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Humidity's Effect on the Dynamic-mechanical Behavior of Phenol-formaldehyde Impregnated Beech Wood Veneer

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The dynamic-mechanical behavior of phenol-formaldehyde-impregnated beech wood (*Fagus sylvatica* L.) veneers was evaluated at various relative humidities. The aim was to identify the plasticizing effects of moisture on phenol-formaldehyde-impregnated veneers. Thin beech veneer samples were impregnated with a low and a medium molecular weight phenol-formaldehyde resin. The measurements were performed with a dynamic-mechanical thermal analysis equipped with a humidity generator. The storage modulus and the loss factor of the uncured phenol-formaldehyde-impregnated and the untreated control veneers were determined at a relative humidity of 15%, 60%, and 85% and a constant temperature of 25 °C. The untreated control veneers exhibited the smallest reduction of the normalized storage modulus. In contrast, the phenol-formaldehyde-impregnated samples exhibited a more intense relative reduction of the storage modulus with increasing relative humidity. This effect was more distinct for samples impregnated with low molecular weight phenol-formaldehyde than for those impregnated with medium molecular weight phenol-formaldehyde. In addition, higher solids contents in the resins led to even smaller normalized storage modulus. Consistently, the loss factor indicated a greater softening of the samples with an increasing humidity and higher solids content of the phenol-formaldehyde.

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INTRODUCTION

During plasticization, the cellular structure of wood is softened. Such plasticization of wood is necessary or useful for several applications in the woodworking industry. Thus, wood plasticization is applied in veneer manufacturing to facilitate the cutting process. Additionally, treatments of wood involving plasticization can be used for wood densification or for molding applications to create materials with improved mechanical properties or wood moldings. Typical wood moldings can be found in the furniture industry as bentwood or chair seats. Furthermore, wood moldings are used as decorative elements for interior trims in the automotive industry.

Plasticization can be induced by different procedures. The most traditional and well-known wood softening treatments involve the use of hot water or steam. Moreover, broad research has been conducted to develop further methods for wood plasticization and flexibilization. Among those studies, the effectiveness of treatments by impregnation with liquid ammonia (Schuerch 1963), formamide (Sadoh 1981), ethylene glycol (Sadoh 1981),

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or furfuryl alcohol (Herold *et al.* 2015) has been documented. However, all those procedures possess disadvantages, and none have led to any market-relevant products.

Apart from those procedures, phenol-formaldehyde (PF), in an uncured state, is also suitable for use as a wood plasticizer. Stamm and Seborg (1944) demonstrated the plasticizing effect of PF *via* the enhanced compressibility of PF-impregnated wood veneer compared to untreated veneers. The applicability of PF for wood densification was later affirmed by Shams *et al.* (2004). Additionally, PF is suitable for use as a plasticizing agent for molding applications (Franke *et al.* 2017) and, even at low retentions, leads to an improved moldability compared to water-saturated samples at ambient temperature (Franke *et al.* 2018). In the process it is required that the used PF resins (or other plasticizing agents) are able to penetrate into the cell walls. Previous investigations employing dynamic-mechanical thermal analysis (DMTA) delivered results verifying a temperature-induced plasticization of PF-impregnated wood samples (Franke *et al.* 2017a). The used PF were water-based resols and therefore the plasticizing effects can be attributed to the mixture of PF and water rather than just to the PF.

PF resin is popular in the wood industry as an adhesive, especially if the bonds require a resistance to heat and moisture, but PF is also suitable for wood impregnation. The advantages of PF-impregnated wood and wood veneers are the improved dimensional stability and reduced water absorption after curing (Stamm and Seborg 1942; Gabrielli and Kamke 2010), as well as an improved weather resistance (Evans *et al.* 2013; Franke *et al.* 2017b). Products for the transformer industry and mechanical applications of phenolic resin-treated and compressed laminated wood and pulp include Dehonit® and Trafoboard® manufactured by Deutsche Holzveredelung Schmeing GmbH & Co. KG and Röchling SE & Co. KG, respectively. Wood moldings of phenolic resin are treated and laminated wood, *e.g.*, for seat shells that are produced by Pagholz® Formteile GmbH.

