THE ROLE OF EXTRACTIVES ON SHORT-TERM CREEP IN COMPRESSION PARALLEL TO THE GRAIN OF PAI WOOD (*AFZELIA AFRICANA* SMITH)

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(Received August 1995)

ABSTRACT

The creep behavior of extracted and unextracted blocks of Pai wood (*Afzelia africana* Smith) was examined in compression parallel to grain to determine the influence of both within-lumen and wall-resident extractives. It was concluded that the lumen-located extractive fractions have no significant effect on short-term creep, while the removal of wall-resident components permitted significant and accelerated creep development. However, as the cell walls of extracted wood were more reactive to moisture, the results of creep tests of this material must be interpreted with caution. It is proposed that the mechanism of influence on compressive creep is one of extractives stiffening the cell walls.

Keywords: Extractives, cell wall, lumen, creep, deformation, deflection.

INTRODUCTION

In addition to cellulose, hemicelluloses, and lignin, wood contains variable quantities of extractives as secondary components in the structure. Although contributing little to the total mass, the extractives can have a considerable influence on many of the physical properties. For instance, it has been observed that the presence of the extractives in wood cell walls enhances dimensional stability and limits its shrinkage (Bland 1971; Hillis 1986); reduces the wettability of wood and/or fiber surfaces (Chen 1970; Jordan and Wellons 1977); retards adhesion and reduces bond quality (Chen 1970; Hancock 1963; Plomley et al. 1976); lowers the heat of wetting (Avramidis and Dubois 1992); depresses fiber saturation point (Wangaard and Granados 1967); reduces permeability (Bosshard 1968; Bailey and Preston 1969); and increases the apparent specific gravity (de Zeeuw 1965).

The structural performance of wood under mechanical stress at the cellular level (disregarding the effect of extractives) has been likened to that of reinforced concrete (Freudenberg 1932), or that of a wound filament "composite" (Mark 1967). Thus the compact, axially orientated cellulose component is considered to control the tensile behavior of the composite (Mark 1967, 1972; Ifju 1964), while the three-dimensional and amorphous lignin protects the hydrophilic polymers from the effects of water (Winandy and Rowell 1984) and controls the compressive response (El-Osta et al. 1981; Klauditz 1952; Wadrop 1965).

Some controversy surrounds the role of extractives in determining the mechanical behavior of wood. Brown et al. (1952), speculating on the effect of removing extractives from wood, considered that the effect on compressive strength parallel to grain would be appre-

Wood and Fiber Science, 29(2), 1997, pp. 161–170

ciable, but less significant on shock resistance, the disparity in behavior being attributed to the fact that the former property depends on cumulative resistance of all components of the material, while the latter is contingent upon internal forces acting only within the cell-wall substance, which they claimed least altered by the removal of extractives. Kellogg and Ifju (1962), however, maintained that the effect of the removal of extractives could be positive or negative depending upon the mechanical property under consideration, and on the location of extractives in the structure. Frequently, the influences of extractives are explained by the following hypotheses:

a) A bulking effect. It has been suggested that because extractives are large molecules (e.g., lignin dimers and polymerized tannins), their presence in the structure keeps wood in a "semi-swollen" condition and hinders the number of available sites for the formation of intermolecular lignin-cellulose and/or intramolecular lignin-lignin bonds (Arganbright 1971; Sarkanen et al. 1967). This effect would be similar to that of moisture penetrating the polymer structure and diminishing intermolecular hydrogen bonding, thus reducing the strength. In addition, studies during nonsteady-state drying from the green condition have suggested that the presence of extractives results in signficiant reductions in both creep and shrinkage at lower temperatures, while increasing both phenomena at higher temperatures (Demaree and Erickson 1976; Erickson et al. 1972; Spalt 1979).

b) A plasticizing effect. The presence of extractives may promote plastic flow (Narayanamurti 1957; Pentoney and Davidson 1962; Spalt 1979; Tarkow and Seborg 1968).

c) An effect of stiffening the cell walls. It has been claimed that extractives exert a small but significant increase in the short-term mechanical properties of wood (Panshin and de Zeeuw 1970; de Zeeuw 1965; El-Osta et al. 1981; Luxford 1931).

Elsewhere, creep deformation has been reported to be dependent upon resin and lignin content (Rose 1965), but similar studies on Indian species gave inconclusive results (Narayanamurti and Verma 1964). More recently, Pizzi and Cameron (1986) presented circumstantial evidence to support the view that tannin extractives within the cell walls of drought-resisting tree species act as "springs," limiting the cracking of cell walls thus behaving in a mechanical fashion similar to cellulose.

