**ORIGINAL ARTICLE** 



# Tribological properties and related effects of compressed, thermally modified and wax-impregnated wood

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#### Abstract

This paper develops a process for the preparation of modified wood with low friction and low wear for tribological applications such as self-lubricating bearings. Two types of wood, beech (*Fagus sylvatica*) and robinia (*Robinia pseudoacacia*), have been studied and the results compared with naturally lubricated native lignum vitae. The process developed consists of plasticisation followed by compression in a mould, thermal modification and subsequent wax impregnation. Plasticisation was carried out by conditioning the samples to a low equilibrium moisture content of 10%, followed by heating to a sample core temperature of 80 °C. This process protects the internal wood structure from mechanical damage during densification. After plasticisation, the wood was compressed in a press mould. A low springback effect (SBE), resulting in compression of up to 40%, was achieved by unloading the mould without opening it. This step optimises compressive strength and hardness. Subsequent heat treatment reduces thickness swelling by up to 85%. Finally, a wax impregnation was applied to reduce friction. Sliding wear tests on modified beech wood have shown that the lowest wear occurs in the cross-sectional orientation (load perpendicular to the fibre ends; rxt orientation). Sliding friction studies using a steel ball on a ball-on-disc tribometer showed that compressed and thermally modified samples impregnated with rapeseed wax or beeswax exhibited coefficients of friction in a range of 0.08 to 0.09. These values are almost four times lower than those of plain compressed wood and even lower than those of lignum vitae, which was used for plain bearings decades ago. This study clearly demonstrates the high potential of compressed, thermally modified and wax-impregnated wood.

# 1 Introduction

Wood is a complex natural nanocomposite with a number of interesting functional properties. One of these is that wood can be used in tribological applications. The tribological properties of wood have been exploited for centuries. Scientific studies of grease-lubricated wood for tribological applications were carried out as early as 1699 by Guillaume Amontons and later in more detail for unlubricated wood pairings by Charles Augustin de Coulomb (Popova and Popov 2020). Subsequently, wood was modified by various processes to adapt it to tribological applications. In the 1920s, pressure-lubricated modified wood bearings came into use (Turk 1927). Hellmanns and Rohde (1943) reported on oil-filled and pressed woods in practical tests in rolling mills. Lazarev (1991) postulated that pressed woods could not be replaced by synthetic polymers for water pump shaft bearings. Sathre and Gorman (2005) conducted the first comprehensive empirical study using wood as a radial plain bearing. In this study, maple wood and basswood were filled with olive oil, mineral oil, peanut oil and beeswax, among others. Beeswax was found to have the lowest static and dynamic coefficients of friction in the range of 0.09 and 0.1. Extending this study, Kim et al. (2008) filled paulownia wood (low density and high porosity) with polyethylene glycol (PEG) and tested it with a pin-on-disc setup. The coefficient of friction was found to be  $\mu = 0.36$ .

Investigations on the friction and wear behaviour of the three main wood orientations were carried out by Friedrich et al. (2021) on lignum vitae, black fibre palm and spruce. Tribological tests were conducted using a 100Cr6 steel ring as a counter sample. The woods were tested in their natural state and black fibre palm and spruce were then filled with sunflower oil. The filled black fibre palm samples exhibited the lowest friction values ( $\mu = 0.12$ ) in the cross section (load

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perpendicular to the fibre ends). Despite higher coefficients of friction, the lowest wear was measured for the much denser lignum vitae samples. In addition to oil impregnation, Liu et al. (2022) carried out a fluorination treatment, which meant that the lubricating oil was better absorbed and stored in the porous structure of the wood analysed. As a result, they observed more stable friction and wear behaviour. A previous investigation by the authors (Waßmann and Ahmed 2020) dealt with the modification of beech wood in order to qualify it for tribological applications. The essential components of this study were to minimise friction by means of wax impregnation and at the same time increasing the contact area of the material to improve the material strength and hardness, which was achieved by compressing the inhomogeneous and porous wood structure by mechanical compression in a heated press mould. This is defined as Thermal-mechanical densification TMD (Li et al. 2017).