The approach for this study was to enable a separate consideration of the plasticizing effects of both water and PF. For this purpose, the present study focuses on the softening behavior of PF-impregnated beech wood veneers at various relative humidity using a DMTA with a humidity generator. With the DMTA, a plasticization can be quantified under defined climatic conditions (temperature and relative humidity). Furthermore, the present study intends to contribute to the use of wood veneers for simultaneous forming, mold fixing, and composite production through target-oriented impregnation and material modification.

EXPERIMENTAL

Materials

Veneer samples

For the present investigation, samples were made from veneers of beech wood (*Fagus sylvatica* L.) (Berlin, Germany). Industrially manufactured veneers have been identified to be unsuitable for DMTA measurements due to structural damages resulting from the manufacturing process. The veneers for this work were manually produced according to Fig. 1. In the first step, a board with standing annual rings was cut into approximately 5-mm-thick slices. Afterwards, the slices were downsized with a wide-belt sander to create veneers with a defined thickness of approximately 0.7 mm in the tangential (T) direction. The final samples were cut from those veneers with dimensions of approximately 45 mm × 11 mm × 0.7 mm (radial (R) × longitudinal (L) × T). All samples

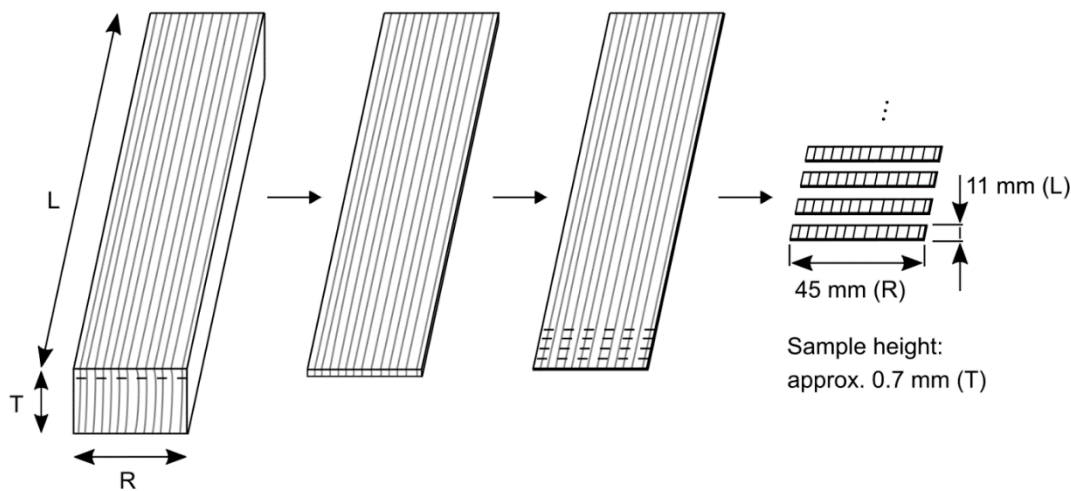were characterized by a defined anatomical orientation and homogeneous annual ring widths.

Fig. 1. Schematic representation of the sample production (L: longitudinal; R: radial; T: tangential)

Phenol-formaldehyde

Two water-based resols of different molecular weights were used for the impregnation: a low molecular weight PF (lmwPF) and a medium molecular weight PF (mmwPF). Both PF resins were products from Prefere Resins Holding GmbH, Erkner, Germany. The main technical specifications of the raw PF-resins are shown in Table 1. For impregnation, the resins were diluted with distilled water to solids contents of 10 wt%, 20 wt%, and 45 wt%.

