

# Mechano-sorptive creep in pulp fibres and paper

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**Abstract** The factors affecting mechano-sorptive creep of wood and paper have been investigated for a long time. It has also been argued that single wood fibres do not exhibit mechano-sorptive creep and that the reasons for the accelerated creep under moisture cycling conditions instead are related to the bonds between the fibres. In order to examine the relevance of this argument, measurements on single pulp fibres of different composition were performed in tension, and the mechano-sorptive creep was compared to that of papers made from the same source of pulp fibres. All fibres tested were found to exhibit an increased creep rate during moisture cycling as compared to constant humidity conditions. Thus, pulp fibres show mechano-sorptive creep and in this sense behave similar to solid wood or paper products made thereof. A linear relation between the creep strain rate during cyclic humidity and the creep strain rate at a constant humidity was also noted for both fibres and paper. This relation was not affected by changes in hemicelluloses content or composition, neither for fibres nor for papers made of these fibres. However, in all cases, papers showed a much higher mechano-sorptive creep than the corresponding fibres they were made of.

## Introduction

Creep in wood and paper is accelerated due to moisture variations, i.e. it shows mechano-sorptive creep. This is a well-known and complex phenomenon that has been extensively studied for half a century. The mechanisms proposed a range from those on molecular mobility (Eriksson and Norén 1965; Gibson 1965; Bethe 1969; Bazant 1985; Navi et al. 2002), physical ageing (Padanyi 1993), material-specific inter-fibre mechanisms (Söremark and Fellers 1993; Haslach 1994; Alfthan 2004) to

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sorption-induced stress gradients (Habeger and Coffin 2000). For cellulosic fibres, earlier studies indicated an absence of accelerated creep (Sedlachek and Ellis 1994; Sedlachek 1995). On the other hand, Habeger et al. (2001) claimed that this result was an effect of improper moisture-changing rates only and have demonstrated mechano-sorptive effects on fibres of Kevlar, Rayon and Lyocell (solvent-spun cellulose fibres) and ramie. Recent measurements on single native spruce wood fibres have also shown a clear existence of mechano-sorptive creep (Olsson et al. 2007; Dong et al. 2010). The purpose of this paper was to clarify to what extent processed pulp fibres exhibit mechano-sorptive creep and, if so, how such an accelerated creep in the fibres compares to that seen for papers made of the same type of fibres.

## Materials and methods

### Fibre handling

Chemically isolated fibres were obtained by maceration of spruce chips (*Picea abies* [L.] Karst.) using a 50/50 % mixture of hydrogen peroxide and acetic acid at 50 °C for 78 h (Wang 1996). This process removed all of the lignin and about 45 % of the hemicelluloses. These fibres are termed holocellulose pulp fibres.

The effect of hemicelluloses composition was studied on fibres from a laboratory spruce pulp obtained using the kraft process with the addition of antraquinon and subsequently bleached. This pulp has a rather high content of hemicelluloses. The different hemicelluloses were then successively removed from the pulp by the treatment with alkali. Fibres were tested after each extraction of the two hemicelluloses. Xylan was first extracted with 1.6 M KOH at 25 °C for 16 h. To remove the glucomannan from this xylan-extracted pulp, extraction in two steps was performed, the first step with 1.6 M KOH and 0.5 M H<sub>3</sub>BO<sub>3</sub> for 36 h at 25 °C and an additional step with 1.5 M KOH and 0.75 M H<sub>3</sub>BO<sub>3</sub> for 23 h at 25 °C. To prevent the decomposition of the cellulose, nitrogen was bubbled through the pulp during all the extractions. The lignin content of the pulp fibres was determined as Klason lignin and acid-soluble lignin. Carbohydrate composition was determined by ion chromatography after acid hydrolysis.

Single fibres were thereafter carefully washed and dried on glass slides. Only straight fibres, presumably with a low microfibrillar angle and longer than approximately 2.5 mm, were used.

