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High temperature and chemical effects on wood stability

Part 1: General considerations

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Summary. The increasing supplies of fast-grown woods grown on short rotations contain significantly higher proportions of juvenile wood with properties different from those of mature wood. Improved processes will be required to produce dried wood that is satisfactorily stable with few distortional or dimensional changes. The basic wood features affecting different forms of instability are considered. Variations in the amounts of cellulose, hemicelluloses and lignins in wood, the changes in them and the degradation products formed under different conditions are discussed. Changes in the nature of hemicelluloses appear to play an important role in conveying stability. The different volumes of heartwood in green timber have an effect on moisture levels and other properties. As industry is increasing kiln temperatures for high-temperature drying, the effect of time-temperature-moisture relationships on stability and degradation are discussed. The effect of ammonia and other chemicals on stability is considered.

Introduction

Increasingly in many countries, the wood from fast-grown trees will supply the general-purpose needs for pulp and solid wood products. The trees will be increasingly from pine or eucalypt species, their rotation ages will decrease and the proportion of the tree used will increase.

Trees form juvenile wood (Zobel, Blair 1976) in their crowns or when young and begin to form mature wood after about 10 years' at a particular level. The exact age of mature wood formation depends on the species and the height of the sample in the tree. The proportion of juvenile wood in fast-grown, short-rotation pine logs can be high (e.g. Zobel, Blair 1976) and increases with decreasing age of the tree. Variable proportions of other features (such as knots and heartwood) can be found in the central zone (or corewood), depending on age and previous silvicultural treatments.

Several studies have shown that, with increasing distance from the pith, the fibre length, density, and cell wall thickness increase rapidly in the first few years; conversely, the microfibrillar angle of the fibre declines. In the mature wood, such changes occur much more slowly and the rate of change of these properties across the stem varies with species and growth conditions. Spiral or sloping grain can be severe (as high as 20 degrees) in the juvenile wood of softwoods. It is low at the pith but reaches a maximum, usually before the fifth growth ring (in *Pinus radiata*

D. Don), and then decreases from a left-hand spiral to a straight-grained condition and sometimes a right-hand spiral in the mature wood (Lowery 1966). Spiral grain results in undesirable properties including twist during drying and in service.

The gradients of growth stress levels in young small-diameter hardwood trees, particularly eucalypts, are greater than in larger trees. Consequently, when small-diameter logs are cut, the resultant splitting and distortion can be greater, so that recovery of sawn wood is affected (Hillis 1978). Improvement of the stability of the raw material in these resources will improve the effectiveness of conversion.

The major forms of instability are those associated with (1) stressed wood in the tree resulting in spring on sawing, (2) distortion on drying, (3) spiral grain resulting in twist on drying, (4) dimensional changes resulting from changes in the moisture content of the wood, and (5) deformation occurring in use. The changes contributing to instability are not confined to one phase or stage but are complex and overlap and interact. This paper considers some of the basic aspects of this complex situation and their influence on stability and strength in order to provide parameters for improved processing. The discussion is largely confined to pine wood.

High-temperature drying techniques overcome some of the problems of instability developed when juvenile pine wood is dried at temperatures lower than 100 °C. Moreover, there are significant economic savings if these techniques are used and the product can be superior in a number of properties. However, in the absence of precisely defined parameters or an adequate understanding of basic wood properties relevant to those techniques, production problems could arise with improperly dried material, particularly if it contains juvenile wood.

Drying

The temperatures used in high temperature drying are increasing. Kiln temperatures of 115 °C and higher have been used commercially for some time together with restraining loads (1000 kg/m² of stack top surface) during the drying period (Christensen 1970; Koch 1972). At 115 °C, about one-quarter of the time and one-half of the energy used in normal drying is taken to produce stress-free, stable material of the same or slightly less strength than normal but with very slight surface checking or with very little cracking around knots, with reduced shrinkage and without internal honeycombing (Koch 1972). The various continuous kilns, through which the wood is moved as it dries, are designed to subject the wood to temperatures of 130 °C and higher and to fast air-streams so as to dry the wood in a few hours (Christensen, Northway 1978; Koch, Wellford 1977; Rosen 1978). The effects of the drying temperatures and time of application on the stability and strength of the dried products have not been defined completely.

The large concrete block usually used as the restraining load in batch processes of high-temperature drying occupies kiln space, absorbs heat and can introduce abrasive particles into the wood. It may be possible to reduce the size of the restraining load if the wood could be made more plastic during the drying procedure. The value of a 'pre-steaming' of green wood or plasticization phase, introduced for this purpose and to assist in the control of distortion, has been debated.