Table 1. Specifications of the Undiluted Low (lmwPF) and Medium Molecular Weight PF (mmwPF)

PF Type	Molecular Weight (Mn)	Solids Content (%)	Viscosity (mPas)	Catalyst
lmwPF	246	45.2	13	NaOH
mmwPF	449	55.9	196	NaOH

Impregnation

Before impregnation, the veneer samples were dried in a desiccator with silica gel until a constant weight was achieved. Subsequently, the impregnation of the beech wood veneers with the PF was conducted in a desiccator under vacuum at about -90 kPa for 24 h. Afterwards, the impregnated samples were conditioned to a constant weight at 15% relative humidity and 25 °C for 24 h. The PF impregnated samples were tested in the uncured state. To minimize the risk of self-curing, each specimen was prepared separately and immediately before the DMTA measurement.

Methods

Dynamic Mechanical Thermal Analysis

The measurements were performed with a DMTA (Eplexor® 25; Gabo Qualimeter Testanlagen GmbH, Ahlden, Germany) with tension clamps from Gabo Qualimeter

Testanlagen GmbH (Gabo Qualimeter Testanlagen GmbH, Ahlden, Germany). To conduct the measurements at a defined climate, the DMTA was equipped with a humidity control device HYGROMATOR® (Gabo Qualimeter Testanlagen GmbH, Ahlden, Germany) and a climate chamber.

All samples were stressed dynamically in the radial direction with a sinusoidal frequency of 1 Hz. The load was applied perpendicular to the grain to minimize the impact of the mostly inert celluloses, in terms of moisture, on the dynamic mechanical behavior. Another advantage of the load applied in radial direction was the higher number of annual rings across the sample compared to a tangential alignment. The DMTA measurements were completed using a humidity sweep at a constant temperature of 25 °C. To quantify the plasticizing effects due to the increasing relative humidity, the storage modulus and the loss factor were determined. The measurements were conducted with a dynamic strain of approximately 0.03% of the clamp distance and a static strain of approximately 0.65% using a clamp distance of 35 mm. Each measurement consisted of three consecutive segments with a different relative humidity (15%, 60%, and 85%) and comprised of 14 measuring points. To ensure valid results, the measurements were repeated on three samples. The following figures display the arithmetic mean of the measured samples. A summary of the segment specific settings, including the relative humidity, temperature, and soak time, are given in Table 2. Because the samples were already pre-conditioned for 24 h at 15% relative humidity and 25 °C after impregnation, the soak time of the first segment was chosen to be shorter than in the subsequent segments.

Table 2. Segment-specific Settings

DMTA Segments	Relative Humidity (%)	Temperature (°C)	Soak Time (h)
1st	15	25	6
2nd	60	25	8
3rd	85	25	8

Moisture content and weight percentage gain

The viscoelastic properties of the impregnated veneers were strongly affected by the moisture content u and the amount of PF inside the wood sample. Thus, the moisture content u and the weight percentage gain (WPG) were determined in addition to the DMTA measurements using samples, which were prepared according to those samples used for the DMTA measurements. The moisture content u was calculated on four samples for each charge according to Eq. 1,

$$u = (m_u - m_0) / m_0 \times 100 \qquad (1)$$

where u is the moisture content (%), m_0 is the weight of the dry sample (g) at 80 °C, and m_u is the weight of the conditioned sample (g).

It should be noted that the moisture content calculation does not refer to the weight of the pure wood but of the whole sample, including the weight of the PF. The reason, as will be shown later, was that the PF seemed to have an immense impact on moisture absorption. Therefore, in addition to the untreated control veneers, the moisture absorption was also determined on the pure resins as further control samples. For this purpose the resins were dried to constant weight at only 60 °C for approximately 24 h to avoid a

polymerization. Subsequent to the drying process, the resins were conditioned at various relative humidity levels of 15% to 90% with a constant temperature of 25 °C.

After the determination of the moisture content, the samples were cured at 140 °C for 90 min to enable a calculation of the *WPG* according to Eq. 2,

$$WPG = (m_2 - m_1) / m_1 \times 100 \tag{2}$$

where *WPG* is the weight percentage gain (%), m_1 is the weight of the dry control sample (%), and m_2 is the weight of the dry, PF-impregnated and cured sample (g). The calculation of the WPG was completed for each charge on twelve samples.