There are a number of issues in the initial study (Waßmann and Ahmed 2020) that need to be addressed before moving on to applications such as self-lubricating bearings. The first issue is that the process sequence needs to be altered from that reported in the initial study. Instead of wax impregnation at the beginning, it is important that impregnation is carried out at the end of the process. The main reason for this change is that wax-impregnated material is cumbersome to bond to other wood; as a result, one is then limited by the finite tree diameter, thereby limiting scalability. This means that no hot wax can be filled into the wood samples prior to compression. However, the wood sample moisture and temperature are decisive parameters for successful plasticisation. This leads to the second issue: The process parameters for plasticising and wood compression must be chosen in such a way as to avoid destruction of the wood structure and to minimise the springback effect (SBE). The SBE is the property of a compressed material to partially or fully return to its original shape after removal of the deformation force (Sanne et al. 2020). It is important to minimise the SBE as much as possible in order to achieve the highest possible compression in the shortest time (see subsection 2.1 for more details). The third issue is to take the swelling and shrinkage behaviour of wood into account. For tribological applications in particular, it is important to know the swelling behaviour of components in order to be able to guarantee shape and position tolerances to a certain extent. Otherwise, there is a risk of components blocking,



• Equilibrium moisture content before pre-heating

thereby adversely affecting the overall function of a system. Niels Morsing (1998) introduced a thermal modification to reduce swelling and shrinkage. As such a treatment also makes the wood brittle (Wehsener et al. 2018), it needs to be examined with regard to friction and wear. The fourth issue is to confirm the preferred wood orientation that should be used in subsequent studies. The final issue is to determine whether the limited and expensive beeswax can be replaced with other waxes that are readily available and less costly. All of these issues are addressed in this paper and together form the core innovation of this work.

This study develops an application-oriented process sequence that, at the end, results in wax-impregnated solid wood with a low SBE, optimised for thickness swelling using an economical and readily available wax. The preferred wood orientation for tribological applications is also presented. Tribological results indicate that low friction and wear are achieved.

# 2 Materials and methods

In this study, beech (*Fagus sylvatica*) and robinia (*Robinia pseudoacacia*) solid woods were used. Beech was chosen for its good impregnability. Robinia was included in the study because of its higher dimensional stability. The identification for these samples can be defined as follows in Fig. 1.

If the duration of thermal treatment or the impregnation medium is not specified, the respective process step has not taken place. The tribological results presented on solid woods have been compared with native lignum vitae, a type of wood used for sliding bearings before the invention of synthetic polymers, and ultra-high-molecular weight polyethylene (UHMWPE), a synthetic polymer currently used for sliding bearing applications.

# 2.1 Sample description, springback effect (SBE) and thickness swelling (TS)

In this section, various sample variants are presented which were investigated with regard to the springback effect (SBE) and thickness swelling (TS). The results of these investigations lead to a *single* variant that is further modified for tribological testing in Sect. 2.2.

	Sample v	ariants				
	MC10	MC10	MC10	MC10	MC17	MC17
	T120a	T120b	T160	T200t24	T120	T160
Processing step PS						
1. Kiln-drying	Drying temperature $T_{oven} = 103 \pm 2$ °C (DIN EN 322)					
2. Conditioning	Relative humidity		RH=65%	RH=90%		
3. Pre-heating	$T = 80 \degree C$					
4. Compression until core temperature is reached	Compression degree CD approx. 40%					
	120 °C		160 °C	120 °C	160 °C	
5. Thermal treatment	Х		Х	200 °C, 24 h X	Х	Х
6. Drying	Drying temperature $T_{oven} = 103 \text{ °C}$					
7. Cutting	Every sample (Fig. 2, right, below)					

**Fig. 2** (a) Press mould with guide pillars (1), heating elements (2), PT100 element (3), unpressed beech sample (4) and thermocouple (5). (b) Idealised process diagram with the values of the MC10T120 variant. (c) Side view of compressed wood with thermocouple (grey). The part between the dotted lines was later sawed, prepared and used for the TS investigations in this study



#### 2.1.1 Sample description

The individual processing steps (PS) and variants for the springback effect (SBE) and thickness swelling (TS) investigations are addressed for the samples listed in Table 1. The dimensions of the delivered wood samples were  $40 \times 18 \times 36$  mm<sup>3</sup>.

**PS 1 - kiln-drying:** All wood samples were oven-dried (Nabertherm N30) in order to determine impregnation and moisture content by weight.

PS 2 - conditioning and PS 3 – pre-heating: Wood must first be plasticised to prevent damage to the wood structure during compression. For this purpose, the softening temperature of the lignin must be set at a defined moisture content of the material. One study (Salmén 1990) provides a good overview of the softening temperature as a function of the moisture content of wood components. Another reference recommends heating wood with an initial material moisture content of MC = 10% in the press to a minimum temperature of T = 80 °C and then pressing it (IaFB e.V. 2010). Kúdela et al. (2018) proposed an initial sample moisture content between 17% and 20%. These modified samples, compressd at ≥ 180 °C, should exhibit a lower springback effect. In this investigation, the samples were first conditioned in a climate chamber (Weiss WK 340) at relative humidity levels (RH) of 65% and 90%. After reaching a mass difference  $\Delta m$  of less than 0.1% in 24 h, they were transferred to the press mould. The samples were brought to a pre-heating temperature of 80 °C in the unloaded press mould (Fig. 2a). The press mould was heated in a range of 200 °C to 210 °C by four heating elements (Fig. 2a - (2)). The temperature was measured with a PT100 element (Fig. 2a - (3)) and controlled by an Arduino 2560 Mega. The core temperature of the sample was monitored using a data logger (Ahlborn Almemo 2290-4 V5) and a thermocouple (Fig. 2a - (5), ZA 9000-FSL Norm E4, FeCu-Ni), which was placed in the wood sample (Fig. 2a - (4) and the schematic in Fig. 2c).