Chemical characteristics that could affect wood stability

Composition of sapwood and juvenile wood

For the purposes of assessing torsional stress and heat, the lignins in the lignin-enriched middle lamella between fibres can be considered as a separate phase, even though a small proportion of hemicelluloses will be present. The lamellae of the cellulose, hemicelluloses and lignins components, which are not continuous around the cell wall (Fengel 1971; Kerr, Goring 1975, 1977), can be considered as another phase of this multicomponent.

Within a growth ring, the cellulose content varies from 40% in the earlywood to 50% in the latewood of *Pseudotsuga menziesii* (Mirb.) Franco, with the lignins content decreasing from 28 to 24% (Kennedy, Warren 1969). Similarly, in the mature wood of *P. resinosa,* the latewood contains less lignins and xylans and more mannans than the earlywood, but there is no difference in the galactans and arabans content (Larson 1966).

There are considerable variations in the cellulose, hemicelluloses and lignins contents of woods of the same species. For example, the amount of lignins can vary between trees of the one eucalypt species and, as such, can affect the development of collapse and other properties (Bland 1971). In the juvenile wood of the southern pines and *Ps. menziesii*, there is less cellulose and more hemicelluloses than in the mature wood (Zobel et al. 1966; Zobel, McElwee 1958; Kennedy, Jaworsky 1960). The cellulose content of *Eucalyptus globulus* Labill. increases with age but mainly after the trees are 12 years old (Celbi 1979). In *P. radiata* and *P. resinosa* Ait. the cellulose content increases and the lignins and pentosans contents decrease from the pith outwards over the first 10 to 15 growth rings, after which all values remain approximately constant (Uprichard, Lloyd 1980; Dadswell et al. 1959; Larson 1966).

Both the amount and the composition of the hemicelluloses change with the age of the tree when they are formed. In *P. radiata* and *P. resinosa* there is a considerable decrease in the galactose content of the wood hydrolysates and there are decreases in the xylose and arabinose contents from the pith outwards to the 20-year-old material; on the other hand, there is an increase in mannose content (Uprichard, Lloyd 1980; Larson 1966). Along the stem axis there is little or no change in the hemicelluloses.

It is considered that species and tree age have a greater effect than genetic selection on the variability of chemical composition, than genetic selection, site quality and silvicultural treatment. The variability within stems can differ within and between pine species and be greater than that of spruce species (Harris 1981). However, increases in the cellulose content have been achieved by breeding *E. globulus* trees (Celbi 1979).

Changes in composition due to secondary differences

Due to the low density of juvenile wood and the flexibility of fast-grown trees under windy conditions, a high proportion of reaction wood is frequently found in the

central portions of stems when compared with mature wood (Zobel, Blair 1976). Both compression wood and tension wood are denser than normal wood and show high longitudinal shrinkage. Compression wood contains substantially more lignins and galactans and less cellulose than normal wood (Timell 1982). Tension wood contains a much higher percentage of cellulose than comparable normal wood and lower lignins and pentosans. The reaction wood from pine and eucalypts contains substantial amounts of galactose whereas comparable normal wood contains none (Dadswell et al. 1958).

The formation of heartwood at different ages of tree growth, which depends on the species and sometimes the rate of growth, introduces further heterogeneity in material being dried. Eucalypt heartwood formation starts after about 5 years' growth whereas *P. radiata* heartwood starts after about 14 years. Heartwood contains more extractives than sapwood and some extractives can penetrate the secondary wall of fibres to affect the shrinkage and stability of wood on drying (Wangaard, Granados 1967). The highly dimensionally stable *Sequoia sempervirens* D. Don Endl., has a high amount of extractives in the cell wall (Tarkow, Krueger 1961).

The heartwood in eucalypts usually, but not always, has a lower moisture content (Hillis 1978) and in many conifers it has a considerably lower moisture content than the sapwood. Furthermore, in heartwood the aspiration and blockage of pits, the presence of tyloses in the vessels of several hardwoods and the blockage of parenchyma and vessels with extractives can contribute to lower permeability and water movement and cause distortion during drying. Variations in the volume of heartwood and consequently the uneven moisture content of a stack of wood affect the rate of drying and the time taken to affect changes in the chemical components so that the desired stability can be achieved.

