

## Bending creep of Maritime pine wood (*Pinus pinaster* Ait.) chemically modified

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### ABSTRACT

The long-term performance of a structural member is determined by its durability and deformation with time. The bending creep behaviour of modified wood was assessed experimentally over a period of 35 days (840 hours).

Four chemical modification processes were used: 1,3-dimethylol-4,5-dihydroxyethyleneurea (DMDHEU), m-methylated melamine formaldehyde resin (MMF), tetraethoxysilane (TEOS) and amid wax (WA). Wood stakes with 20·10·200 mm RTL dimensions of Portuguese Maritime pine (*Pinus pinaster* Ait.) from sapwood part of the stem were used for evaluated the primary creep. Experiments were conducted at bending stresses amounting to 0.1, 0.2, 0.4 of the mean immediate wood bending strength obtained at equilibrium moisture content (EMC). Applying the same stress level (SL, 0.2), wood creep was also determined at the constant low and high moisture content.

As results: Between low and medium SL (8 and 16 N/mm<sup>2</sup>), unmodified wood at indoors conditions did not show any effect in the creep factors ( $k_c$ ). However, at high SL (35 N/mm<sup>2</sup>) a slight increase (not significant) in the  $k_c$  was found. It seems that the  $k_c$  was nearly independent of the SL.

In the lumen fill modification (TEOS and wax), the deposited material has not affected the creep behaviour under various SL.

The cell wall modification (with DMDHEU and MMF resins) did not show any differences in the  $k_c$  for low and medium SL (8 and 16 N/mm<sup>2</sup>). However, resin modification under high SL (35 N/mm<sup>2</sup>) has shown a significant reduction related to unmodified wood. Between both types of resin (DMDHEU and MMF) and levels of modification (WPG), significant effect was not found.

At saturated conditions, lumen fill modification (TEOS and wax) did not show any effect on creep. In the cell wall modification (DMDHEU and MMF resin), significant reduction was recorded due to the embrittlement effect imparted by the modification (deposit of resin in the cell wall).

### KEY WORDS

chemical modification, creep factor, indoor climate, saturated conditions

## INTRODUCTION

All materials experience a significant increase of deflection when loaded over a period, even at indoor climate. In the full-scale wood structures, the creep phenomenon occurs at stress level (SL) of 1.5 up to 2.1 N/mm<sup>2</sup>, according to EC 5 code (ENV 1995:1 suggestion at service limit state, SLS). Therefore, a creep factor  $k_c$  of 0.60 is suggested in a lifetime of 50 years.

In laboratory conditions, SL of 10 N/mm<sup>2</sup> was the most used. The latter SL is the most close to the service limit state (SLS) of EC 5 and simultaneously can produce displacements to be captured by sensitive measurement instruments, linear variable displacement transducers LVDT's. However, in many applications of wood, higher SL can be installed. Occasional stock loads at horizontal members and most of times in the wood columns. The latter's are never perfectly straight and when axial loaded, the creep phenomenon can arouse due to the secondary bending moment from the slight column curvature, known as *P $\delta$  effect*.

The visco-elastic properties of wood have been an interesting topic for scientific investigations in the past (Buffon 1740 in Clouser 1959; Schniewind 1968; Armstrong 1972; Bach 1973; Grossmann 1976; Boyd et al. 1982; Bodig and Jayne 1982; Dinwoodie et al. 1991; Liu 1994; Ranta-Maunus and Korttesmaa 2000). One of the major problems in this field has been the lack of systematic experimental data and the role of natural wood defects into it. Regardless the moisture change through the wood (mechano-sorptive effect, MSE), the creep behaviour at constant conditions is a challenge for render the modified wood into material for structural purposes. In the chemically modified wood, the bending stiffness did not change significantly, at one hand (Krause 2006; Bollmus 2011; Lopes et al. 2013b). On the other hand, the equilibrium moisture content (EMC) decreased in-line with the improvement of the anti-swelling efficiency (ASE) and stiffness stabilization efficiency (SSE). In the latter, different extend changes in the modified wood can take place (Militz 1993; Krause 2006; Bollmus 2011; Pfeffer 2011; Lopes et al. 2013a; Lopes et al. 2013b).

Under cyclic moisture conditions (in the MSE of creep wood) the resin based modified wood performed best than unmodified wood or lumen fill modified specimens (Norimoto et al. 1992; Epmeier et al. 2007a; Lopes et al. 2013c). Low EMC and "good" relations moisture,

with low water movements through the wood, were the explanation for the best performances in the MSE. However, Epmeier et al. (2007a) with MMF treatments (with a slight increase in the EMC compared to unmodified wood) have shown a good performance of modified wood in the MSE creep behaviour. The creep factor decreased up to 50%, i.e. the EMC has not the main role on the creep phenomenon, but also the embrittlement effect imparted by the (deposit of) resin in the cell walls.

Caused by the resin deposit and reaction in the cell walls, the embrittlement effect on the creep behaviour has not been assessed so far, by itself as an isolated variable under constant conditions, indoor climate with RH of 65%, saturated conditions and as well under various stress levels. The comparative study under saturated conditions will answer at some issues: Practical applications (roofs leading to seepage on rafter framing, e.g.); isolating the variable of embrittlement (deposit of resin in the cell walls) where the wood MC can be neglected and assess the effect of the enhanced hardness/compression strength in the creep behaviour. Therefore, the study of the deposited materials in the lumen (TEOS and wax) was also the aims of this work.