Tropical hardwoods are known for their high compressive strength along the grain and, for this reason, are often used in structural applications. However, little is known about the contributions of the various extractive components to the creep behavior of these woods. The availability of this information is useful in helping to explain the performance of timber in structures and thus broadening the potential of utilizable species.

The objective of the work was to demonstrate that extractive components, especially those resident within the cell walls of Pai wood (*Afzelia africana* Smith), contribute to the creep resistance of the material. To this end, sequential extraction was performed using solvents able to penetrate the cell walls by varying degrees, and the creep behavior of both extracted and unextracted blocks of the wood examined in compression parallel to the grain direction.

MATERIALS AND METHODS

The material used in these creep tests consisted of matched, unextracted, and extracted blocks of Pai wood. All blocks were of heartwood, dried in an oven for 72 h at 90°C under nitrogen before being allowed to equilibrate in a controlled environment (60% RH, 20°C). Extraction was performed using sequentially: toluene, ethanol, an ethanol and toluene mixture (2:1 v/v), and finally water. Each stage in the sequence consisted of 72-h continuous extraction using the solvent and an equal period of reconditioning in the environment given above. The preparation of specimen blocks and the extraction procedures have been described by Ajuong (1994). The blocks studied

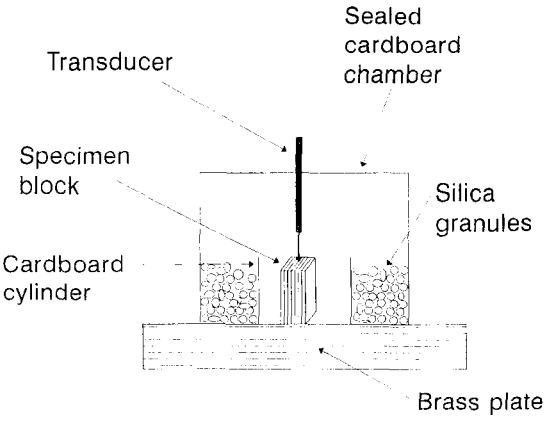

FIG. 1. Diagrammatic representation of specimen assembly for near oven-dry creep tests.

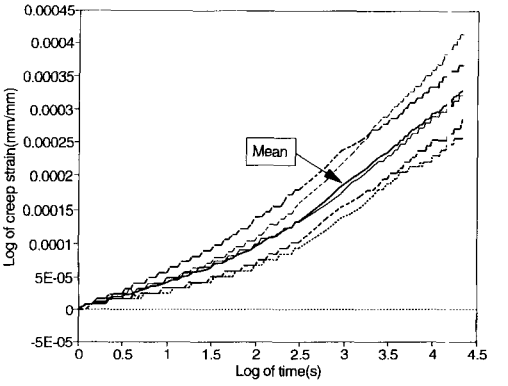

FIG. 2. Individual block creep variability around the mean of the ethanol extracted Pai wood from Zaïre.

under the ambient room conditions consisted of material from Zaïre and Nigeria. Matched blocks from the Zairean material were also examined under near oven-dry conditions. They were enclosed while under test in an assembly containing dry silica gel granules (Fig. 1). This arrangement ensured little moisture content change (0.07%/h) during the testing period.

Equipment

An oedometer was used to apply constant parallel-to-grain compressive loads to blocks. The suitability of this machine for creep testing of small wood blocks has been described and applied by Breese and Bolton (1993). The machine was enclosed in a small room heated to 20 ± 1°C by a thermostatically controlled electric heater. The temperature close to the test block was monitored by a thermocouple system, and both the temperature and the relative humidity of the environment were constantly recorded. The equipment was insulated against external vibration by being mounted on a sturdy concrete pillar set in sand.

The deformation of the wood block under test was measured by means of a displacement transducer linked to a linear voltage differential transformer.

The test blocks were carefully prepared using a sledge microtome. Conditioned matched blocks from both wood sources were pretested in compression along the grain using an Instron universal testing machine (Model 4301 H0695) to determine their respective ultimate strengths. When creep testing, stresses of 614 MPa and 345 MPa were applied, equivalent to 20% of the ultimate compressive strength. The load was applied for 6 h, and then removed, allowing the blocks to recover for a further 6 h. From each set of blocks, five groups of blocks were creep tested—the unextracted blocks (as control) and four groups exposed to different degrees of extraction.

