21	0.11	a	5.16	0.15	a	16.40	2.17	a; b
WPC GWP	65.82	1.81	a	4.04	0.19	b	5.12	0.30	a	18.31	1.81	a

MV: mean value; SD: standard deviation; HG: homogeneous group (groups with the same letter are not statistically different; $\alpha = 0.05$).

some extent, influence the desired interaction between hydroxyl groups of the fiber surface and the anhydride groups of the coupling agent.

However, further investigations are needed to verify these assumptions to the effect of lignin on bonding in WPCs and the influence of defibration temperatures in general.

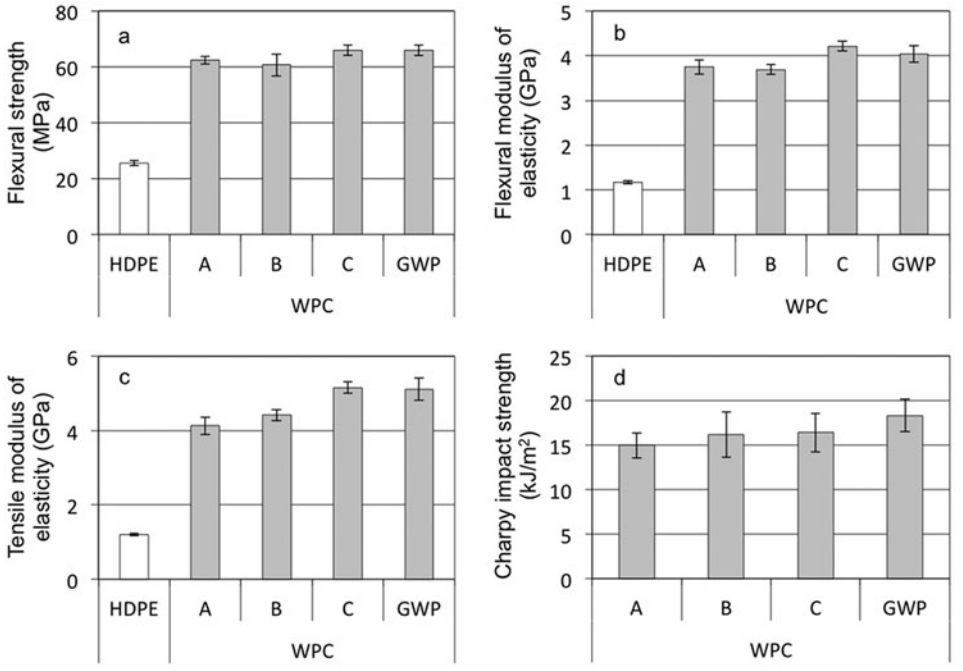

Figure 4. Mechanical properties of specimens: Flexural strength (a); Flexural modulus of elasticity (b); Tensile modulus of elasticity (c); Charpy impact strength (d).

As a side-effect of this study, the reinforcing effect of fiber application on the flexural and tensile properties was on an extraordinarily high level. The addition of wood fibers and coupling agent into pure HDPE increased in the case of WPC C flexural strength, flexural modulus of elasticity, and tensile modulus of elasticity by 159%, 260%, and 330%, respectively (Figure 4). For rough comparison only, in former studies the addition of wood fibers into polymer increased flexural strength, flexural modulus of elasticity, and tensile modulus of elasticity at maximum by 34–125%,[1, 2, 8] 84–186%,[1, 2, 8] and 40–225%,[1, 2, 5, 7, 8] respectively. The high reinforcing effect indicated that the combination of production process and material composition in this study were suitable to produce accurate WPCs and therefore could act as a guide for further studies.

The Charpy impact strength of pure HDPE could not be determined in conformance with DIN EN ISO 179-1 (2010). The reason for the failed Charpy impact strength determination was that using unnotched specimens did not lead to a break of the pure HDPE specimens but to deformation. However, this problem could be avoidable by using notched specimens for the test.

Tensile strength could not be determined in conformance with DIN EN ISO 527–2 (2012). The reasons for the failed tensile strength determination were specimen failures in the area of the clamping device. It is probable that they are caused by the high test speed (1 mm/min), the small specimen dimensions (type: 1BA) and, most of all, the inhomogeneous fiber distribution within the dumbbell-shaped specimens. A total of 33 out of 40 tested WPC specimens failed in the area of the clamping device and all of them failed at the specimen end with higher quantity of long fibers (fiber bundles). Therefore, it is evident that the end with higher quantity of long fibers was a weak point on dumbbell-shaped specimens. It can be assumed that the fibers were not reinforcing on that point due to an orientation rectangular to force direction. However, this problem could be avoidable by improving the injection molding process.