This study is limited at one single species (pure sapwood of Portuguese Maritime pine), four modification methods (DMDHEU, MMF, TEOS and wax) and analyzes of creep in bending under constant conditions. The wood-moisture interaction plays a decisive role on the creep behaviour and durability of a wood component. It was not seen in this study because it will be covered in a separate approach.

## MATERIALS

The wood material was flawless Maritime pine (*Pinus pinaster* Ait.) from pure sapwood part of the stem with 2–3 mm annual ring width and a mean dry density of 505 kg/m<sup>3</sup>. Stakes with 20·10·200 mm RTL were used. All specimens were conditioned up to constant weight at a relative humidity (RH) of 55 ± 5% and 20 ± 2°C of temperature. Ten specimens per modification and SL were included, resulting in a total of 239 specimens, i.e. 55 controls and 184 modified specimens.

The modification methods investigated were 1,3-dimethylol-4,5-dihydroxyethyleneurea (DMDHEU), N-methylol melamine (MMF), tetra-alkoxysilane

**Tab. 1.** Number of specimens per modification and abbreviations

Abbreviations	0	D1	D2	i1	i2	T	WA		
Chemical	–	DMDHEU		MMF		TEOS	Wax		
Concentration	ctrl	medium	high	medium	high	high	high		
Density and WPG	20	20	20	20	20	20	20	tab. 2	
MOE and MOR	10	10	10	10	10	10	10	fig. 2A, B	
Stress Level – SL	8 N/mm <sup>2</sup>	10	10	10	8	–	10	10	fig. 4A
	16 N/mm <sup>2</sup>	10	–	10	–	–	–	–	fig. 3A, B
	35 N/mm <sup>2</sup>	12	10	11	9	10	10	12	fig. 4B
$f_{c,o,saturated}$	16 N/mm <sup>2</sup>	13	10	10	8	12	8	6	fig. 5A
Full Sat.	16 N/mm <sup>2</sup>	10	10	–	–	–	–	–	fig. 5B

(TEOS) and amid wax (WA). Additional information about treatments can be found in different works, Militz (1993); Epmeier et al. (2004); Donath et al. (2004) and Scholz et al. (2009), respectively. Lopes et al. (2014) also presented a summary description.

**METHODS**

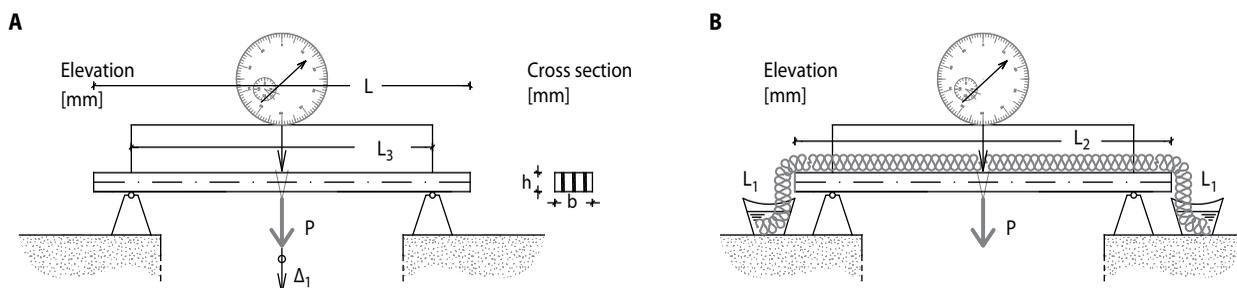
The testing facilities has included one room with 20 m<sup>2</sup> able to hold up to 20 individual creep specimens and providing controlled and monitored environment conditions with RH between 55 to 65% and temperature 20±2°C. The standard creep tests were made at three points bending (3pb) under constant load with 160 mm of span, see fig. 1A.

The standard annual rings (parallel to its faces) were placed perpendicular to the supports. Specimens were loaded on the radial surface, at the horizontal position, in 4mm width of the steel-ring. Care was taken

to minimize the impact loading caused by the weights application. The loads were gently applied at approximately 20 seconds.

The weight load was selected in such way to ensure the bending stresses of: 0.1, 0.2 up to 0.4 of the mean modulus of rupture (MOR) determined with EMC at 65% of RH. Three bending stress levels (SL) were used (normal or lower, medium and high, respectively 8, 16 and 35 N/mm<sup>2</sup>).

To assess the creep in saturated conditions, two distinct experiments were performed. In the former, moistening was in the upper side of the specimen in the compression zone, see fig. 1B. A strip of filter paper, with 20 mm of width, was extended throughout the length development of the specimen, plus 15 mm, and its ends dipped in two containers (app. 20 ml). To keep the water saturation by capillarity, without needed to clean the impurities in the filter paper, distilled water was used. The evaporation losses were compensated by the water replacement every two days. The end tops of the speci-



**Fig. 1.** Load arrangement for the creep tests in bending of 20.10.200 mm RTL specimens: A – indoor conditions, B – saturated conditions

mens were sealed with paint to avoid moisture absorption longitudinally to the grain and then one directional moisture movement was achieved (tangentially). Specimens were saturated forty-eight hours before the loading start up to the end of the test.

In the second moistening setup, specimens were saturated with tap water and wrapped with plastic film to avoid any exchange with outside environment moisture. Therefore, high MC was kept constant over the running tests.