RESULTS AND DISCUSSION

Behavior under ambient conditions

Graphs showing the typical variability of creep of individual blocks about the mean are given as Fig. 2 for air-dry ethanol-extracted blocks. This variability was observed for both the air- and oven-dry conditions for both the unextracted and extracted wood blocks. Figure 3 summarizes the observed creep behavior under ambient room conditions for the unextracted blocks and those subjected to various levels of extractive removal for Pai from Zaïre. Each curve is an average of 5 replicates.

Examination of these curves (save for the toluene-extracted blocks) indicates that the more extractives were removed, the greater was the creep sustained. In contrast, the blocks refluxed in toluene displayed reduced creep

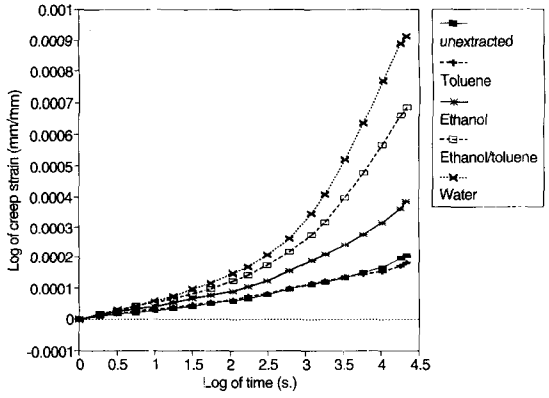

FIG. 3. Summary graphs of creep behavior for Pai from Zaïre at ambient room conditions using unextracted wood and blocks at various levels of extractives removal.

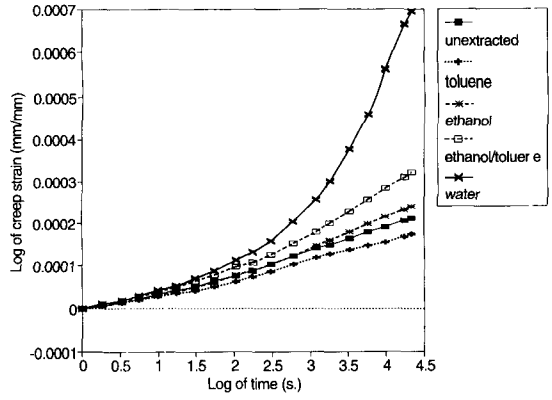

FIG. 4. Summary curves for creep response at room conditions using Pai wood from Nigeria.

compared with unextracted specimens. The parabolic model of Bodig and Jayne (1982) considers the total creep to be the sum of the instantaneous deformation and time-dependent creep:

$$Y(t) = a + bt^m \quad (1)$$

where $Y(t)$ = creep strain as a function of time, a = instantaneous deformation, t = time in seconds, and b and m are constants.

In the manner of Breese and Bolton (1993), only the deformation occurring after the elapse of 1 sec following the application or removal of load was considered to be time-dependent behavior and included in the analysis. Thus, neglecting the instantaneous term, and taking logarithms Eq. (1) becomes:

$$\text{Log}Y(t) = \text{Log}b + m \log t \quad (2)$$

Upon transformation, the data showed linear behavior between the logarithm of creep strain and the logarithm of time. Analysis of curve gradient using a multiple comparison approach was adopted because it is more appropriate to time-dependent behavior than comparison of momentary creep strain values produced after similar periods of elapsed time. Multiple comparisons analyses of extract components mean curves gradients using Fisher's least significant difference (LSD) revealed that under ambient conditions (Fig. 3) only the removal of the within-wall ethanol-toluene solubles and water-extractable components caused significantly different behavior from that of the unextracted blocks, notwithstanding the apparent similarity in the behavior of these two components. The creep of toluene-extracted specimens produced a behavior similar to that of the unextracted wood.

The results of creep tests of the replicate wood (Nigerian sourced) are presented in Fig. 4. Adopting similar multiple comparisons analyses, the data from this material displayed similarity in the creep behavior after extraction of the ethanol and ethanol-toluene leached components; but the creep behavior after removal of the water-soluble fractions was shown to be significantly different from that following extraction using other solvents.

When the behavior of air-dry, unextracted blocks (Fig. 3) was compared with that of its counterpart in the oven-dry state (Fig. 5), no significant differences in creep response were observed. This implies that the creep behavior of the unextracted wood (e.g., for this hardwood) is not significantly affected by moisture content changes below 6% moisture content, the level of moisture attained at reconditioning to air-dry following drying under nitrogen at 90°C.