Determination of ash residue

Due to the imprecise declaration of the catalyst sodium hydroxide in the safety data sheets, the ash residues of pure PF resins were determined. The ashing of the cured and dried PF resins was carried out in a muffle furnace at 1000 °C for approx. 4 h. The determination of ash residue was performed once for each PF type. The ash residues *AR* were determined according to Eq.3,

$$AR = (m_c - m_a) / (m_b - m_a) \times 100 \tag{3}$$

where *AR* is the ash residues (%), m_a is the weight of the dry and empty porcelain jar (g), m_b is the weight of the dry and cured PF in the dry porcelain jar (g), and m_c is the weight of the incinerated PF-impregnated and cured PF samples in the dry porcelain jar (g).

RESULTS AND DISCUSSION

Dynamic Mechanical Thermal Analysis

The dynamic-mechanical behavior, which makes it possible to draw conclusions about the plasticization of the samples, was evaluated by the storage modulus *E'* and the loss factor tan *δ*. According to DIN EN ISO 6721-1 (2011), the storage modulus is proportional to the stored energy and represents the stiffness of a viscoelastic material. The loss factor is a measure of the damping of a viscoelastic material (DIN EN ISO 6721-1 2011). Both the storage modulus and the loss factor can be used as indicators for plasticizing effects.

Table 3 shows the absolute values of the storage modulus at the initial relative humidity of 15%. The storage modulus of the lmwPF-impregnated samples (1757 MPa to 1965 MPa) was approximately in the range of the untreated samples (1801 MPa) at 15% relative humidity. In contrast, the storage modulus of the mmwPF-impregnated samples was distinctly increased compared to the untreated control samples in a rather dry environment. A higher solids content of the mmwPF resulted in a higher storage modulus. Considering the storage modulus, no softening of the PF-impregnated samples was detected under dry conditions.

Figure 3 displays the results of the normalized storage modulus as a function of the relative humidity for the lmwPF- and mmwPF-impregnated and the untreated control samples at a constant temperature of 25 °C. Because wood is an inhomogeneous material with considerably varying material properties, the results of the storage modulus were normalized for better comparability.

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Table 3. Arithmetic Mean of the Absolute Values of the Storage Modulus of the PF-impregnated Samples at 15% Relative Humidity, Based on Three Experiments Each

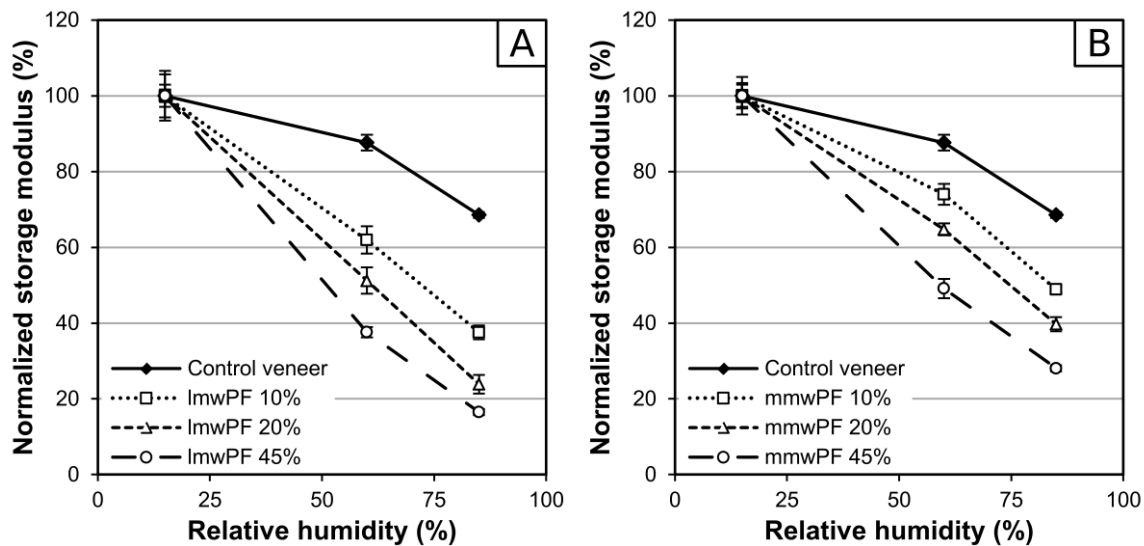| PF-Type	Storage Modulus at 15% Relative Humidity and 25 °C (MPa)		
	10% Solids Content	20% Solids Content	45% Solids Content
lmwPF	1879 (107)	1965 (129)	1757 (29)
mmwPF	2075 (103)	2270 (37)	2761 (92)
Control Veneers	1801 (53)		