MOR and static  $MOE_{st}$  in bending were performed according to DIN 52186 standard with 1.5 mm/min speed test on a Zwick/Roell ZMART.PRO 10 kN universal test machine (Ulm, Germany).

$MOE_{dyn}$  tests were carried out using the Grindosonic MK5 (J.W. Lemmens N.V.; Leuven; Belgium). More information about this procedure can be consulted in Hearmon (1966) and Machek et al. (2003).

Data taker DT 515 series 3, with 9 Digital Channels with 0.3 hz of acquisition rates, coupling with 8 linear variable displacement transducers LVDT's with 0.001 mm precision and a thermo-hygrometer (CPC 1/4-TH no 67226) fed with  $\pm 24$  tension volts were used over a period of 200,000 hours. LVDT's gauges measured the curvature, between the load supports.

Dates of temperature, RH and displacements were recorded with a PC-based data logging procedure automatically at 5 s time of intervals (for the first 4 hours) and at each 30 minutes (next days) until the end of test was reached.

Deflection was measured at mid-span from the top (compression side) of the specimen, according to the fig. 1A. From this curvature, static MOE with three points bending (referred to hereinafter as  $MOE_{st,3pb}$ ) was calculated 60seconds after loading.

The creep compliance was recorded from the total deflection ( $\Delta_t$ ). Therefore, relative creep ( $\phi_t$ ) and creep factors were calculated. The creep factor ( $k_c$ ) was useful for designing purposes to predict the long-term deflection ( $u_{fin}$ ) from the elastic deflection ( $u_{inst}$ ). The  $k_c$  is the increase of deflection with time  $d_c$  divided by the initial elastic deflection, after 60 s ( $\delta_0$ ). Where  $d_t$  is the deflection and  $f_t$  is the relative creep at time t. The relative creep ( $\phi_t$ ) is the deflection at a specific time divided by the deflection after 60 s.

$$u_{fin} = u_{inst} \times (1 + k_c) \quad (1)$$

$$k_c = (\delta_t - \delta_0) / \delta_0 = \delta_c / \delta_0 = -\delta_t / \delta_0 - 1 = \phi_t - 1 \quad (2)$$

To present the relative creep, the power function model (PFM, see Eq. 3), with three creep coefficients, was used. Where t is the time elapsed and  $\beta_0$  represents the elastic deformation and can be calculate from the MOE, the size of the specimens for any given load, according to the material behaviour ( $\Delta_1$ , fig. 1A). The coefficient  $b_0$  is a constant that describes the displacement along the z-axis in a positive direction (EC 5). For the measured relative creep,  $\beta_0$  is always 1 (one) according to the definition of relative creep, i.e. the visco-elastic creep started exactly when the initial deflection was measured (after 60 s).

$$\Phi_t = \beta_0 + \beta_1 \cdot t^{\beta_2} \quad (3)$$

The second term  $\beta_1 \times t^{\beta_2}$  is the creep deflection. Where the coefficient  $\beta_1$  describes the slope of the relative creep curve and  $\beta_2$  is the power factor that modifies the influence of time. This second term grows with time in approaching asymptotic limit.

All variables were characterized by the mean value ( $\bar{x}$ ), the median (horizontal line in the box plot) and standard deviation (box) for un- and modified wood. The whisker plots displayed the 5 and 95% percentile range (see fig. 2, e.g.). For comparative purposes, a t-test analysis was carried out to verify whether a change was significant or not. The level for significance was set at  $p < 0.05$ .

To fitting creep curves and forecasting data, the ORIGIN<sup>®</sup> statistical software was used. For unconstrained models, the default algorithm used by ORIGIN<sup>®</sup> is the Gauss-Newton method of finding the minimum of function that is a sum of squares of non-linear functions.

## RESULTS

Table 2 shows the density of wood specimens for un- and modified wood in equilibrium conditions, 65% of RH. The deposit of chemical is the main reason for the increase density. Two types of chemical modified wood were used: cell wall reaction or resin based with 1,3-dimethylol-4,5-dihydroxyethyleneurea (medium and high concentration, assigned D1 and D2 respectively) and m-methylated melamine formaldehyde resin MMF

(i1, i2); Lumen fill with tetraethoxysilane (T) and amid wax (WA).

The concentration of solution correlate with weight percent gain (WPG) acquired by the wood material. Before all impregnations, wood material was dried over 24 h at 103°C ( $W_{unmodified}$ ). After impregnations and curing, wood material was dried to access the dry weight ( $W_{modified}$ ). The weight percent gain was obtained by the difference between both weight measurements for each type of modification methods. Standard WPG were found.