**PS 4 - compression:** An idealised process diagram is shown in Fig. 2b for a sample identified as MC10T120. As soon as the temperature of 80 °C was reached, compression of the porous wood structure began, increasing the density of the material. The stamp velocity was approximately 0.2–0.3 mm/s. The stamp displacement of 16 mm was achieved when the press mould was completely closed. The load applied by the hydraulic press was approximately 100 kilonewtons. The press mould for variant MC10T120a (Table 1) was fully opened when the core temperature was

reached. For the variant MC10T120b (Table 1), the mould was unloaded and only opened when there was no visible sign of water vapour.

PS5 - thermal treatment: A property of compressed wood is that the first moistening phase leads to decompression. As the moisture content increases, the wood is plasticised, releasing the energy stored in the microfibrils (Norimoto et al. 1993), resulting in increased swelling. The material swells in a defined area, but never returns to its previous compressed state. Technically, the dimensional difference between kiln dry after compression and kiln dry after wetting cycles is called Recovery of Set (RoS) (Seborg et al. 1945; Stamm 1948). Without affecting the hygroscopic system, RoS occurs to the extent that compressed wood can swell back to its original state (Morsing 1998). This behaviour can be reduced through hydrophobisation. One way is by altering the hydrophilic components of the wood. This can be achieved by thermal treatment (Sandberg et al. 2013; Kúdela et al. 2018). In this method, the effect of reduced water absorption is due to the degradation of hydrophilic components (Hill 2006), the conversion from hemicellulose into furfural polymers (Stamm 1964; Norimoto et al. 1993) and an increase in crystallinity (Norimoto et al. 1993). According to Morsing (1998), a RoS of thermo-mechanically densified (TMD) wood can only be eliminated from a temperature of 200 °C onward and an exposure time of at least 20 h. However, brittleness increases as a result of the thermal treatment (Wehsener et al. 2018). Taking all of this into consideration, the compressed samples were stored in an oven at 200 °C for a defined period of time.

**PS6 - drying:** All wood samples were oven-dried (Nabertherm N30) to determine impregnation and moisture content by weight. In addition, the sample thicknesses for the SBE tests were determined.

**PS 7 - cutting:** The modified samples were cut into three pieces using a table saw (Proxxon Micromot FET) (Fig. 2c, dotted lines), whereby only the central piece was used for

thickness swelling investigations. The final samples had dimensions of approximately  $20 \times 20 \times 15$  mm<sup>3</sup>.

#### 2.1.2 Springback effect (SBE)

The springback effect (SBE) (Sanne et al. 2020) is defined by the following equation (Pelit and Emiroglu 2020),

$$SBE \ (\%) = \frac{h_2 - h_1}{h_1} x \ 100 \tag{1}$$

where  $h_1$  is the sample thickness compressed in the press mould and  $h_2$  is the thickness after compression and kiln-drying.

#### 2.1.3 Thickness swelling (TS)

The thickness swelling (TS) is given as,

$$TS \ (\%) = \frac{h_3 - h_2}{h_2} x \ 100 \tag{2}$$

Here  $h_3$  is the sample thickness in the compression direction after conditioning. As the swelling times vary for the different variants, the most hydrophobic modification was chosen as the time indicator. This means that when MC10T200t24 reached a mass difference  $\Delta m$  of less than 0.1% in 24 h (Kern ABT 220-5DM), all variants were transferred to the next higher humidity level. The humidity levels were RH = 50%, 75% and 98%.