Standard deviations are in parenthesis

Fig. 3. DMTA humidity sweeps; normalized storage modulus as a function of the relative humidity for the impregnated and native control veneers at a constant temperature of 25 °C. The standard deviation is illustrated by whiskers. A) Samples impregnated with lmwPF; B) Samples impregnated with mmwPF

The normalized storage modulus decreased distinctly with increasing relative humidity for all investigated samples. A reduced storage modulus can generally be an indicator for an enhanced plasticization associated with a reduction of the energy required for chain mobility (Rials and Glasser 1984). In the present case, the plasticization of the samples can be attributed to the increasing moisture content and to the solids contents of the PF resins.

At the same time, the PF-impregnated samples displayed a stronger reduction of the normalized storage modulus with increasing relative humidity than the untreated control samples. Even those samples impregnated with a solids content of 10% showed a reduction of the normalized storage modulus of approximately 51% (mmwPF) and 62% (lmwPF) at 85% relative humidity. In contrast, the untreated control samples revealed a distinct slight reduction of the normalized storage modulus (approximately 31%) at a

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similar relative humidity. A higher solids content of the PF resins resulted in a more pronounced effect. This was possibly related to the water absorption due to the catalyst sodium hydroxide, which was used in the PFs. This hypothesis is discussed more precisely in the section titled "Moisture Content".

Consistent with an enhanced moldability of the PF-impregnated veneers compared to water-saturated veneers (Franke *et al.* 2018), the reduction of the storage modulus was more pronounced for the PF-impregnated veneers than for the untreated control veneers. In addition, the reduction of the normalized storage modulus was more distinct for the lmwPF-impregnated samples than for the mmwPF-impregnated samples. This observation seemed coherent because the extent of cell wall penetration is strongly dependent on the molecular weight of the PF: Lower molecular weight results in easier penetration of the PF into the wood cell wall (Furuno *et al.* 2004). Earlier studies proved a greater PF cell wall uptake for lmwPF than for mmwPF/hmwPF samples due to the smaller molecular weight (Franke *et al.* 2017c; Biziks *et al.* 2019). In agreement with these findings, those samples impregnated with lmwPF and a solids content of 45% revealed the lowest normalized storage modulus at 85% relative humidity, displaying a reduction of approximately 83% (Fig. 2). In contrast, the mmwPF-impregnated samples with a solids content of 45% showed a reduction of approximately 72%.
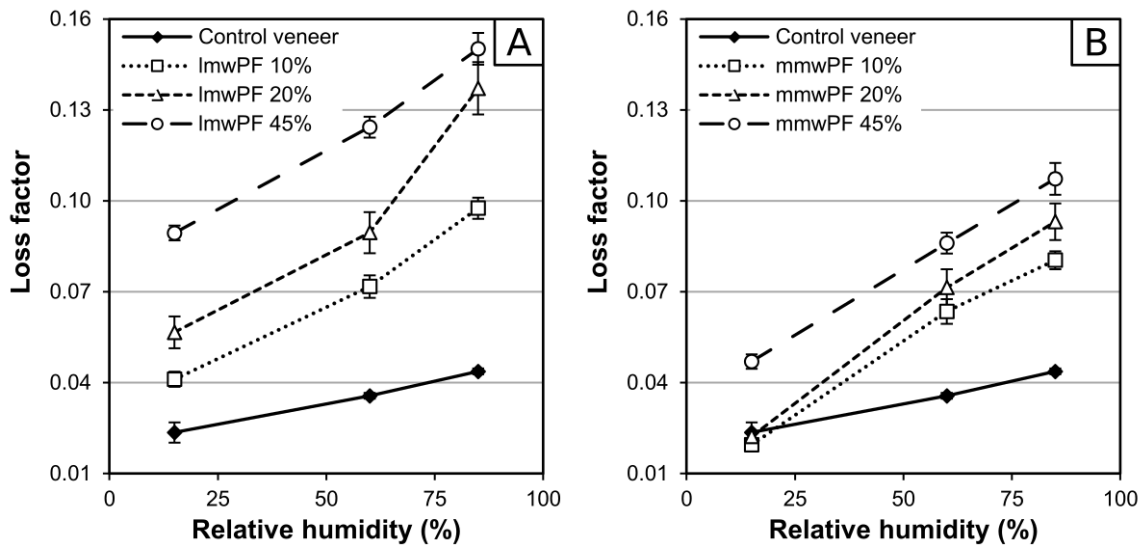