**Tab. 2.** Density at indoor climate and WPG (n = 20)

Ctrl and modification		0	D1	D2	i1	i2	T	WA
Density	kg/m <sup>3</sup>	556	652	770	646	765	741	1058
	stdv	23	29	16	28	15	19	17
	CV	4	4	2	4	2	3	2
WPG	%	–	16.2	34.3	16.0	35.5	29.7	102.1
	stdv	–	1.5	3.4	1.7	2.5	3.7	10.3
	CV	–	9	10	11	7	12	10

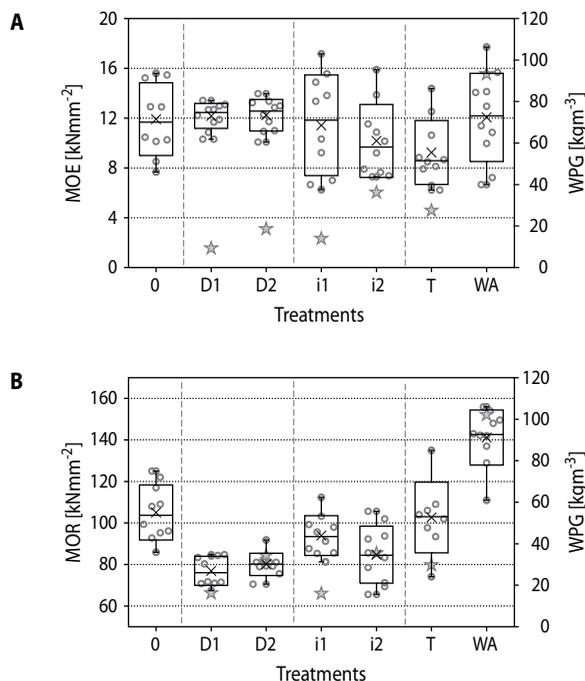
For abbreviations, see tab. 1

All treatments increased the density significantly, which was expected, according to Militz (1993); Epmeier et al. (2004); Donath et al. (2004) and Scholz et al. (2009), respectively for different chemicals. The increasing density does not lead directly to a significant effect in the stiffness material or in the creep behaviour. Two effects are involved with offsetting: at one hand, the cross section inertia was increased by the bulking induced by the modification (Krause 2006; Bolmus 2011); at the same time, chemical deposit (in the cell walls and/or in the lumen) lead to higher mass. Both lead to obtain similar eigen-frequency of the material (stiffness).

Figure 2 shows the MOR and MOE in bending for un- and modified pinewood in stakes with 20 × 10 × 200 mm RTL. Star with five nozzles shows the WPG imparted by the modification. DMDHEU resin has shown a MOR reduction up to 25% regardless the level of modification and MMF resin with high concentration showed up to 20% of reduction in MOR. For low concentration of MMF and TEOS, the average of MOR did not change significantly but specimens with amid wax showed an increasing MOR up to 30%.

Stiffness (MOE) did not show any significant effect regardless the type of modification (cell wall reaction

or with lumen fill principle) and concentration levels of solution used.

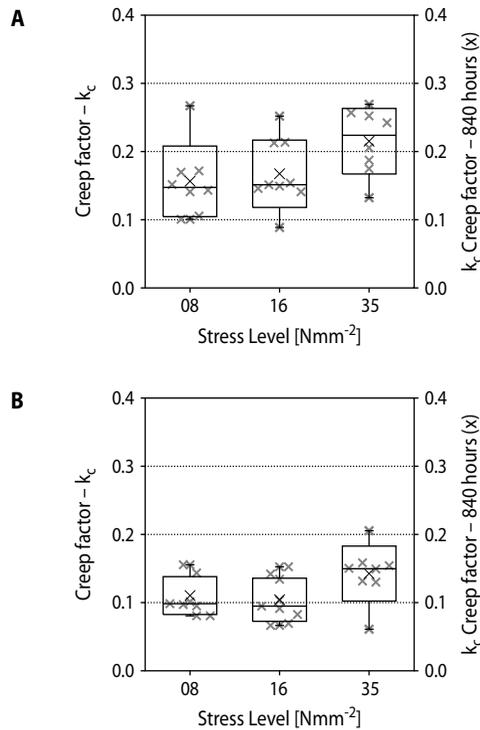


**Fig. 2.** MOE (A) and MOR (B) in 3 pb bending for un- and modified pinewood (WPG, ☆)

For abbreviations, see tab. 1.

Figure 3 shows the effect of SL on the creep compliance over a period of 35 days (840 hours). In three SL, only control and modified wood DMDHEU D2, 2.3M were used. Because D2 had highest reduction on strength, MOR. Under low and medium SL, the unmodified pinewood did not show any difference of the  $k_c$ , fig. 3A. However, under high SL the mean value of the  $k_c$  was increased, not significantly. DMDHEU resin showed lower and more homogeneous  $k_c$  with significant difference under high SL (35 N/mm<sup>2</sup>).

Figure 4 shows the effect of modification under SL on the creep compliance over a period of 35 days (840 hours). Low SL (8 N/mm<sup>2</sup>, fig. 4A) did not show any difference on the  $k_c$  between any type of modification (cell wall or lumen fill modification). However, under high SL (35 N/mm<sup>2</sup>) the resin-based modification showed significant lower  $k_c$ . Both cell wall modification (with DMDHEU and MMF resins) have shown similar behaviour at indoor conditions, with a reduction up to 50% compared to control.