#### 2.2 Tribological tests

#### 2.2.1 Sample description

Table 2 shows the process parameters for the preparation of beech and robinia samples for tribological testing. The parameter values for the process steps PS 1 to PS 7 were

	Sample variants				
	MC10T120	MC10T200t3	MC10T200t24		
Processing step PS					
1. Kiln-drying	Drying temperature Toven = $103 \pm 2$ °C (DIN EN 322)				
2.Conditioning	Relative humidity RH=65%				
3.Pre-heating	T = 80  °C				
4.Compression until core temperature is reached	$T_{core} = 120 \ ^{\circ}C$				
5. Thermal treatment	Х	200 °C for 3 h	200 °C for 24 h		
6.Drying	Drying temperature $T_{oven} = 103 ^{\circ}\text{C}$				
7. Cutting	Every sample (Fig. 2c)				
8. Impregnation	Vacuum for 5 min. with different waxes				
9. Conditioning	Omitted for reasons of comparability				
10. Surface finishing	Sandpaper, Grit: P120, P600				

Table 2 Overview of the individual process steps of the variants for tribological studies

defined *after* successful execution and evaluation of the tests from subsection 3.1. The parameters of the variant MC10T120b from Table 1 were selected, as these provide a good compromise between the SBE value and press loading duration. Process steps PS 8 and PS 10 were added specifically for tribologically tested samples.

**PS1 - kiln-drying to PS7 - cutting:** Process steps PS 1 to PS 7 are identical to those described in subsection 2.1. The parameter values and sequence of the steps are the same as for variant MC10T120b (best variant of subsection 3.1).

**PS8** - impregnation: Samples were impregnated with either hydrogenated rapeseed wax (Tefawax R50, slip melting point mp=47–54 °C (enclosed data sheet)) or animal stearic acid (BAEROCID L-1 A, melting point mp=57 –61 °C (enclosed data sheet)). A chamber equipped with a rotary vane vacuum pump (Labovac PK 8D,  $p_{min} = 2 \times 10^{-4}$ mbar) was used for wax impregnation. The duration of wax impregnation at 100 °C was 5 min. The degree of filling varied with the degree of compression and the wood type.

**PS 9 - conditioning:** Typically, samples are brought to a defined equilibrium moisture content MC prior to testing (DIN 50014). As the dimensional stability of the different variants is defined by the parameters of the respective modification steps and the type of wood, which influences the density and thus the friction and wear, this process step was omitted.

**PS 10 – surface finishing:** lane (checked by a knife-edge square) and plan parallelism (checked using a depth sensor) were achieved using a milling machine (3018 Pro) and silicon carbide grinding paper (BUEHLER–MET II).

#### 2.2.2 Disc grinding machine

A disc grinding machine (Jean Wirtz -type: Polo 250/2, Fig. 3, left) was used for the abrasive wear tests. For this purpose, cuboids with dimensions of  $28 \times 28 \times 10$  mm<sup>3</sup> were inserted into the sample holder and placed on grinding paper with a grain size of P120. After setting the following test parameters: nominal contact pressure p of approximately 0.08 MPa, test duration t=5 min., synchronous mode and velocity level 1 (parameter setting on the machine), the machine was switched on. The grinding disc (Fig. 3, right, dark grey) and sample holder (Fig. 3, right, light grey) rotate synchronously in the same direction. Due to the off-centre setup, the relative test speed varies between the samples and the grinding disc. The structural complexity of natural wood, coupled with the dynamic testing procedures, makes it very difficult, if not impossible, to estimate the real contact areas.

#### 2.2.3 Ball-on-disc tribometer

Tribological testing was performed using a standard ball-ondisc tribometer (Fig. 4, left). In the setup used in this study, a fixed ball (the counter body) slides against a moving sample (the body). The sample (1) is clamped in a chuck (2). The ball holder (4) is attached to a measuring arm (3). The measuring arm (3) is mounted in such a way that when a load (normal force) is applied using a given dead weight (5), the sample (1) experiences a normal force (or load). When the measurement is started, the chuck (2) and the attached sample (1) begin to rotate, causing relative movement between the flat sample and the ball. The sliding friction occurring





**Fig. 3** Left: Disc grinding machine (Jean Wirtz -type: Polo 250/2) with wood samples. Right: Schematic diagram showing the direction of rotation of the grinding disc (dark grey), sample holder (light grey) and samples (brown) in synchronous mode **Fig. 4** Standard ball-on-disc tribometer used for tribological investigations (Anton Paar TRB 3) with flat sample (1), chuck (2), measuring arm (3), counter sample holder (4), dead weight (5), LVDT sensor (6). Right: Thermal and compressed modified beech wood sample with wear track after a tribological measurement fixed on a AISI 52,100 / 100Cr6-disc (L longitudinal, T tangential, R radial)



between these two bodies causes a displacement of the measuring arm (3) along the red arrows. This displacement is detected by means of a sensor (standard LVDT) (6). The product of the displacement and the known spring constant of the measuring arm (3) results in the friction force output by the system. The quotient of the measured friction force and the known normal force provides the Coefficient of Friction (CoF) output.