Fig. 4. DMTA humidity sweeps; loss factor as a function of the relative humidity for the impregnated and native control veneers at a constant temperature of 25 °C. The standard deviation is represented by whiskers. A) Samples impregnated with lmwPF; B) Samples impregnated with mmwPF

In addition to the results for the storage modulus, Fig. 4 displays the results of the loss factor as a function of the relative humidity for all samples at a constant temperature of 25 °C. In agreement with findings from the storage modulus, an elevation of the relative humidity from 15% to 85% led to an increase of the loss factor for all investigated samples. However, this effect was much more pronounced for the PF-impregnated veneers compared to the native control veneers. A high loss factor is, in addition to a low storage modulus, another indication of a plasticization that corresponds to sample softening.

The results showed that higher solids contents of the PF corresponded with higher loss factors at the same relative humidity. Moreover, the lmwPF-impregnated samples

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displayed a higher loss factor than the mmwPF-impregnated samples under similar conditions. For example, those samples impregnated with lmwPF and 45% solids content showed a loss factor of approximately 0.15 at 85% relative humidity. In contrast, the mmwPF-impregnated samples had a loss factor of approximately 0.11 at 85% relative humidity. The dependence of the plasticization on both the solids content and molecular weight of the PF has already been suggested by Franke *et al.* (2018) and can be confirmed in the present study.

The influence of moisture on the softening of wood components is well-known. Crystalline cellulose does not interact noticeably with water (Cave 1978) and therefore does not display any moisture-induced softening (Salmén and Olsson 1998). Kelley *et al.* (1987) identified the glass transitions of hemicelluloses and lignin. These authors showed the shift of the glass transitions of amorphous wood components towards lower temperatures with rising moisture contents. However, the main softening under wet conditions is attributed to the glass transition of lignin (Irvine 1984; Olsson and Salmén 1997). It was found that the glass transition of lignin occurs under water-saturated conditions approximately between 60 °C and 90 °C (Irvine 1984). In contrast, the glass transition of hemicelluloses under water-saturated conditions is below ambient temperature (Kelley *et al.* 1987; Olsson and Salmén 2003). Therefore, the results obtained that showed a decreasing storage modulus and an increasing loss factor during an increasing relative humidity were most likely caused by a relaxation of the hemicelluloses. Whether the results were also caused by a decreasing glass transition of lignin due to the PF impregnation is not clear. On the one hand, the results obtained suggested a decreasing glass transition of lignin in the range of the test temperature (25 °C). In contrast, the relatively high volume of previous data under water-saturated conditions contradicts such a reduction of the glass transition.