The observed similarity of toluene and ethanol-toluene extracted specimens (Figs. 3 and 5) was examined to find out whether the treat-

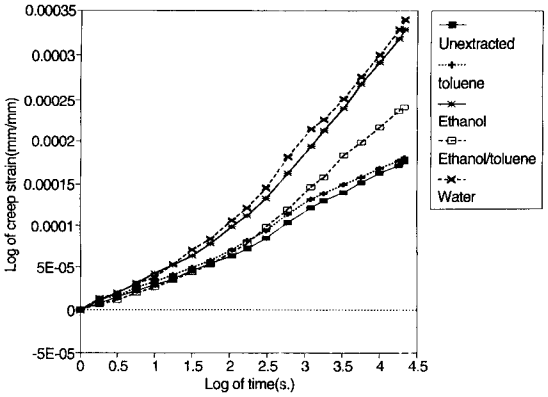

FIG. 5. Summary graphs of creep behavior for Pai from Zaïre at oven-dry conditions for the unextracted wood and wood at various degrees of extractives removal.

ments caused the observed behavior or whether it was merely evidence of variability in creep. The values of individual block gradients were compared using a *t*-test for paired comparisons. The analyses under oven-dry state (Fig. 5) revealed a similarity between the effects caused by the removal of the toluene and ethanol-toluene soluble components. This was to be expected given the similarity of the chemical nature of such substances (aliphatic compounds of nonpolar and of limited polarity) occupying the lumens and the large voids in the structure. Thus it seems reasonable to suggest that the difference in effects noted earlier under air-dry conditions may be due to the impact of adsorbed moisture and to variability in creep.

Comparison of behavior under ambient conditions with that after doubling the extraction time showed no significant differences in behavior. This suggests that the observed modifications in creep response could only have been caused by the removal of extraneous substances from the blocks, and not by thermally induced modification.

Behavior in oven-dry state

Graphs representing the behavior under oven-dry conditions using Pai from Zaïre are given as Fig. 5, each curve being an average of 5 replicates. The purpose of testing in the oven-dry state was to isolate any effects of moisture from those caused by the presence or absence of extractives. These graphs show the same general trends in behavior as observed under ambient room conditions. They demonstrate again increasing creep development with stepwise depletion of extractive components from within the cell walls. An observation of note is that the curves describing the creep behavior of both the toluene-extracted and the ethanol-refluxed specimens have reversed positions with those representing the creep of unextracted and ethanol-toluene leached material. Under the dry state, the analyses applied earlier were followed, but no significant differences in behavior were observed between the unextracted wood and toluene-extracted blocks. The ethanol-toluene refluxed specimens, though similar in their behavior to the toluene-extracted blocks, showed behavior significantly different from unextracted wood. They also differed in their behavior from specimens lacking other wall-resident components recovered by ethanol and/or water.

DISCUSSION OF RESULTS

The creep deformation of Pai wood under room conditions was found to be linear with respect to the logarithm of time. This might be expected given the low levels of stress applied and the ambient environmental conditions (Holzer et al. 1989; Schniewind 1968). The marked differences in the responses of the ethanol-toluene extracted and the water-refluxed blocks from the rest of the treatments may be caused by the increased level of extractive removal and the associated increase in moisture adsorption with its plasticizing action as described in previous reports (Holzer et al. 1989; Pentoney and Davidson 1962; Schniewind 1968). The curves reveal a progressive increase in susceptibility to creep with increasing extractive removal by water and ethanol, bulking solvents capable of recovering components from the cell walls (Morgan and Orsler 1969).

The comparative creep resistance of wood

extracted with toluene was unexpected. Traditionally toluene is regarded to be a non-wood-swelling solvent able to recover only lumen-located extract components, which add only weight to the wood, and have little effect on mechanical behavior (Arganbright 1971). As noted earlier, the blocks were allowed to re-adsorb moisture after drying under nitrogen and thus contained some surface-bound moisture (5.5% MC). It is believed that a double desorption phenomenon, involving moisture and water-soluble extractives, may have taken place from the cell-wall interior to the internal wall surface and from the interior of blocks to their surfaces in a manner similar to that described by Spalt (1979) and Hse and Kuo (1988). In addition, the high temperature of the extraction medium (toluene bp 117°C, 1 atm) may also have facilitated vapor phase migration of resinous components (Jordan and Wellons 1977; Spalt 1979). It is postulated that the desorption of moisture from cell walls and associated preferential redistribution of extractives towards the wall surface may give rise to a slight shrinkage (coalescence) with increased intermolecular H-bonding among wall microfibrils on sites vacated by both moisture and extractive substances. The consequent creation of a more reactive wall surface, coupled with microfibrillar coalescence, would produce an aggregated polymer structure more able to resist compressive stress (Spalt 1958). This behavior was not, however, replicated by oven-dry specimens.