The measurement environment was enclosed in a Plexiglas (PMMA) box. Unless otherwise stated, the following parameters were used for the tribological investigations: normal force F = 10 N, sliding velocity v = 0.1 m/s, sliding distance s = 1,000 m or 10,000 m, test track radius r = 5 mm, relative humidity RH =  $30 \pm 2\%$ , temperature  $T = 20 \pm 2$  °C, ball (AISI 52100 / 100Cr6) diameter d = 6 mm, the tested orientation of the wood samples was the cross section (rxt orientation) (shown in Fig. 3, right).

Each modification has different swelling characteristics resulting in a significant difference in compression after conditioning. Therefore, in order to compare the modification processes with one another, all tribological measurements were conducted on kiln-dried wood in a dry environment.

Measured wear in this study is defined as particle removal *and* plastic contact deformation. The specific wear rate k, plotted in the following figures, is defined according to Eq. 3 (Gesellschaft für Tribologie e.V. 2002; Lancaster 1973) as,

$$k = \frac{W_V}{s \cdot F_N} \tag{3}$$

where  $W_V$  is the wear volume in mm<sup>3</sup> measured by noncontact optical profilometry. Due to the inhomogeneous wear track widths resulting from the structure of the materials examined, these are average values. They are calculated from the respective minimum and maximum values of the considered wear region. Since the sliding distance s is given in m and the normal load  $F_N$  in N, k is given in mm<sup>3</sup>/Nm.

# **3** Results and discussion

#### 3.1 Springback effect

The springback effect (SBE) was calculated for the different modifications according to Eq. 1 and is plotted in Fig. 5. The highest arithmetic mean value of all sample variants of 9.7% is represented by variant MC10T120R (moisture content MC=10%, core temperature  $T_{core} = 120$  °C, Robinia R). The largest scatter can be assigned to the MC17T120 samples. As the core temperature increases, the SBE value generally decreases, with the exception of the variant MC10T120b. The modification procedure for this variant is the same as for MC10T120a, but there was no direct opening of the press mould once the core temperature had been reached. Instead, the mould remained in this state until no rising water vapour could be optically detected, at which point a significant decrease in SBE and scatter was observed.

The assessment of the samples and the determination of the sample thicknesses were carried out in accordance with PS 6 as described in subsection 2.1. There were no visible external defects. The structural defects shown in Fig. 5 (below) only became apparent after the sample preparation in accordance with PS 7. Figure 5 shows the samples with the lowest SBE of the respective variant (indicated by the minimum of the scatter).

In the case of sample variants MC17T120 and MC17T160, which were prepared based on the parameters set by Kúdela et al. (2018), a low SBE can be explained by defects in the wood structure. In this case, a large part of the mechanically



**Fig. 5** Above: Average SBE (marked in red) from five modified samples per variant. Below: Cross-sectional view of the various samples with a view of the rxt orientation (not to scale). The identification is

made up of the sample moisture content (MC) before the samples were modified, the sample core temperature  $(T_{core})$  when the press mould was opened and the wood type (R=Robinia, no specification=Beech)

stored energy is dissipated by destruction of the wood structure. This is mainly due to the high vapour pressure present inside the sample, where temperatures must be higher than the core temperature in the destroyed areas. This is evident from the colour gradient of the material (Fig. 5), which becomes darker as the temperature rises. The size of the scatter in the measured values for MC17T120 can be linked to the colour gradient and the extent of the visible structural defects. The SBE increases with lighter colour and fewer defects.

Samples with an equilibrium moisture content of MC = 10% show no visible defects under the microscope (magnification of 500x). Even the variant MC10T200t24, which was heated to a core temperature of 200 °C for 24 h, exhibited no visible structural defects.

The question now arises as to why the MC10T120b modification variant results in a reduced SBE value with significantly less scatter than the equivalent MC10T120a modification. This effect can be explained by the unloading of the sample and the dwell time associated with the unloaded, but closed, press mould. This allows the pores of the wood to open slightly, allowing water vapour to escape, thereby reducing the vapour pressure inside the samples. The release of water vapour also leads to significant reduction in the sample moisture content to a value of MC < 1%. This causes a direct increase in the softening temperature of lignin and hemicellulose (Salmén 1990; Pettersson et al. 2021), leading to a transition from the rubbery to the glassy

region. The reduced moisture also leads to the formation of new hydrogen bonds between structural elements (Norimoto et al. 1993). These bonds have a positive influence on a low SBE. The effect shown for MC10T160 can also be explained in this manner, with the sample variant achieving a lower final moisture content with the press mould closed.