Moisture Content

The moisture content was determined as a function of the relative humidity for the lmwPF and mmwPF-impregnated and for the native control samples, as well as for pure PF resin (Fig. 5). Expectedly, the moisture content of the examined samples increased with increasing relative humidity. Apart from the samples impregnated with a solids content of 10% and 20% mmwPF, which exhibited almost equal moisture contents, a higher solids content of the used PF resulted in a higher moisture content under the same humidity conditions. Especially at impregnations with a solids content of 45%, the lmwPF samples showed higher moisture contents than the mmwPF samples. For example, the moisture content of the samples impregnated with lmwPF containing 45% solids content was approximately 36.4% at a relative humidity of 85%, whereas the moisture content of the samples impregnated with mmwPF containing 45% solids content was only approximately 26.5%. This finding could be attributed to the amount of sodium hydroxide that was used as catalyst in the PF resins. An attendant experiment has shown a strongly hygroscopic behavior of sodium hydroxide. However, the exact amounts of sodium hydroxide were unknown, but according to the before mentioned hypothesis, the lmwPF should contain more sodium hydroxide than the mmwPF. With a declaration between 1% to 2% sodium hydroxide for the lmwPF and 1.1% to 2% sodium hydroxide for the mmwPF, the safety data sheets were unfortunately not helpful in this regard.
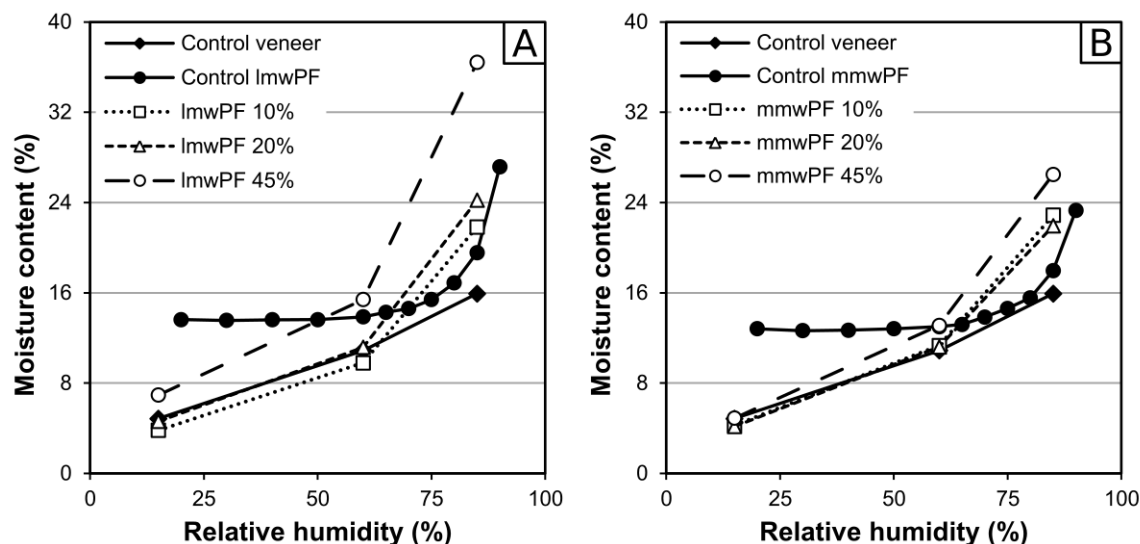
Fig. 5. Moisture content as a function of the relative humidity for the impregnated and native control veneers as well as the pure PF (Control lmwPF/Control mmwPF) at a temperature of 25 °C. A) Samples impregnated with lmwPF; B) Samples impregnated with mmwPF

The results of the pure resins seemed to be consistent with the results of the PF-impregnated samples. Up to a relative humidity of 65%, the moisture contents were almost constant, but subsequently the moisture content slowly increased at first and later enormously increased. This observation was similar for both resins but more pronounced for the lmwPF than for the mmwPF. In addition, the results of the pure resins supported the assumption that the lmwPF contained more sodium hydroxide than the mmwPF.