Measurements were here compared with the previously reported measurements on wood fibres (Olsson et al. 2007). These fibres had been carefully isolated from a single annual ring of a spruce tree (*Picea abies* [L.] Karst.). From a 150 µm thin slice of transition wood, the wood between early- and latewood, fibres had been isolated mechanically under a light microscope using very fine tweezers to retain the natural constitution of the secondary wall (Burgert et al. 2005).

## Paper

Isotropic laboratory sheets with a grammage of  $47 \text{ g/m}^2$  were made from the chemically isolated macerated spruce fibres, holocellulose pulp fibres, after beating in a PFI mill for 3,000 revolutions in order to have a good bonding between fibres.

For the bleached laboratory spruce kraft pulp and the corresponding hemicelluloses-extracted pulps, isotropic laboratory sheets with a grammage of  $30 \text{ g/m}^2$  were made.

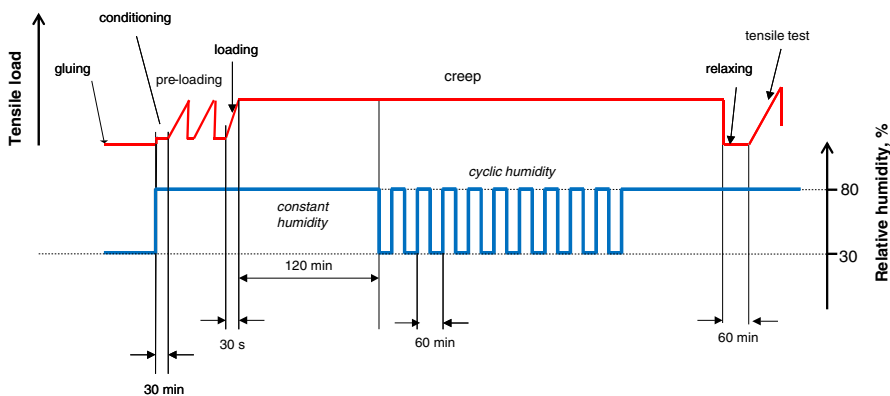
The sheets were dried under restraint in the STFI dryer for 5 min. Prior to testing, the sheets were cycled 6 times between 90 % RH and 50 % RH at  $23 \text{ }^\circ\text{C}$  in order to eliminate the influence of creep due to dried-in restraints from the sheet making.

## Mechanical measurements

All creep tests were made in a Perkin Elmer dynamic mechanical analyser (DMA), having a load capacity of 7 N. The testing procedure for the single fibre testing is shown in Fig. 1.

The air-dried fibres were glued with cyanoacrylate glue onto metal holders mounted in the DMA with a gap of 2.1 mm (for the previously tested wood fibres, a clamp gap of 1.2 mm had been used). After conditioning at 80 % RH and  $30 \text{ }^\circ\text{C}$  at a load of 10 mN, two load cycles between 10 and 50 mN were applied to uncurl the mounted fibre. Loading of the fibre was then gradually applied during approximately 30 s to a load in the range of 40–70 mN which was then kept constant throughout the test while registering the elongation. This load was in the range of 25–70 % of the breaking load for all fibre types tested. Firstly, creep in a constant climate of 80 % RH and  $30 \text{ }^\circ\text{C}$  was measured for 2 h. Thereafter, the climate was cycled 10 times between 30 % RH and 80 % RH with a cycle time of 1 h.

The fibre was unloaded and allowed to relax for a minimum of 1 h at zero load and 80 % RH. Thereafter, a tensile test was performed with a loading speed of 50 mN/min from which the elastic strain at the load used in the creep test was determined.



**Fig. 1** Schematic representation of the creep experiments

For some fibres, the stress level was determined based on the cell wall cross-sectional area obtained as an average along the fibre by the measurements using confocal laser scanning microscopy (CLSM).