#### 3.2 Thickness swelling

Figure 6 shows the thickness swelling (TS) of differently modified variants. The non-thermally modified variant MC10T120 serves as a reference. An increase in core temperature to a value of 200 °C and a dwell time of one hour showed a decrease in swelling, particularly at a high relative humidity (RH=98%). This effect was significantly increased by extending the holding time to 24 h and is clearly visible at all humidity levels.

In general, it can be stated that TS reduces with increasing temperature and holding time. This is due, on the one hand, to the already mentioned degradation of elastically stored energy and, on the other hand, to the degradation of hydroxy groups. Furthermore, the filling with wax after compression and thermal treatment has a slight effect on the swelling behaviour, which can be seen by comparing the two variants MC10T200t24 (without wax) and MC10T-200t24rs (wax impregnated after compression and thermal modification). It is very likely that the hydrophobic wax



Fig. 6 Thickness swelling values with average (red swelling with increase in temperature and dwell time

natural beech wood for various orientations (rxt, lxt, lxr) and different moisture contents (MC) caused by tests on the disc grinding machine

isolates some hydroxy groups in the matrix, preventing them from binding to hydrogen molecules.

#### 3.3 Friction and wear

#### 3.3.1 Disc grinding machine

A selection of the variants investigated in the previous chapter are now examined for friction and wear. Since the material used has orthotropic properties, it is important to first clarify which orientation should be investigated as a matter of preference. For this purpose, abrasive wear tests were carried out on native beech wood with different moisture contents (MC = 0%, 10%, 15%) and orientations. The results of this study are shown in Fig. 7. By comparing the arithmetic mean values, it can be seen that the wear resistance decreases with increasing moisture content. The orientations lxt (tangential section) and lxr (radial section) show similar characteristics in terms of mean value and scatter. The rxt orientation (cross section) clearly has the highest wear resistance among the native beech woods studied.

If the values of the unmodified samples of the rxt orientation are compared with those of compressed samples

(MC10T120), a significantly higher wear resistance can be seen (Fig. 8). When the compressed samples are additionally thermally modified at 200 °C for 24 h (MC10T200t24), a slight increase is observed on average, with the scatter of the individual measured values overlapping with MC10T120. However, compared to the synthetic polymer ultra-high-molecular weight polyethylene (UHMWPE), the wear is approximately four to five times higher.

The results of Figs. 7 and 8 can be justified based on literature studies as follows. The difference between the observed wear values can be explained by the differences in hardness. The carbides of the grinding disc cannot penetrate as deeply into hard materials. Consequently, fewer particles are removed in hard materials than in soft materials. Additionally, the different orientations (Fig. 7) possess varying shear strengths. The lxr and lxt orientations have approximately the same shear strengths (Roš 1925) and show strong overlap in the measured values. Transverse to the fibres (rxt orientation), the shear strength is several times higher (Kollmann 1951). As a result, when an indenter (abrasive grain) penetrates the wood and attempts to remove material tangentially, the increased shear strength results in less wear.

Fig. 8 Wear volume (Wv) of natural and modified beech wood in rxt orientation and ultra-highmolecular weight polyethylene (UHMWPE) by wear tests conducted on a disc grinder. Unmodified MC0%, MC10% and MC15%: Untreated beech wood with a moisture content of 0%, 10% or 15%, MC10T120: Compressed beech wood with a moisture content of MC = 0%. MC10T200t24: Compressed and thermal modified beech wood with a moisture content of MC = 0%

Fig. 9 Specific wear rate (k) versus average coefficient of friction (CoF) derived from five measurements per variant after a sliding distance of 1,000 m. The scatters shown correspond to minima and maxima. #1: MC10T120Rrs, #2: MC10T120bw, #3: MC10T120rs, #4: MC10T120as, #5: MC10T-200t3rs, #6: MC10T200t24rs, #7: Lignum vitae, #8: unfilled and compressed beech wood



#### 3.3.2 Ball-on-disc tribometer

A direct comparison of the specific wear rate versus the average coefficient of friction (hereafter referred to as CoF) of the samples filled with different waxes resulted in a grouping of the results (Fig. 9). The wear values of compressed and wax-impregnated samples (#1: MC10T120Rrs, #2: MC10T120bw, #3: MC10T120rs, #4: MC10T120as, #5: MC10T200t3rs) exhibit the lowest friction and wear values. This indicates that the type of wood or the type of wax does not seem to have a major influence on the result, with the variant MC10T120Rrs (#1) showing slightly higher wear and the MC10T120as (#4) variant (stearic acid) exhibiting slightly higher friction (CoF=0.09 to 0.12). Lignum vitae (#7), which is naturally filled with resin, is characterised by slightly higher friction and wear than the compressed and wax-impregnated samples. Maximum wear values are exhibited by MC10T200t24rs (#6). Significantly higher CoF values, ranging from 0.29 to 0.32, were measured for unfilled and compressed beech wood (#8).