The findings that the removal of toluene-soluble materials from the blocks produced behavior similar to that of the unextracted wood confirms the suggestion that the creep response of extracted wood is not influenced by the removal of lumen-located extractives.

Although some reports suggest that extractives only bulk cell walls and have no chemical bonding with the main wall polymers (Hse and Kuo 1988; Laks 1991; Spalt 1979), the present work suggests otherwise. It is proposed that the low molecular weight, polar extractives (such as epiafzelechin) removed by water frequently contain in the side chain sugar residues (King et al. 1955) and are chemically bonded through the side chain to wall polymers (lignin hemicelluloses and/or cellulose). This would also explain the difficulty encountered in their complete removal using solvent extraction. Evidence from previous reports supports this view (Bate-Smith 1962; Hillis and Carle 1959; Sarkanen et al. 1967; Sinclair et al. 1960). Thus the more polarized and the smaller are these compounds, the greater are the possibilities of ether bonding involving the structural wall polymers. Polarity implies shorter distances separating the extractive molecules from matrix surfaces, producing stronger extractives-polymer intermolecular bonding. A consequence of strong bonding will be reduced mechanical properties on extractive removal. This is clearly demonstrated in the present work: the removal of water-soluble extractives resulted in the greatest creep deformation.

The replicate wood from Nigeria produced less time-dependent deformation than the Zairean wood under the same conditions (Fig. 4). The increased resistance of the replicate material to creep-inducing stresses may be attributed to anatomical differences: the Zairean wood appeared to have interlocked grain, while the replicate material had straight grain. It may thus be that the Zairean wood is more susceptible to moisture effects. The results of the dry state analyses showed similarities in behavior following extractive removal using toluene and ethanol-toluene. This was to be expected: first, both types of components removed constitute small amounts of the total extract, and second, both are comprised of relatively high molecular constituents located in the lumen and in the gross wall capillaries. As noted earlier, such components will have little effect on the mechanical properties. However, the behavior caused by the wall-resident, nonpolar constituents is worth noting; it is believed to be the result of a mechanical adhesion. These compounds are usually present in the structure as polymerized insoluble products and may block the interfiber spaces and prevent chain slippage in unextracted wood.

Thus their removal is likely to increase chain flexibility and induce greater creep deformation. Conversely, the materials that caused the greatest creep deflection were not only preponderant in quantity but also highly polar. These findings concur with the suggestion that the influence of extractives on mechanical behavior is not only contingent upon their location in the woody plant cell but also on the amount present (Anon 1987; Arganbright 1971).

Spalt (1958) suggested that extractives are infiltrated into the wall structure prior to lignification when the structural polymers are dispersed. It is envisaged that the low molecular, polarized components, like the water-soluble fractions, can form ether bonds with hemicelluloses and cellulose in the cell walls. Such bonds, once established, may be sustained during lignification and the subsequent strength building processes described by Spalt (1979). Results of the present work in the oven-dry state therefore suggest that the influence of extractives on compressive creep parallel to grain also depends on the chemical nature of the components comprising the total extractives in wood.

The mechanism of the extractive effect

This study has shown that under ambient and oven-dry conditions, lumen-located extractives do not exert any significant effect on the creep behavior of wood. However, the removal of components resident within the cell-wall area does influence the creep behavior. Creep accelerates with the gradual depletion of extractive substances from the cell wall. Because extractives may be present as embodiments in the cell-wall structure, they have been considered as bulking agents (Choong 1969; Wangaard and Granados 1967). From the observations of oven-dry creep tests (Fig. 5), it is clear that the effect on creep behavior is different from that of a bulking agent, since if the extractives had behaved as bulking agents, then their removal from the structure would have produced increased resistance to creep-inducing stresses by chain coalescence and greater intermolecular H-bonding. Nevertheless, it is possible that the components removed by ethanol-toluene may behave as bulking agents: they produced lower creep response compared with ethanol solubles, and their removal seems to improve resistance to creep. This agrees with the findings that the creep of wood depends on lignin and resin contents (Rose 1965).