Paper samples with a width of 2–5 mm were mounted in the DMA with a clamp distance of approximately 13 mm. After conditioning at 80 % RH at a load of 50mN, the paper was strained with a load corresponding to stresses between 1 and 7 MPa, and the creep was recorded using a similar test scheme as for the tested fibres. This load was in the range of 20–50 % of the breaking load for all paper types tested.

### Humidity generation

The climate surrounding the sample in the DMA was generated with a humidity generator (Wetsys). A dry and a water-saturated stream was regulated by a humidity probe giving a relative humidity of  $\pm 0.3$  % stability. The small volume, 20 cm<sup>3</sup>, of the testing chamber made it possible to rapidly change the RH. The sample temperature was kept constant using a thermostated chamber set to 30 °C.

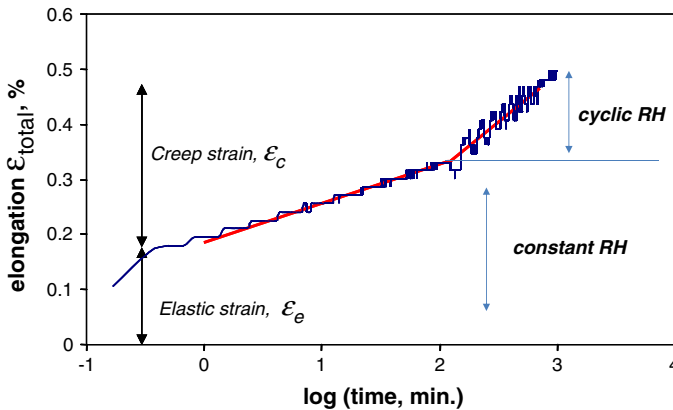
### Creep evaluation

The strain during a creep test may be viewed as composed of both an elastic strain,  $\varepsilon_e$ , and a creep strain,  $\varepsilon_c$  (Eq. 1 and Fig. 2);

$$\varepsilon_{\text{total}} = \varepsilon_e + \varepsilon_c \quad (1)$$

The elastic strain,  $\varepsilon_e$ , the strain that developed during loading, was considered to be confined to times below 1 min in these measurements. Thus, the creep strain,  $\varepsilon_c$ , was taken to be dominating the straining at times above that.

The creep rate was assumed to be a logarithmic function of time. Thus, here, the creep strain rate was evaluated by a linear regression analysis of the creep strain,  $\varepsilon_c$ , versus logarithmic time, and given as % strain/log time.



**Fig. 2** Schematic illustration of the creep experiments and definitions of strains. The figure illustrates a test of a single fibre experiment

For the cyclic creep period, an apparent creep rate was calculated as the average increase in creep strain over logarithmic time. This creep strain rate depends on the time period of the humidity cycle. Thus, the creep strain rate will be lower, the longer the cycling time period, provided that the cycle time is long enough for the mechano-sorptive phenomena to turn over into a constant humidity creep. At a cycle time of 1 h, the fibres and the thin papers here tested had reached the state of constant humidity creep. Thus, it was then possible to compare the rates of creep here determined with those measured earlier for wood fibres of 1.5-h cycling time by converting those rates into creep rates of 1-h cycle time by a simple subtraction of the extended creep period (creep between 30 and 45 min of each creep cycle at each RH being subtracted).

In order to compare mechano-sorptive creep properties for different materials, a mechano-sorptive creep ratio, here a 1 h MSC-ratio, was defined as follows:

$$\text{MSC}_{1 \text{ hour}(\text{cycle time})} = \varepsilon_{c(\text{cyclic } 1 \text{ h})}(t) / \varepsilon_{c(\text{constant})}(t) \quad (2)$$

where  $\varepsilon_{c(\text{cyclic } 1 \text{ h})}(t)$  is the creep strain rate at a cyclic humidity and a cycle time of 1 h, and  $\varepsilon_{c(\text{constant})}(t)$  is the creep strain rate in constant humidity.