Figure 10 shows the average CoF over a distance of 1,000 m for selected samples. The running-in behaviour,

judged by a strong change in CoF as a function of the sliding distance, is clearly visible for some samples. At the end of the running-in stage, steady state friction sets in (Blau 2006). Strictly speaking, this state is characterised by a more or less constant CoF as a function of sliding distance. However, since sliding friction is a dynamic contact situation, slight variations in CoF with sliding distance may occur in this state, where the CoF increases or decreases slightly and slowly with sliding distance (Straffelini et al. 2016). It is possible that a tribological system will never reach this stage. This is the case for the unfilled, compressed beech (#8), which exhibits a sharp increase in CoF up to 100 m and sometimes shows an unsteady course. The CoF increases steadily up to the end of the measurement never reaching steady state friction. The situation is completely different for the lubricated samples. These show varying running-in stages followed by steady state friction. In this category, naturally grown lignum vitae (#7) has a relatively pronounced running-in behaviour and a relatively high CoF. A long running-in time is also observed for the MC10T120as variant (#4). The MC10T200t24rs (#6) sample, on the other hand, is characterised by a small and continuous increase

700

-MC10T120as (#4)

--- MC10T200t3rs (#5)

800

900

600

(#8)

(#7)

(#4)

(#6) (#3)

(#5)

1,000

2164





Fig. 11 Optical microscopy of wear marks on selected samples that were not cleaned at the end of the friction and wear measurement. The respective friction partners are arranged in columns (Above: wood sample, below: steel ball)

in the CoF up to the end of the experiment. The samples MC10T120rs (#3) and MC10T200t3rs (#5) do not exhibit any pronounced running-in behaviour and show stable steady state friction curves over 1,000 m. For these samples, even at longer measuring distances of up to 10,000 m (not shown), a low CoF was measured, indicating that there was no reduction in the lubricating effect of the rapeseed wax.

Figure 11 shows selected samples and their wear characteristics. The samples of the unfilled variant MC10T120 have powdery wear particle adhesions on both sides, which take on a brown to black charred colour as a result of thermal input. According to Kollmann (1951) wood undergoes

"self-carbonisation" at a temperature of 270 °C. Erchinger (2009) defined a range from 250 to 350 °C.

The nature and shape of the wear characteristics of the selected samples were examined by optical microscopy (Fig. 11). The MC10T200t24rs variant exhibits by far the highest level of wear, characterised by almost no particles on the wear track, with clear cracks appearing on the running track, mainly along the medullary rays. The steel ball shows a mixture of finely ground base substrate and wax, which adheres well to the surface.

Low wear is observed for the variants MC10T120rs (not shown, almost identical wear mark compared to



MC10T200t3rs, wood colour: Fig. 5) and MC10T200t3rs (Fig. 11). There are almost no wear particles on and in the running track. A homogeneous and transparent transfer film can only be observed on the steel ball, indicating the presence of a small amount of base substrate.

Powdered wear particles can also be assigned to the samples of the modified Robinia (MC10T120Rrs). A deposit is clearly visible, particularly in the pore-rich early wood regions and on the steel ball. Optically, it appears to consist of predominantly light-coloured rapeseed wax. Compared to MC10T200t3rs, the wax adhering to the counter body is not melted and is significantly more pronounced, indicating that the lubricant was released too early. Slightly higher wear values for the variant MC10T120Rrs are attributed to the higher degree of wear in the early wood areas (Fig. 11). Compared to the modified beech wood samples, these are more pronounced, resulting in a more inhomogeneous structure of the contact surface. The lower density and hardness in these areas enables the counter body to penetrate deeper into the material, resulting in increased lubricant (wax) release.

The wear track (Fig. 12 – (a and b)) shows some scratches in the feed direction, which are lost in the noise of the surface finish. Straight-line scratches that do not run parallel to this can be attributed to mechanical processing. The curved lines visible in the track initially give the impression that defects in the surface structure have occurred. After conditioning (@ RH=98%) of the tribologically tested specimens, these features opened up from an oval to a circular shape with a diameter of approximately 0.1 mm. Tribologically unstressed areas also exhibit these features. Furthermore, the number and expression in the early wood are significantly higher, supporting the assumption that these are vessels of beech wood. Considering that the degree of wear (Fig. 12 –(c)) and a CoF of around 0.08 do not deviate significantly from each other (comparison of 1 km and 10 km measurements), the tribological system is stable and intact even after a sliding distance of 10,000 m.