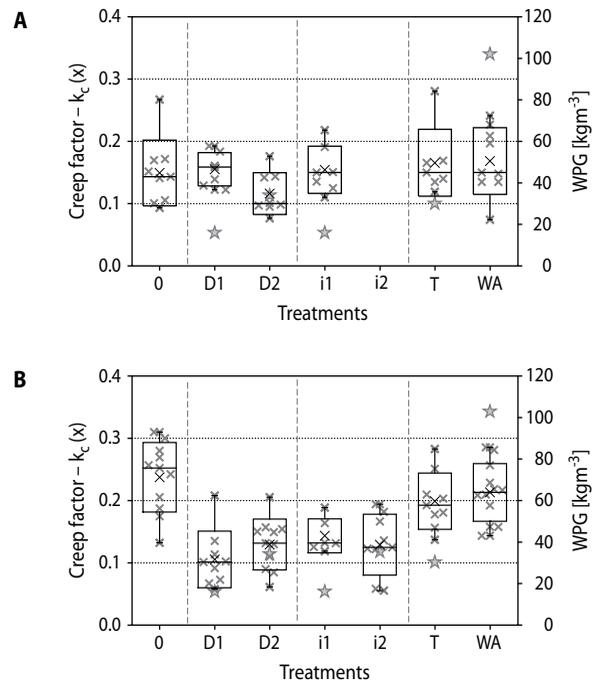


**Fig. 3.** Compliance of creep factor ( $k_c$ ) under three stress levels for: A – unmodified pine and B – modified wood with high concentration of DMDHEU resin, D2

Figure 5A shows the relative creep ( $\phi_t$ ) of small beams in three points bending and saturated compression zone over all creep test 35 days (840 h) according to fig. 1B. Specimens with lumen fill (TEOS and wax) showed no effect on the creep performance with compression zone saturated.

The cell wall modified specimens, with DMDHEU and MMF resins, have shown a significant reduction of the  $\phi_t$ . Under wet conditions, reduction of the  $\phi_t$  was roughly similar to the retention in the impact bending strength (IBS) experienced by both treatments: 60% for DMDHEU and 30% for MMF resin (Lopes et al. 2013b).

Figure 5B shows the creep behaviour under full-saturated conditions. The average of MC in the full-saturated specimens was higher than the fibre saturation point (FSP), with 60% and 48% for control and DMDHEU resin (D1, 1.3M), respectively. The performances between both test specimens, 0 and D1, were assumed to be alone resultant of the embrittlement effect caused by chemical modification. Above the FSP no effect of MC was involved.



**Fig. 4.** Creep factors for: A – low SL 8 N/mm<sup>2</sup>, and B – high SL 35 N/mm<sup>2</sup>. Weight percent gain (WPG, ☆)

For abbreviations, see tab. 1.

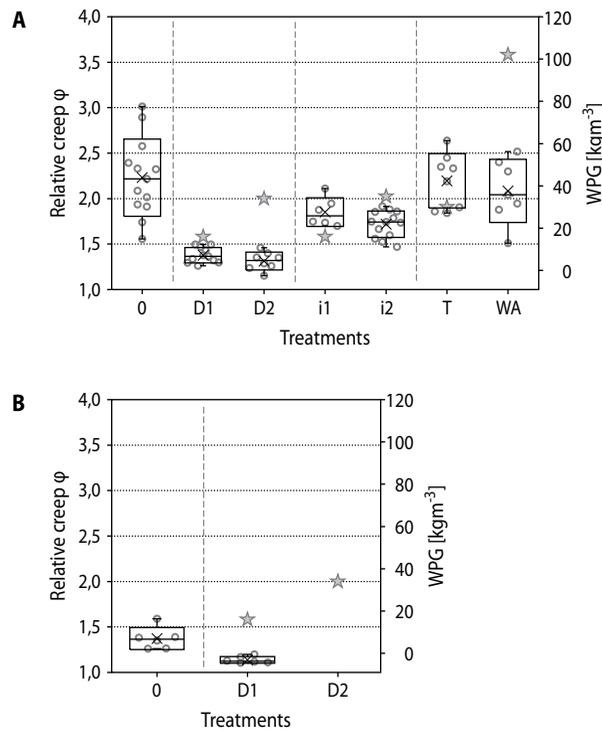
Both setups, saturated compression zone in fig. 5A or full saturated conditions in fig. 5B, led to a similar conclusion regarding the  $\phi_t$  and IBS behaviour. The association between creep behaviour and the IBS was made for two reasons:

- The water saturation increases the MC of the wood material and isolates the effect incorporated by modification, the embrittlement effect.
- The IBS is the most used variable to describe the embrittlement effect imparted by the modification in the cell wall modification (DMDHEU and MMF resin).

For cell wall modification, creep and IBS reductions were similar. In other way around, despite the positive effect in the IBS (Lopes et al. 2013b), lumen fill modification with wax did not reveal any effect in the saturated bending creep, fig. 5A. For this distinct and contradictory behaviour between modification types, no correlation of creep behaviour and IBS was presented.

The remaining treatments (MMF, TEOS and wax) were not tested in full-saturated conditions, in fig. 5B. The similarity of results presented in fig. 5A and b of the

tested specimens (0-ctrl and D1); And the unaffected creep behaviour by specimens with lumen fill (TEOS and wax, WA and T in fig. 5A); As well as expressed by unchanged physical properties, e.g., the EMC and ASE in Donath et al. (2004) and in Lopes et al. (2013a), were the main reasons.



**Fig. 5.** Relative creep: A – with saturated compression zone (see fig. 1b), and B – full saturated, for un- and modified wood (WPG, ☆) under SL 16 N/mm<sup>2</sup>

For abbreviations see tab. 1.

## DISCUSSION

For unmodified wood, the minor effect of the MC and SL in the creep under constant conditions had been presented before: Clouser (1959) conducted a study with small beams of Douglas fir species with 6 and 12% of MC under very high SL, greater than 60 up to 95% of MOR. He has shown that the time to failure increased as the SL was decreased and a slight (but not significant) effect of MC on creep was shown at indoor climate, 60% of RH. Dinwoodie et al. (1990) has shown a slight effect of the SL on the relative creep  $\phi_t$  with red wood, between 30 up to 50% of MOR.