Creep properties were also compared using the normalised creep strain  $\psi(t)$  given by the following:

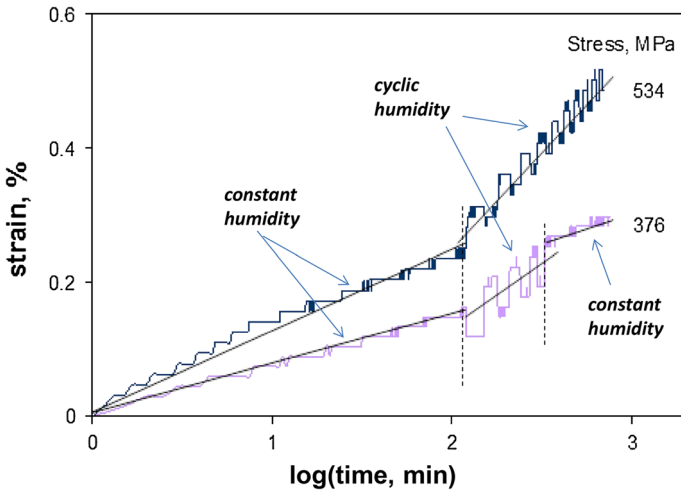
$$\psi(t) = \varepsilon_c(t) / \varepsilon_e \quad (3)$$

where  $\varepsilon_c$  is the creep strain, and  $\varepsilon_e$  is the elastic strain given in the tensile test by the applied creep load. In this way, the direct effect of different stiffness, E-modulus, of the material was compensated for.

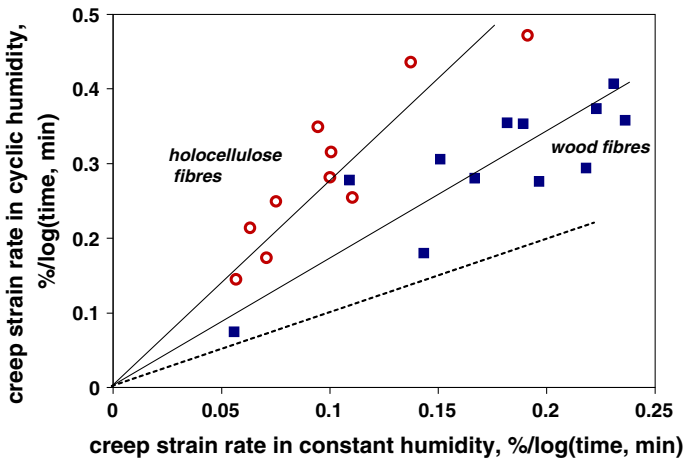
## Results and discussion

### Fibre testing

Figure 3 shows typical examples of creep curves for two holocellulose pulp fibres tested at different stress levels. Data are displayed as creep strain versus logarithmic time. The strain was set to zero at a loading time of 1 min ( $\log(\text{time, min}) = 0$ ), ignoring the actual initial loading of the fibre. The first creep from  $\log(\text{time, min})$  0 to 2.08 (1 to 120 min) occurred at a constant humidity of 80 % RH. The second phase involved periods at 30 and 80 % RH, each period being of 30-min duration. For the fibre loaded at the lower load, a second period of constant creep was applied at  $\log(\text{time, min})$  2.55 (360 min). The creep behaviour of the fibres may, as seen from Fig. 3, approximately be represented as being linear to the logarithmic time. As expected, fibres subjected to higher stress showed increased creep strain rates (Olsson et al. 2007). All fibres showed an increased creep strain rate during the cyclic humidity period compared to the first period at a constant high humidity of 80 % RH. A period of constant climate after the cyclic condition also showed the low creep strain rate, as indicated in one of the curves. It should here be pointed out that it is of great importance to be able to evaluate the creep in both constant and cyclic humidity for the same fibre due to the large variability between fibres.