Anatomical and ultrastructural characteristics that could affect stability

In juvenile wood the shrinkage of both hardwoods and softwoods varies according to the combined effects of the density of the wood, the average microfibrillar angle of the cell wall and the extent of lignification (Meylan 1972). The larger microfibrillar angle in juvenile wood fibres results in greater longitudinal shrinkage and less transverse shrinkage than in mature wood.

Because of variations in cell wall porosity, in amounts of hemicelluloses and lignins, and in the degree of crystallinity of cellulose, the packing density of the wood can vary (Jayme, Krause 1963). Packing density is more relevant than bulk density to a number of properties including water-adsorption capacity and chemical reaction (Kellogg, Wangaard 1969). Species with high basic densities have high packing densities and the packing density was appreciably higher in latewood than earlywood, with the differences being greater in mature than in juvenile wood (Perng, Tajima 1981).

The stresses formed in the developing sapwood as a result of growing conditions can affect the anatomy and properties of the wood so that the resultant reaction woods have wider growth rings than normal woods, higher densities, greater microfibrillar angles and show greater than normal instability and shrinkage during

drying. Also the peripheral growth stress, which can reach high levels in hardwoods, is associated with increased thickness of the cell wall, change in microfibrillar angle and intensity in lignification (Nicholson et al. 1975; Boyd 1977).

Physical changes that could affect stability

Deformation and plasticity

Deformational changes depend on several factors including the moisture content and the temperature of the wood.

Effect of moisture

The absolute value of deformation or creep of loaded wood increases with increasing moisture content. However, the ratio of the total deformation at a given time and temperature to the initial deformation when the load was applied is affected little by the moisture content if it is maintained at a constant level (Armstrong 1972). If the moisture content of the wood is changed during the application of a load then a greatly enhanced deformation occurs. The rate of change of the deformation depends on the rate of change in moisture content and the amount of deformation depends on the amount of change in moisture content. This mechano-sorptive component of deformation (Grossman 1976; Arima, Grossman 1978) commonly arises when undried wood is kept under load while it dries.

Deformation under load can be large and take place rapidly when, for example, the surfaces of dried veneer are moistened at room temperature or when wet veneer is dried, but it cannot be explained in terms of the effects of water movement alone (Armstrong 1972). Deformation induced at room temperatures is recoverable when the sample is unloaded and subjected to moisture cycling.

It is assumed that the change in moisture content causes changes in bond configuration of the non-crystalline regions of adjacent cellulose molecules and of hemicelluloses with the cellulose, resulting in swelling or shrinkage. The crystalline regions of cellulose would resist movement and, for permanent stability, it would be necessary to increase the compliability of the rest of the fibre wall. As a result, during autocontraction of the wall on drying, more hydrogen bonds would be formed, so that the new positions would be held in place.

Effect of temperature and other factors

The compliability of fibres can be improved by raising the temperature above the glass-rubber transition, or softening, points. In addition, the uptake of water, or diluents of low molecular weight, would loosen the bonds between the hemicellulose and lignin polymers to enable their flow and to assist movement of the non-crystalline regions of cellulose.

Changes in the nature of some isomers could decrease the energy required to enable movement and also result in lowered softening points. Hillis and Rozsa

(1978) have shown that the softening points are lowered by heating undried wood (plasticization) for two hours at 90 °C and lowered substantially by heating at 100 °C. It is expected that subsequently, when the material cooled, the polymers would harden and form bonds in the new positions to which they have flowed. Not only is the softening point of hemicelluloses lowered significantly by a plasticization phase (Hillis, Rozsa 1978), but the rate of change of torsion angle under the same torsional load (a measure of decrease in rigidity) is three times higher at this softening temperature.

The softening changes depend on a temperature-time-moisture relationship affected by the chemical composition of the matrix. The latter differs between softwoods and hardwoods and possibly parts of the same tree such as sapwood and heartwood, juvenile and mature wood. The time and temperature of heating undried wood before drying commences must be sufficiently great to allow the temperature of the interior of the material being dried to reach conditions adequate not only for plasticization or softening of the wood but also for bringing about changes to increase subsequent stabilization of the wood.

P. radiata pine wood under restraint is pre-steamed at 100 °C for 2 h (the plasticization phase (Mackay, Rumball 1971, 1972)) in some commercial high temperature drying processes. Under laboratory conditions, pre-steaming makes the wood less rigid and a lower torque is required to straighten studs during drying (Mackay 1973). The initial heating of the undried wood can be achieved by a pre-steaming period or by heating the wood to high temperatures quickly by a pre-heating period before drying commences.