This study shows that three factors influence the wear of modified wood: Firstly, the modification step PS 4 compression, which causes the radial densification of the samples leads to a homogenisation of the density across the cross section, resulting in a uniform degree of wear for beech wood. Secondly, the duration of the thermal treatment plays an important role. Thermal treatment for extended durations, such as the MC10T200t24rs variant, showed even higher wear than unfilled compressed beech wood. This increased wear and the clear formation of cracks indicate a reduction in strength and embrittlement of the material due to the thermal treatment. Experiments carried out on a hardness testing machine (test values: HBW 10/250/30) showed that the compressed and thermally modified specimens ( $T = 200 \text{ }^{\circ}\text{C}$ , t=4 h) developed cracks from the indentation point. However, the samples heated to 200 °C for 3 h (MC10T200t3rs) show significantly less wear and no obvious material defects (for the wax filled variants). Finally, wax impregnation of the compressed samples has a significant impact on wear. Due to the diffuse porous structure of wood, it is hypothesised that the lubricant is evenly distributed throughout the material, ensuring a continuous and steady release. The presence of almost no wear particles on the wood samples and the transparent transfer film adhered to the steel ball suggest that wear is predominantly due to plastic-elastic deformation. The lowest friction and wear can be assigned to the variants MC10T120rs (#3), MC10T120bw (#2) and the thermally modified variant MC10T200t3rs (#5). A comparison between the variants MC10T120rs and MC10T-200t3rs shows no differences in the form and characteristics of the observed wear.

An important overall result of the tribological tests in this work is that all thermally modified woods were sufficiently filled with wax by the PS 8 - impregnation step to achieve approximately similar reductions in friction and wear. Furthermore, the friction and wear values of the samples impregnated with different waxes show a strong overlap, which can be attributed to the very similar chemical composition (mainly palmitic and stearic acid). Despite having the highest melting point (mp = 57 –61 °C, see enclosed product data sheet) and the highest friction of the filled samples. An important finding is that beeswax can be replaced by the much cheaper and readily available rapeseed wax.

Bearings made from modified and wax impregnated wood are currently undergoing tribological testing to determine whether the modification process developed in this paper provides sufficient dimensional stability under variable relative humidity and test loads.

# 4 Conclusion

This study develops an alternative method for the preparation of wax impregnated modified wood based on beech (*Fagus sylvatica*) and robinia (*Robinia pseudoacacia*). This method makes it possible to bond modified woods prior to wax impregnation, thereby enabling scalability. Furthermore, relevant effects such as SBE and thermal swelling are taken into account. The main contributions of this investigation are listed below:

- The alternative method restructures the already published modification sequence. Plasticisation and compression followed by thermal treatment were carried out at the beginning and wax impregnation using rapeseed wax, stearic acid or beeswax was performed at the end of the sequence.
- The process parameters of plasticisation and compression in a mould were set with regard to SBE and

structural defects. Samples with an initial MC = 10% exhibited intact wood structures. Samples with a higher initial moisture content were characterised by larger structural defects and were, therefore, disqualified for tribological applications. A further positive influence on the SBE was achieved by relieving the press mould from the compression load, but only opening it after the visible water vapour had escaped. Parts produced in this way can be further modified inside or outside the mould.

- Samples were subjected to thermal treatment at 200 °C for up to 24 h to reduce the swelling of the wood. In this manner, thickness swelling was reduced by up to 85%. Subsequent wax impregnation after thermal treatment has an additional positive effect.
- With regard to abrasive wear resistance, the three main orientations of the wood were examined on a disc grinding machine. For untreated beech, the rxt orientation exhibited the highest wear resistance, which could be further doubled by compression. Compressed and thermally modified samples showed a strong overlap with the only compressed variant.
- Friction and wear tests were carried out on a ball-on-disc tribometer. It was found that different types of waxes resulted in similar coefficients of friction (approximately CoF=0.08–0.1), with animal stearic acid showing the highest values in terms of the shape and characteristics of the friction curves. Notably, the readily available and cheap rapeseed wax can replace the much more expensive beeswax. Wood samples that were additionally thermally modified at 200 °C for 24 h showed low coefficients of friction, but also an increase in wear with clear cracks in the wear track. Beech wood compressed by up to 40%, thermally modified at 200 °C for 3 h and impregnated with rapeseed wax (MC10T2003rs) did not show these defects and exhibited the lowest friction and the lowest wear of all the samples tested.

Ongoing investigations are underway to determine whether the modification parameters developed provide sufficient dimensional stability under varying relative humidity and tribological test loads.

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**Data availability** All data generated or analysed as part of this study are stored on the university's servers and are available onrequest from the corresponding author.

#### Declarations

Competing interests The authors declare no competing interests.

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