Ash Residue from the PF Resins

The ash residue corresponds to the inorganic content in the PF samples and allows conclusions to be drawn about the amount of sodium hydroxide. An ash residue of approximately 3.9% was determined for the lmwPF. In contrast, the ash residue of mmwPF was approximately only 2.9%. Therefore it can be assumed that the sodium hydroxide content was distinctly higher in the lmwPF than in the mmwPF. That finding supported the hypothesis that the moisture content of the impregnated samples can be attributed to the sodium hydroxide content of the used PF resins.

Weight Percentage Gain

The WPG was determined as a function of the varying solids content. The results are collected in Table 4. The results showed only minor differences in WPG between the lmwPF- and mmwPF-impregnated samples. Yet, the WPG of the mmwPF-impregnated samples was always higher than the WPG of the lmwPF-impregnated samples.

Biziks *et al.* (2019) investigated the WPG of PF as a function of various molecular weights. The results in their study revealed no clear trend. The observed differences in the WPG were not attributed to the molecular weight of the resins but to differences in the wood samples, *e.g.*, differences in the distribution of annual rings (Biziks *et al.* 2019). A study from Franke *et al.* (2017c) shows similar results in terms of the WPG as reported by Biziks *et al.* (2019). However, it should be noted that the WPG does not provide any information on the PF distribution in the wood structure. In contrast to the WPG, the bulking coefficient, which allows conclusions about the PF uptake into the cell walls, is

strongly related to the molecular weight of the PF (Franke *et al.* 2017c; Biziks *et al.* 2019). The authors reported that lmwPF induced a greater bulking coefficient than PF resins with a higher molecular weight. Hence, it can be assumed that despite the slightly lower WPG, the lmwPF is present in the cell walls in higher amounts than the mmwPF. It is worth noting that in the study by Franke et al (2017c) the same PF resins were used as in the present study.

The differences in WPG may also be due to different amounts of PF on the sample surface. Although the WPG influenced the plasticization of veneers, as evident in Fig. 2 and Fig. 3, the small differences in the WPG between the two resols at similar solids contents seemed to have a minor impact on the dynamic-mechanical behavior only.

Table 4. Weight Percentage Gain as Arithmetic Mean for Different Solid Contents Based on 12 Experiments Each

PF Type	Weight Percentage Gain (%)		
	10% Solids Content	20% Solids Content	45% Solids Content
lmwPF	13.41 (1.31)	22.00 (1.44)	46.01 (2.40)
mmwPF	16.82 (1.8)	23.03 (0.84)	48.52 (1.88)

Standard deviations are in parenthesis

CONCLUSIONS

1. Dynamic mechanical thermal analysis (DMTA) proved to be a feasible method for detecting changes in the dynamic-mechanical properties of wood due to defined changing humidity conditions.

2. The DMTA measurements showed an enhanced plasticization of PF-impregnated veneers with increasing relative humidity and solids content. To achieve efficient plasticization by phenol-formaldehyde (PF), high weight percentage gain (WPG), and high moisture contents were explicitly necessary.

3. The lmwPF-impregnated samples revealed greater moisture-induced softening than the mmwPF-impregnated samples, likely due to higher moisture absorption and easier cell wall penetration.

4. The determination of the ash residues from the PF resins leads to the conclusion that the observed high moisture absorption of the PF-impregnated samples is probably related to the amount of the hygroscopic catalyst (sodium hydroxide) in the PF resins used.

5. The obtained DMTA results showed the distinct impact of the PF type characterized by different molecular weights, and of the WPG on the plasticization of wood. The findings strongly emphasized the major influence of the relative humidity on the plasticization of PF-impregnated veneers. Therefore, the molecular weight and the WPG, as well as the relative humidity proved to be important parameters for molding applications and wood densification and should be considered in future applications.

ACKNOWLEDGMENTS

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