In the high-temperature drying process, less torque is developed with the faster drying rates, but probably more elastic deformation occurs at the expense of plastic deformation (Mackay 1973). Pre-heating with steam at superatmospheric pressures and above the glass transition temperatures for short periods results in a lowered torsional modulus in pulpwood (Hoglund et al. 1976). Also, after pre-heating in saturated steam at 120 °C, thermochemical pulps have been produced at reduced energy costs and with properties superior to those obtained by steaming at atmospheric pressure (Sinkey 1979).

In the plasticization stage, conditions that may reduce the required weight of the restraining load may not necessarily be the optimum to improve the stability of the final product. The outer few millimetres of a board, which determine most of the rigidity, reach 100 °C and pass the softening points within the first few minutes of the heating period. The interior of the boards will reach the softening temperatures more slowly and, if adequate plastic flow and set is not achieved before drying is completed, the correction of distortion could be temporary. This would be revealed when the moisture content of the outer layers increases and loss of rigidity results.

These principles are involved in the press-drying process for papermaking, when the pulp webs are dried simultaneously under both heat (above 100 °C) and pressure (about 5.5 MPa) (Byrd 1982) to produce papers having considerably improved strength properties as well as dimensional stability on exposure to moisture. A considerable part of fibre bonding is achieved if the total restraint in press-drying (at 400 °F, 204 °C) is maintained during removal of moisture in the critical range between 65 and 20% moisture content (based on initial wet weight)

(Setterholm, Benson 1977; Horn 1979; Back et al. 1979). The improved adhesion obtained is considered to be due to the increased rate of flow of the hemicelluloses on the surface of the fibres as a result of a lowered softening point (Luce 1964; Horn 1979).

Hygroscopicity and stability

Drying at temperatures from 100 to 180 °C results in a reduction of equilibrium moisture content (e.m.c.) (Suematsu et al. 1980; Orman 1955) by as much as 3% compared with air-dried wood (Schneider 1973; see also Koch, Wellford 1977) with no effect due to wood species (Suematsu et al. 1980). Raising the temperature from 130 to 180 °C results in only a slight decrease in e.m.c. in the woods of pine and beech (Schneider 1973). When, during the drying of *P. radiata* wood in a deformed state, the temperature is raised from 25 to 80 °C for 24 hours (Arima, Grossman 1978), some of the latent capacity of the timber to recover its shape with change of moisture content is lost. Also, drying green *Ps. menziesii* and *Tsuga heterophylla* (Raf.) Sarg. at increasing temperatures in the range 32–100 °C and *P. radiata* in the range 25–93 °C progressively lowered the e.m.c. and reduced the rate of moisture change and shrinkage and swelling (Kininmonth 1976). *Pinus radiata* dried at 77 °C and also at 115 °C swells 12–35% less and 17–28% less respectively than air-dried material when subsequently exposed to high humidity. It appears that the effects are dependent on time as well as temperature.

An increase in the temperature (over the range 70–200 °C) and in the time of exposure of dried wood to those temperatures reduces moisture sorption. Kiln drying at high humidity reduces hygroscopicity more than drying at low humidity, and higher temperatures are needed to achieve similar results on dry wood (Kininmonth 1976). Reductions in e.m.c. and the decreased rate of moisture uptake of wood dried up to 93 °C are not surface phenomena but involve the wood as a whole (Kininmonth 1976).

When wood is heated, changes in the nature of cellulose, hemicelluloses, lignins and extractives can modify its hygroscopicity, stability, diffusibility or permeability. Controlled heating of undried wood (as in the plasticization phase) could result in chemical change and a decrease in the hemicelluloses content without the loss of strength observed after prolonged heating. A low or reduced hemicelluloses content has been associated with the high dimensional stability of *Tectona grandis* L. f. heartwood or heat-treated dried oak wood (Burmester 1975; Burmester, Wille 1975; Wise 1952). Heating pine wood for 7 hours at a dry bulb temperature of 130 °C (wet bulb 90 °C) increases stability, reduces the pentosans content from 11.0 to 9.1% and the cellulose content slightly (Potutkin and Shirayena 1975). The stability of dried wood (e.g. the response to moisture gradients and cycling during use and storage) depends on the time and temperature of heating undergone in both the wet and the dry states. The kinetics of thermal degradation during the different stages in drying have been considered by Skaar (1976).