Accumulated evidence (Hse and Kuo 1988; Spalt 1979; Choong and Achmadi 1991) indicates that extractives participate in desorption processes because, being heat-sensitive at high temperatures, they may become redistributed within cell walls or be completely desorbed. Under the influence of heat and mechanical stress, the water-repellent components become less viscous, solubilize and, together with the water-soluble fractions, permit, by lubrication, flow and/or repositioning of microfibrils in "awkward" positions. If the extractives of this wood were thus behaving as plasticizers, they would produce, contrary to the findings of this work, greater creep deformation in the unextracted compared to the extracted blocks, as previously documented by Erickson et al. (1972); Narayanamurti (1957); Pentoney and Davidson (1962); and Spalt (1979).

83.5% of the extractive content of this wood consisted of tannins (the material removed by both ethanol and water, and soluble in a mixture of acetone-water (1 : 1 v/v)). These compounds are known astringent substances (Erickson 1968). Their close association with microfibrils in cell walls may confer increased stiffness to the chains in the manner described by Panshin and de Zeeuw (1970), and their removal will give rise to increased flexibility and creep deformation. This is in agreement with the findings of this work where the greatest reductions in creep resistance were observed on the removal of the ethanol and water-soluble substances. The present observations support the proposal by Pizzi and Cameron (1986) that tannins function as "springs" in cell walls disallowing deformation. Simi-

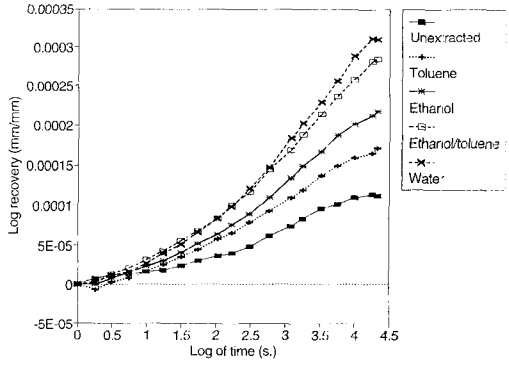

FIG. 6. Summarized curves for creep recovery for the unextracted wood and wood at different levels of extraction at the ambient conditions.

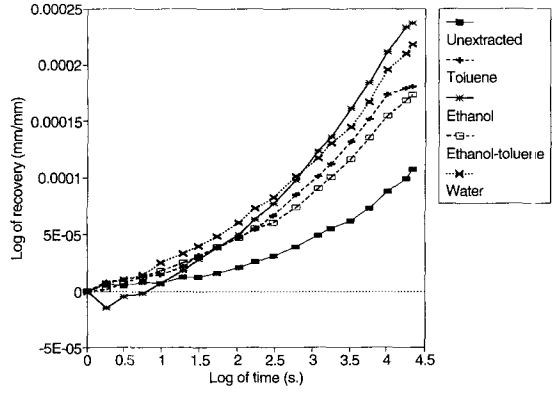

FIG. 7. Summary graphs for creep recovery for the unextracted and solvent extracted wood from Zaïre under oven-dry conditions.

larly, the loss in wall rigidity (a property frequently associated in mechanical studies with cell-wall lignin) obtained by Narayanamurti and Verma (1964) lends support to the view that lignin and extractives are bonded by ether bonds. It is envisaged that partial removal of the water-soluble extractives may result in the distortion of such bonds, weaken the structure of lignin in the cell wall, and eventually lead to losses in wall rigidity in compression. The recovery curves given in Fig. 6 indicate that only 34% of the creep deformation under the ambient conditions is recovered. This value increased to 70% in the oven-dry state (Fig. 7) and clearly shows the need to exercise caution when interpreting results of creep tests for extracted wood.

CONCLUSIONS

The creep behavior of extracted and unextracted blocks of Pai wood has been examined under air-dry and oven-dry conditions to find out the contributions made by the various extractive components to the creep behavior of wood and, to explain the possible mechanisms of extractives effects. The following conclusions have been drawn:

1) The nonpolar, high molecular weight lumen-located extractive components did not cause any significant differences in the creep behavior when compared to that of the unextracted wood, while the removal of polarized extractives from within the cell walls accelerated creep development. Implicitly, the presence of extractives increases the resistance to creep.

2) The components recovered by the ethanol-toluene mixture resembled in their behavior that of a bulking agent; their removal from wood improved the resistance to creep-inducing stresses.

3) The cell walls of the extracted wood were found to be more reactive to moisture effects. Consequently, there is need to exercise caution when interpreting the results of creep tests on extracted material.

4) Based on the analyses of creep behavior, it has been proposed that the extractives exert their influence on creep by the mechanism of stiffening and hardening cell walls in which they are resident.

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