When this work started, the assumption was that modification would change the bending creep behaviour: In the cell wall modification (DMDHEU and MMF resins) the imparted embrittlement effect presented by the IBS (Krause 2006; Mai et al. 2007; Bollmus 2011; Lopes et al. 2013a); And dimensional stabilization presented by ASE as well as the reduction in the EMC (in the same works) or SSE in Lopes et al (2013b).

Despite cell wall modification reduced the EMC (or showed high dimension stability), all creep results were not different under low and medium SL (8 and 16 N/mm<sup>2</sup>) at indoor conditions (RH of 65%). Under high SL (35 N/mm<sup>2</sup>), both cell wall modification (with DMDHEU and MMF resin) have shown less creep (app. 50%), with a reduction close to the SSE. However, despite these resin based specimens showed lower EMC, the embrittlement effect was involved as well. To neglect the effect of different EMC presented by un- and modified wood and isolate the effect of resin deposit, specimens in full saturated condition were tested. Specimens modified in cell wall (with DMDHEU and MMF resin) have shown less creep with different extend, 50% and 25%, respectively. At one hand, wood MC and its changes ( $\Delta MC$ , app. 50%) were assumed to be a parameters influencing the creep behaviour with no significant role under very high SL (Clouser 1959). In this experiment with modified wood (cell wall reaction, DMDHEU and MMF resins) two effects were mixed: the EMC reduction and resin-deposit in the cell wall (imparted the embrittlement effect, according to Rowell 1996; Mai et al. 2007; Xie et al. 2007). The latter acts as holder/clamp of fibres. Therefore, lower EMC and smaller  $\Delta MC$  imparted by modification, such as presented by resin-based modification (DMDHEU and MMF resins), only displayed reduced primary creep behaviour in high SL (35 N/mm<sup>2</sup>), app. 50%.

On the other hand, saturated condition has shown the effect of deposit of resin imparted by the modification, as isolated variable. The MC above the FSP led to neglect the effect of MC in un- and modified wood with resin-based.

For comparative purposes, the  $k_c$  reduction could be associated at the similar decreasing in the EMC experienced by modified specimens with both types of resin (DMDHEU and MMF) for wet conditions, in 87% of RH (Lopes et al. 2013a). However, Epmeier et al. (2007a) and (2007b) with similar modified wood (furfu-

rylation and MMF resins) not provided any correlation between their own results of creep and EMC variation. MMF modification has shown similar MSE reduction in Epmeier et al. (2007b) as well as bending creep under saturated compression in this study.

Lumen fill modification (TEOS and wax) have not shown any effect in the relative creep  $\phi_t$  at indoor climate or saturated conditions. These results have come in line with unaffected physical properties (EMC and SSE, e.g.) as well as any negative effect in the mechanical properties (IBS) showed by specimens with wax and TEOS.

Table 3 shows the mean creep factor accessed experimentally under SL (8, 16 and 35 N/mm<sup>2</sup>) over a period of 840 h (35 days) with non-significant values in brackets and significant values *italicized*. For both types of modification (cell wall reaction or lumen fill), under low SL no significant effect was found, regardless the WPG imparted by the modification. Concentration solution of cell wall modification (DMDHEU resin) in the medium SL were shown similar results to unmodified wood.

Resin-based modified specimens with various concentrations of DMDHEU and high concentration of MMF, at high SL have shown significant less  $k_c$  than

unmodified specimens had. The embrittlement effect, imparted by the resin deposit in the cell wall, is the main explanation by the significant reduction in the long-term creep behaviour. At each set of wood specimens, un- or modified and for different SL, no significant effect were found, see the last two lines in the tab. 3.

### Correlation analyses

Often density is regard as being closely relate to the resistance or deformation. Table 4 shows the main correlations between density, stiffness and deformations in the creep tests under different SL (8, 16, 35 N/mm<sup>2</sup>). Uneven of pattern correlations of each modification are not significantly different.

As far as unmodified wood were concerned, moderate correlations were found, not significant. In modified specimens, the correlations were not consistent and lower in the most cases.

As a summary, the prediction of performance criteria based exclusively on density should not be formulated for modified wood. Although, with vibration technique ( $MOE_{dyn}$ ), un- and modified with wax and DMDHEU resin have showed significant correlations.

**Tab. 3.** Results of creep tests at indoor climate (65% of RH),  $k_c$  under 8, 16 and 35 N/mm<sup>2</sup> SL over 35 days by un- and modified pine. Mean, standard deviation, coefficient of variation and p-value were presented