**Fig. 3** Typical examples of creep strain,  $\varepsilon_c$ , versus logarithmic time for two holocellulose pulp fibres (the creep strain is taken to be zero at one minute of loading)



**Fig. 4** Creep strain rate at cyclic humidity versus creep strain rate at constant climate (logarithmic time) for holocellulose pulp fibres (*circles*) and wood fibres (*squares*), respectively. Linear regression lines for the two different fibre materials are indicated. The slope of those lines equals the MSC-ratio for the respective fibre type. The *dotted line* indicates a 1-to-1 relation

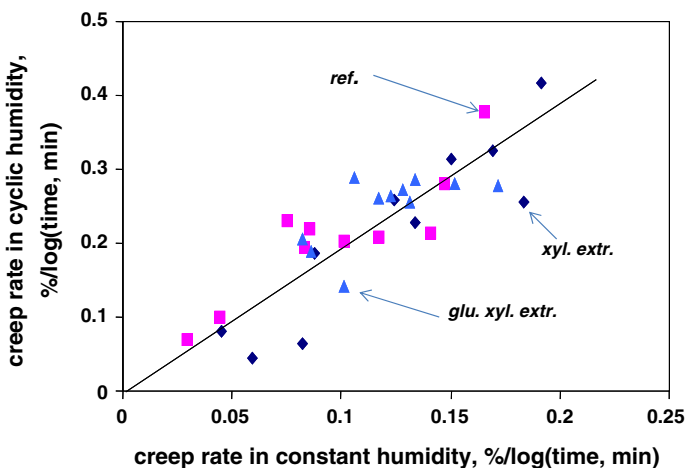
The holocellulose pulp fibres show a mechano-sorptive creep behaviour similar to that observed earlier for wood fibres (Olsson et al. 2007). Thus, the earlier claims (Sedlacek and Ellis 1994; Sedlacek 1995) that single pulp fibres do not show mechano-sorptive creep cannot be taken as a general conclusion. It is, however, possible that those experiments were made at unfavourable humidity cycling rates, 20 min as compared to the 60 min here used, and thus, the effect could be too small to be observable. The pulp fibres here tested had also a low microfibrillar angle. In

the case of fibres with high fibrillar angle, the mechano-sorptive creep diminishes (Dong et al. 2010) which could be another explanation for these earlier claims.

Figure 4 compares the creep properties of previously tested spruce wood fibres (Olsson et al. 2007) with the measurements of the holocellulose pulp fibres. All tested fibres showed a higher creep rate at cyclic humidity than the corresponding creep rate at constant 80 % RH. It was obvious that the pulp fibres had a higher MSC-ratio than that of the wood fibres, i.e. a higher mechano-sorptive creep rate as compared to that at constant humidity. For the holocellulose pulp fibres, the MSC-ratio was 2.8 as compared to a MSC-ratio of 2.1 for the wood fibres. The higher creep rates, both in cyclic and constant humidity, were for both fibre types mostly related to a higher applied stress. The main difference between the two types of fibres was their chemical composition. The holocellulose pulp fibres contained 70 % cellulose and 30 % hemicelluloses, while the wood fibres were composed of 45 % cellulose, 26 % hemicelluloses and 29 % lignin.

The creep properties of the kraft pulp fibres and the corresponding fibres extracted from the different hemicelluloses are compared in Fig. 5. For all these pulp fibres, a higher creep rate at cyclic humidity than the corresponding creep rate at constant 80 % RH was observed. There was, as seen in Fig. 5, for all fibre types some scatter in the creep measurements. However, there was no statistically significant difference in the mechano-sorptive creep ratio, MSC-ratio, between these fibre types and thus no effect of hemicelluloses extraction on the mechano-sorptive creep properties. The average MSC-ratio for the fibres was 2.0, a value similar to that of the previously tested wood fibres but lower than that of the holocellulose pulp fibres.