Physical Properties. An overview of the achieved physical properties of all specimens is given in Table 2. WPC densities were in the range of 1091–1104 kg/m^3 with no significant differences between WPC types. Compared with pure HDPE, the WPC densities were about 22% higher. Such density increases were, in all probability, induced by the compression of wood fibers (cell wall density about 1500 kg/m^3) and were reported in former studies.[12]

For thickness swelling and water absorption, 5 h of immersion in boiling water led to the same ranking: WPC GWP always had the lowest mean values, followed by WPC C, WPC A, and WPC B (Figure 5 a–b). Statistically significant differences were found for water absorption but not for thickness swelling, which may be caused by lower measuring accuracy of thickness swelling determination. However, existing differences in water absorption and thickness swelling revealed that the defibration conditions influenced physical WPC properties. The general statement that the wood fiber type or defibration condition can influence physical properties of fiber-based WPC is also in accordance with former studies.[6, 12]

In contrast to Migneault et al.,[6, 12] who discovered that a great fiber length led in fiber-based WPC to higher water absorption and volumetric swell, fiber length was no suitable explanation for all the results in this study. In fact, fiber length could be used as an explanation for the results of WPC GWP, since the shortest fibers led to lowest thickness swelling and water absorption, but not necessarily for the results of WPC B; medium fiber length led to highest thickness swelling and water absorption. In all probability, selective fiber lumps, which showed up for unknown reasons, especially in WPC B after water

Table 2
Physical properties of specimens

				1 h in boiling water						5 h in boiling water					
	Density (kg/m^3)			Water absorption (%)			Thickness swelling (%)			Water absorption (%)			Thickness swelling (%)		
Specimen	MV	SD	HG	MV	SD	HG	MV	SD	HG	MV	SD	HG	MV	SD	HG
HDPE	901	9	b	—	—	—	—	—	—	—	—	—	—	—	—
WPC A	1098	22	a	1.36	0.07	b	1.43	0.41	a	3.50	0.06	b	3.12	0.57	a
WPC B	1091	26	a	1.61	0.14	a	1.33	0.64	a	4.47	0.27	a	3.34	0.83	a
WPC C	1101	28	a	1.25	0.06	b	1.16	0.37	a	3.34	0.09	b; c	2.98	0.23	a
WPC GWP	1104	27	a	1.22	0.07	b	0.99	0.32	a	3.14	0.19	c	2.37	0.47	a

MV: mean value; SD: standard deviation; HG: homogeneous group (groups with the same letter are not statistically different; $\alpha = 0.05$).

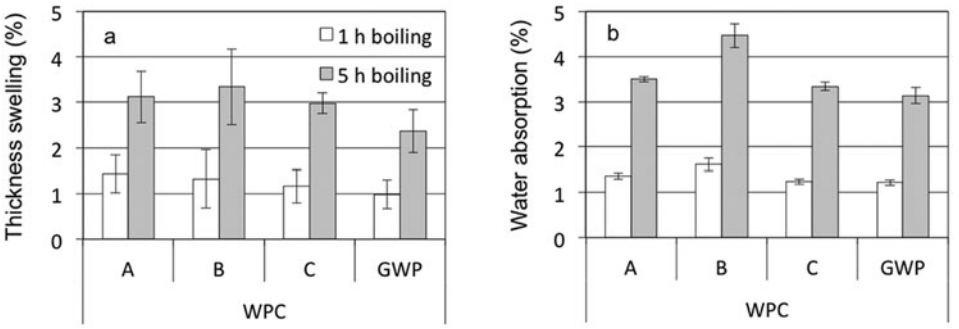

Figure 5. Physical properties of specimens: Thickness swelling (a); Water absorption (b).

immersion, allowed higher water penetration and caused the higher thickness swelling and water absorption. Further investigations are needed to verify the appearance of these fiber lumps.

For thickness swelling and water absorption, 1 h of immersion in boiling water led almost to the same tendency as 5 h of immersion in boiling water, whereas a lower percentage and less significant differences were found (Figures 5a and 5b). However, if only a rough estimation or comparison of WPC properties is required, it seems possible to evaluate thickness swelling and water absorption even more quickly than with the boiling water test of E DIN EN 15534–1 (2012).

Conclusion

This study was designed to investigate the effects of defibration conditions on mechanical and physical properties of fiber-based WPC. The data in this experiment revealed that differing defibration conditions led to statistically significant differences in the tested flexural, tensile, and impact properties as well as in the water absorption of WPC. Overall, TMP C and GWP performed best in fiber-based WPCs. This indicated that not necessarily great fiber lengths but mild defibration conditions could be advantageous, because of their tendency to lower thermal wood degradation and less lignin-covered fiber surfaces. The achieved extraordinarily high reinforcing effects of fiber application on flexural and tensile properties of WPCs verify the combination of production process and material composition and justify the use of fiber-based WPCs in general. According to the results in this study, neither the TMP- nor the GWP-producing industry is at a disadvantage in the area of supplying fiber-based WPC producers, because grinders and refiners seem to be equally suitable. Thus, it could be considered that the participation in the promising new fiber-based market does not primarily depend on pulp suitability but rather on pulp production cost and prices.

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