Stress Level		0	D1	D2	i1	i2	T	WA
SL 8 N/mm <sup>2</sup>	$k_c$	0.15	0.16	0.11	0.19	–	0.18	0.18
	stdv	0.05	0.03	0.03	0.03	–	0.07	0.06
	cv	35	17	23	18	–	37	34
	p-value	–	[0.61]	[0.21]	[0.78]	–	[0.84]	[0.67]
SL 16 N/mm <sup>2</sup>	$k_c$	0.14	0.12	0.11	–	–	–	–
	stdv	0.02	0.05	0.04	–	–	–	–
	cv	18	43	33	–	–	–	–
	p-value	–	[0.37]	[0.08]	–	–	–	–
SL 35 N/mm <sup>2</sup>	$k_c$	0.22	0.11	0.14	0.14	0.13	0.18	0.21
	stdv	0.05	0.05	0.04	0.03	0.05	0.03	0.05
	cv	22	43	28	19	38	14	21
	p-value	–	0.00	0.02	[0.07]	0.01	[0.24]	[0.79]
p-value <sup>a</sup>	SL 8 N/mm <sup>2</sup>	–	–	–	–	–	–	–
	SL 16 N/mm <sup>2</sup>	[0.27]	[0.24]	[0.11]	–	–	–	–
	SL 35 N/mm <sup>2</sup>	[0.10]	[0.12]	[0.11]	[0.28]	–	[0.96]	[0.58]

<sup>a</sup> Correlation between each treatment, but at different stress levels.

For abbreviations, see tab. 1.

**Tab. 4.** Coefficients of correlation for modified specimens and pine control at each stress level and different variable (n=10), [non-significant values] in brackets

ctrl / Modification			0	D1	D2	i1	i2	T	WA
SL – 8 N/mm <sup>2</sup>									
Creep deflection	vs.	MOE <sub>dyn</sub>	[–0.34]	–0.89	[–0.46]	–0.80	–0.63	–0.56	–0.72
Relative creep	vs.	MOE <sub>dyn</sub>	[–0.13]	[–0.03]	[–0.27]	–0.67	–0.59	[–0.44]	–0.62
SL – 16 N/mm <sup>2</sup>									
Creep deflection	vs.	MOE <sub>dyn</sub>	[–0.06]	[–0.51]	[–0.03]	–	–	–	–
Relative creep	vs.	MOE <sub>dyn</sub>	[–0.32]	[–0.40]	[–0.41]	–	–	–	–
SL – 35 N/mm <sup>2</sup>									
Creep deflection	vs.	MOE <sub>st,3pb</sub> <sup>a</sup>	[–0.37]	–0.80	[–0.50]	[–0.51]	[–0.05]	–0.67	–0.78
Relative creep	vs.	MOE <sub>st,3pb</sub>	[–0.43]	[–0.31]	[–0.10]	[–0.09]	[–0.27]	[–0.35]	–0.68
All SL [ 8, 16, 35 N/mm <sup>2</sup> ]									
Density	vs.	MOE <sub>st,3pb</sub>	[+0.49]	[+0.34]	[+0.31]	[+0.53]	[+0.06]	[+0.13]	[+0.39]
Density	vs.	MOE <sub>dyn</sub>	[+0.51]	+0.61	+0.64	[+0.48]	[+0.30]	[+0.30]	+0.57
MOE <sub>dyn</sub>	vs.	MOE <sub>st,3pb</sub>	+0.97	+0.71	+0.93	+0.97	+0.85	+0.95	+0.92
Creep deflection	vs.	MOE <sub>dyn</sub>	[–0.53]	–0.61	[–0.46]	–0.77	[–0.43]	–0.65	–0.77
Relative creep	vs.	MOE <sub>dyn</sub>	[–0.50]	[–0.06]	[–0.16]	–0.57	[–0.45]	[–0.41]	–0.66

<sup>a</sup> These relationships are presented in fig. 6. For abbreviations, see tab. 1.

Correlation between relative creep and MOE<sub>dyn</sub> was moderate for the control and TEOS, however it was significant for specimens modified with wax. In the cell wall modified specimens under low SL, the correlation between relative creep and MOE<sub>dyn</sub> was significant for MMF resin but have shown minor correlation for DM-DHEU resin. However, at high SL the MMF resin has shown minor correlation.

For relative creep the following pattern can be drawn: control and lumen fill modification (TEOS and wax) have shown the best correlation, moderate up to significant. The correlation presented by MMF resin was not confirmed by similar resin-based modification, DMDHEU.

Any pattern cannot be extracted from these correlation analyses. To confirm these findings, Epmeier and Kliger (2005) have shown similar conclusion, despite to be in the mechano-sorptive effect in creep. They presented correlations for unmodified Scots pine (0.05 up to 0.99 as R-value). However, for modified specimens, between relative creep and MOE<sub>st</sub> they presented minor correlation in acetylation, MMF resin and thermowood treatments (0.02 up to 0.4 as R-value).