The hemicelluloses are the wood components with the highest moisture sorption capacity and could be assumed to influence the MSC-ratio. As seen from Table 1,



**Fig. 5** Creep strain rate at cyclic humidity versus creep strain rate at constant climate (logarithmic time) for pulp fibres with various hemicelluloses content; *ref.* bleached spruce kraft fibres, *xyl. extr.* xylan-extracted bleached kraft fibres, *glu. xyl. extr.* glucomannan-extracted fibres of previously xylan-extracted bleached kraft fibres. A linear regression line for the fibres is indicated

the proportion of hemicelluloses to cellulose is considerably higher for wood fibres compared to the pulp fibres. The much higher lignin content of the wood fibres should, however, be considered as a non-hygroscopic component that then changes the proportion of non-water sorbing components, thus giving a value of 0.35 for the proportion of hemicelluloses to these components. Although the relative change in glucomannan content was rather limited (Table 1), it is highly unlikely that the hemicelluloses content as such or the proportion of xylan versus glucomannan had an influence on the MSC-ratio. For wood samples, a complete removal of hemicelluloses has been shown to reduce both the constant and the cyclic creep (Fioravanti et al. 2006; Navi and Stanzl-Tschegg 2009). However, as a rather severe treatment with sulphuric acid was used in this case, other structural changes on the fibre level cannot be excluded. For the extracted wood samples, the moisture sorption was also too slow to reach equilibrium during the testing cycles investigated. As in the wood fibre structure it is the cellulose that almost completely controls its longitudinal properties (Bergander and Salmén 2002), a low contribution of hemicelluloses to mechanosorptive creep is not surprising.

It has recently been shown that the cellulose microfibril angle is of prime importance for the MSC-ratio; the MSC-ratio being lower, the higher the microfibril angle, MFA, and approaching zero at a MFA of 45° (Dong et al. 2010). Although the holocellulose pulping is performed at what is considered as very mild conditions, one cannot exclude some alterations of the cellulose structure and microfibrillar angle which could instead be the reason for its higher MSC-ratio.

In Fig. 6, the normalised creep strain rate at constant humidity is given as a function of the E-modulus for the two types of fibres, the holocellulose pulp fibres and the wood fibres, respectively. Apparently, the holocellulose pulp fibres have in general a much higher stiffness than the wood fibres which to some extent is a consequence of the much higher cellulose content being 70 % for these pulp fibres in relation to 45 % for the wood fibres. The span of E-modulus was larger for the holocellulose pulp fibres than for the wood fibres. This can partly be due to a larger variation in the microfibrillar angle of these fibres which were not chosen from any particular place of the wood tissue. However, one could not exclude that the few holocellulose pulp fibres with low E-modulus had to some extent been damaged at the end of the creep experiment, thus showing a lower than expected modulus. To some extent, the very high values for the pulp fibres could imply a high orientation

**Table 1** Chemical composition of fibre materials as well as their mechano-sorptive creep ratio, MSC-ratio

Fibres	Cellulose	Xylan	Glucomannan	Lignin	Proportion hemi/ cellulose	MSC- ratio
Wood	45	7.5	18.5	29	0.6	2.1
Holocellulose	70.5	9.5	20	0	0.4	2.8
Bleached kraft	76.5	7.5	16	0	0.3	2.0
Xylan extracted	83	2.5	14.5	0	0.2	2.0
Glucomannan and Xylan extracted	87	2.5	10.5	0	0.15	2.0



of cellulose microfibrils, an increased orientation of microfibrils occurring during the holocellulose pulping. Such a low microfibrillar angle for the holocellulose pulp fibres could thus be the reason for the higher MSC-ratio for these fibres as compared with the other samples in accordance with the earlier studies of an increasing MSC-ratio with decreasing microfibril angle (Dong et al. 2010).