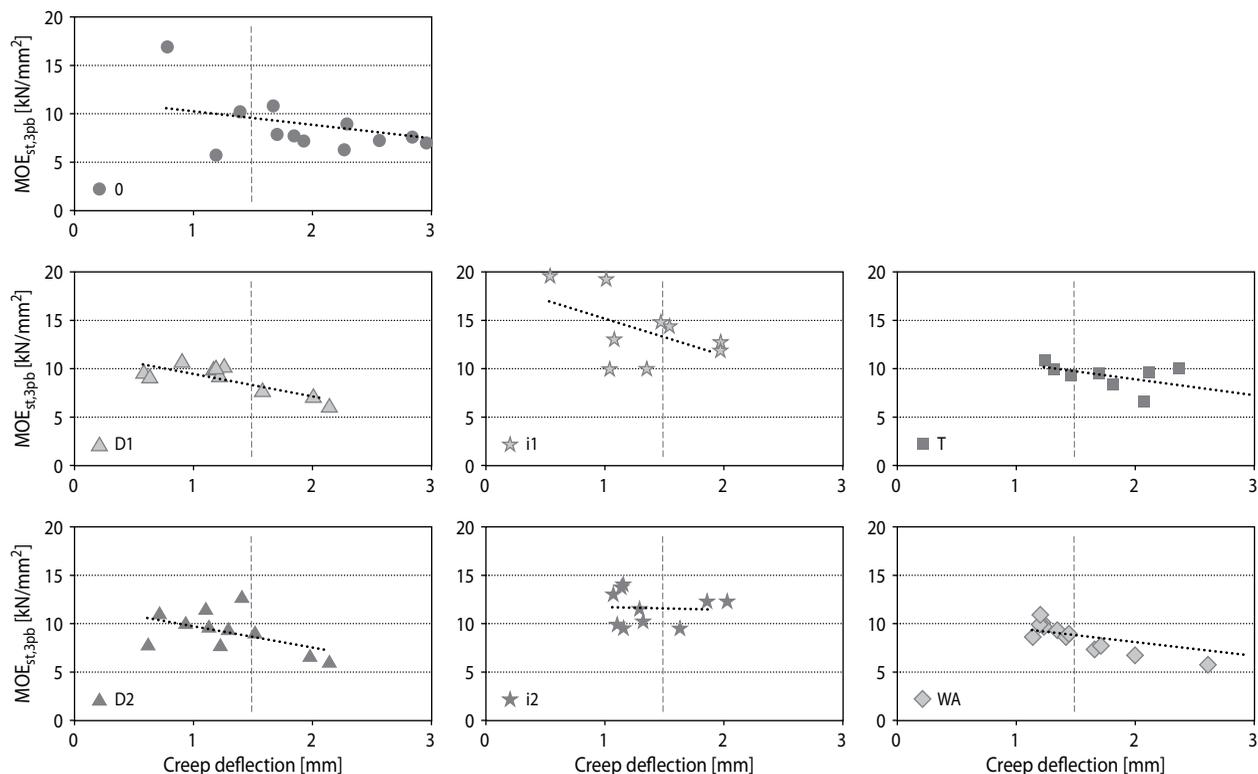
Creep deflection and stiffness have shown the best correlations. Moderate up to significant for all specimens, un- and modified (0.34 up to 0.89, as R-value), except for resin based with high concentration (0.03 and 0.05, as R-value).

Control and lumen fill treatments (TEOS and wax) correlated better than the modified specimens (resin-based modification, DMDHEU and MMF). As well as relative creep, from these correlation analyses in the creep deflection, any pattern cannot be extract.

The unaffected results in creep associated to the best correlation for different variables studied in this study, confirms the findings that the deposit material in the lumen has no effect in the creep.

The high correlation between creep deflection and MOE<sub>st</sub> could be expected, as the MOE<sub>st</sub> was calculated from the (creep) deflection after 60 s. In fact, two deflections were compared: the deflection after 60 s (under a short-term load) and the deflection after 5 weeks under a sustained load. However, low and medium SL are not enough to mobilize the same SL to reach in the standard MOE according to DIN 52 186.

The short-term deformation (MOE<sub>st</sub> or MOE<sub>dyn</sub>) was moderate to significantly correlated to the long-



**Fig. 6.** Correlation scatter plots between creep deflection [mm] and  $MOE_{st,3pb}$  [ $kN/mm^2$ //GPa] under SL of  $35 N/mm^2$  over a period of 35 days

For abbreviations, see tab. 1.

term deformation, creep deflection. This indicates that, measuring the stiffness (of a structural member), it should be possible to predict its long-term performance under load.

In Figure 6, scatter plots for the correlation between creep deflection (on the x-axis) and  $MOE_{st,3pb}$  (on the y-axis) are shown. The corresponding coefficients of correlation can be consulted in tab. 4 – Creep deflection vs.  $MOE_{st,3pb}$ . For specimens modified in the lumen fill (TEOS and wax), significant correlations were found (0.67 up to 0.78, as R-value), and unmodified wood has shown moderate correlation (0.37 as R-value).

Cell wall modified specimens (DMDHEU and MMF resin, with medium concentration) moderate correlation were found (0.5 up to 0.8, as R-value), not significant. In the resin-based modification, it seems that for high level of concentration the correlation disappeared.

## CONCLUSIONS

At indoor climate (65% of RH) and up to medium SL, all modification have shown no difference at creep behaviour. At high SL significant less creep was found in the cell wall modified specimens, similar to the SSE, up to 50%. The embrittlement effect imparted by the resins was the main reason to the reduction in the primary creep.

In saturated conditions, cell wall modification showed an enhanced relative creep. This behaviour with moisture is roughly similar to the EMC reduction in wet conditions, 87% of RH and in the same amount of the IBS reduction.

In these creep results for constant environment, indoor and saturated conditions, or even SL, modification with lumen fill (TEOS and wax) did not show any difference to unmodified pinewood.

Unmodified pinewood has shown a slight increasing of the relative creep at high SL ( $35 N/mm^2$ ), not

significant. In general, for un- and modified wood with both active principle (resin based, 1,3-dimethylol-4,5-dihydroxyethyleneurea and methylated melamine as well as TEOS and wax), the relative creep over 35 days/840 hours appears to be unaffected in the domain of stresses up to 35 N/mm<sup>2</sup> (0.4 of MOR).

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