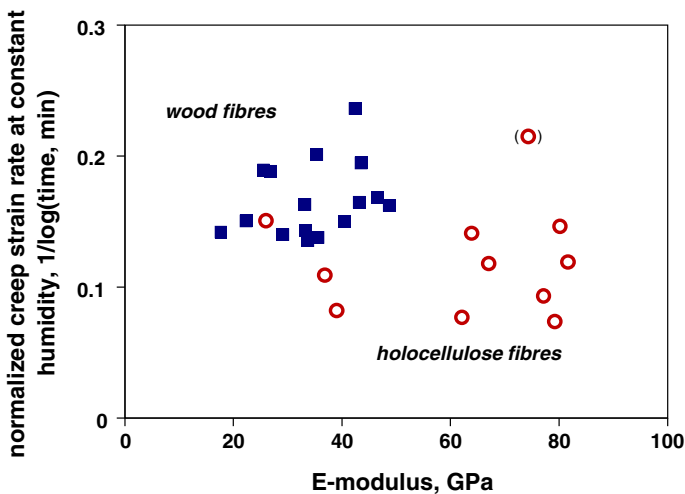
From Fig. 6, it is also apparent that the normalised creep strain rate at constant humidity seemed to be independent of the elastic modulus of the fibres. Apart from the outlier for the holocellulose pulp fibres, the trend is that these fibres displayed a higher modulus and a lower normalized creep strain rate than did the wood fibres.

#### Paper creep: fibre creep

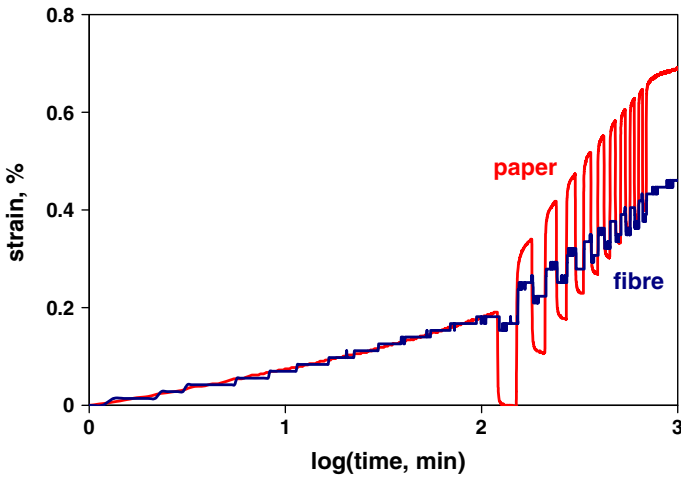
For paper, the origin of mechano-sorptive creep has in the past been related to different structural levels (Alfthan 2004; Coffin 2005; DeMaio and Patterson 2006). As indicated by the examples in Fig. 7, paper exhibited a much higher creep strain rate at cyclic humidity than did the single fibres that this paper is composed of, when compared at the same creep strain rate at constant humidity.

When comparing the creep strain rates in cyclic humidity versus that at constant RH for the holocellulose pulp fibres and an isotropic paper made thereof, it is apparent that the relations between the paper and the fibres were different with a much higher mechano-sorptive creep for the paper, see Fig. 8. The different measuring points were obtained by applying different levels of creep stress to the samples. The MSC-ratio was 2.8 for the fibres and 4.5 for the paper made thereof.

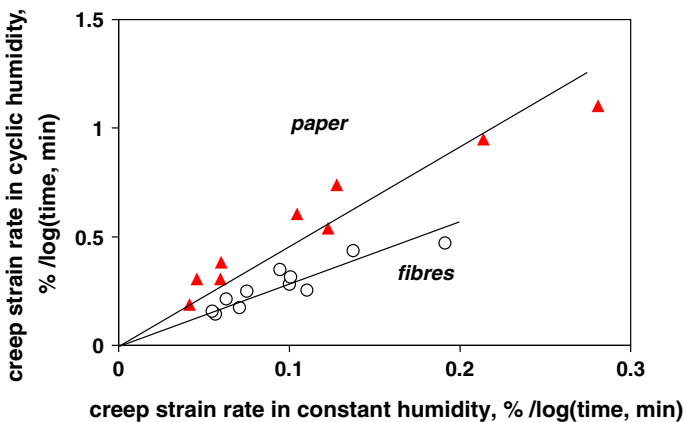
When making papers from the bleached pulp fibres as well as of the same fibres extracted from hemicelluloses, the MSC-ratios also were much higher than those for the corresponding fibres. For these papers, the MSC-ratio was 5.5, being nearly



**Fig. 6** Normalized creep strain rate at constant humidity versus the fibre elastic modulus, taken from static stress scans at 80 % RH, for holocellulose pulp fibres (circles) and wood fibres (squares), respectively



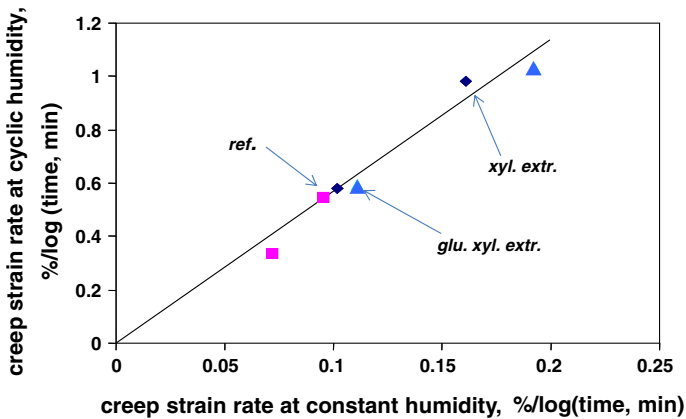
**Fig. 7** Creep strain,  $\epsilon_c$ , versus logarithmic time for a single pulp (holocellulose) fibre and a paper sample made from the same pulp. The samples were loaded to achieve the same creep strain rate in constant 80 % RH



**Fig. 8** Creep strain rate in cyclic relative humidity versus creep strain rate at constant climate (logarithmic time) for isolated holocellulose pulp fibres and for a paper made of these fibres, respectively

three times higher than that of the corresponding pulp fibres that they were made of. Also, as seen from Fig. 9, the extraction of the different hemicelluloses, xylan and subsequently glucomannan did not affect the MSC-ratio. Apparently, as also found for the fibres, the hemicelluloses themselves or the content of hemicelluloses does not play a role in the phenomena governing mechano-sorptive creep.

The much higher mechano-sorptive creep strain rates in paper as compared to fibres indicate that in paper, there must be additional creep effects apart from those coming from the straining of the fibres in its length direction. Bending and straitening of fibres as well as bond deformations could likely be factors of



**Fig. 9** Creep strain rate at cyclic humidity versus creep strain rate at constant climate (logarithmic time) for papers with various hemicelluloses content; *ref.* bleached spruce kraft paper, *xyl. extr.* paper of xylan-extracted bleached kraft fibres, *glu. xyl. extr.* paper of glucomannan-extracted fibres of previously xylan-extracted bleached kraft fibres. A linear regression *line* for the papers is indicated

importance. However, as shown here that single pulp fibres exhibit mechano-sorptive creep, this implies that those factors of the paper sheet are not the only origin of the phenomena of mechano-sorptive creep.

## Conclusion

Single fibre measurements of creep at constant and cyclic humidity conditions showed that mechano-sorptive creep occurred for pulp fibres of highly different composition. Thus, pulp fibres behave in general in the same manner as solid wood or paper products made thereof. The measurements indicated an apparent linear relation between the creep strain rate during cyclic humidity and the creep strain rate at constant humidity, here defined as a mechano-sorptive creep ratio—MSC-ratio. For pulp fibres, no influence of the hemicelluloses content or hemicelluloses composition on the MSC-ratio was found. A somewhat higher MSC-ratio for holocellulose fibres could probably originate from a higher microfibril angle for those fibres.

Papers made of the different fibre types here studied were shown to exhibit a much higher mechano-sorptive creep ratio than did the fibres they were composed of, compared at the same creep strain rate at constant humidity. To some extent, this could be a reflection of the distribution of fibres within the paper, with loading of fibres not only in their longitudinal direction. For the papers, no effect of the hemicelluloses content or composition was seen. This fact could probably imply that the hemicelluloses themselves are not contributing to the phenomena of mechano-sorptive creep.

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