Heating wood with moisture contents below 15% and at temperatures of about 100 °C would result in physical changes that decrease the accessibility of hydroxyl groups in hemicelluloses to water (hornification), with a resultant decrease in

swelling or mobility. The rate of transference of heat to the interior of dry pieces would be slower than with wet wood. There is evidence that, at very high temperatures, the hemicelluloses in paper and hardboard may be changed to furfural polymers, which are less hygroscopic towards water (Stamm 1964), or that acetal linkages between hemicelluloses and cellulose are formed (Back 1967).

Changes in wood components at high temperatures and with chemicals

High temperatures

The rate of change of both the chemical and physical nature of the components of wood increases with increasing temperature.

Skaar (1976) found that, whereas the thermal degradation of dry wood appears to follow a first-order chemical reaction, the reaction rates for wet wood are ten times greater or more, and the rate of degradation increases rapidly with increasing temperature. Approximately 53 days' heating at 100 °C is required to reduce the modulus of rupture (MOR) of dry wood by 5% whereas only 0.67 and 0.2 days are required to achieve the same effect by steaming wet wood at 100 °C and at 120 °C respectively. The MOR (after conditioning) of spruce, pine and fir dried at a dry bulb temperature of 116 °C is significantly lower than that of material dried at 66–80 °C (Huffman 1977). On the other hand, there is little loss in strength of *P. radiata* wood dried at 120 °C with a drying period of 15 to 16 hours (F. J. Christensen, personal communication; see also Sumi 1982).

Reports of the effect of high-temperature drying (particularly above 175 °C) on the strength and brittleness of wood vary possibly because of the different chemical and anatomical nature of the wood dried and different methods of heating. Hardwoods and refractory species with lower permeability than the pines have the greatest strength losses and these species include *Ps. menziesii, Abies balsamea* (L) Mill. and *Picea glauca* (Moench.) Voss. (Salamon 1963; Koch 1971; Schaffer 1973; Schneider 1973; Smith, Siau 1979; Yao, Taylor 1979). The presence of wane, and knots that dry faster than clear wood, may cause problems due to localized overdrying (Rosen, Bodkin 1978).

The maintenance of undried or rewetted wood at ambient temperatures for long periods or its exposure to higher temperatures for shorter periods can lead to degradation and change of hemicelluloses, and even cellulose, that affect (mainly deleteriously) the properties of the cell wall and the wood (cf. Feist et al. 1973; Hillis 1975; Hillis, Rozsa 1978; MacLean 1954, 1955; Rusche 1973; Troughton, Rozon 1974; Sumi, McMillen 1979; Millet, Gerhards 1972). Part of this change involves the release of acetic acid (MacLean 1953; Feist et al. 1973; Kubinsky, Ifju 1973), particularly from hardwoods, which can catalyse further decomposition.

Changes in, or loss of, hemicelluloses play key roles in the strength and stability of wood dried at high temperatures and of press-dried paper. The improved strength and stiffness properties of press-dried paper sheets are apparently related directly to the hemicelluloses content and location. It is important in this process that the peak temperatures are reached very quickly to allow the interaction of water and hemicelluloses to take place over the longest period of time available.

This facilitates flow of the hemicelluloses on the fibre surface and their softening in the cell wall to promote fibre flexibility (Horn 1979). The lignins do not have time to flow but the bonds with the hemicelluloses hinder subsequent cleavage with water and loss of paper strength (Byrd 1979).

It was found that a minimum heating period of 2 hours at 100 °C was required for significant plasticization, or lowering of the softening points of tightly sealed samples of green *P. radiata* (Hillis, Rozsa 1978), with other species, rapid pre-heating to 100 °C in less than 2 to 3 hours has been recommended, with the maintenance of a wet bulb depression of less than 5 °C (Sumi, McMillen 1979). In view of the potential degradation of wet wood by heat, the pre-steaming or pre-heating period should be kept to the minimum time required to achieve plasticization in the interior of the board.

Chemicals

As well as heating with steam, wood can be plasticized with urea and particularly with liquid ammonia (Schuerch 1964). Exposure to ammonia vapour under pressure has less effect than immersion in anhydrous ammonia, when changes in the crystalline state of the cellulose take place. Apparently, the vapour pressure of ammonia must reach a certain threshold for this change to occur (Bariska et al. 1969). At lower pressures, ammonia vapour adsorbs in, and reacts with, other components of the cell wall. Green wood requires less exposure to saturated ammonia vapour at room temperatures than dry wood to achieve a reduction in bending stiffness which apparently is temporary and most of the original stiffness is regained when water and ammonia are removed (Davidson, Baumgardt 1970). Oven-dry maple wood exposed to saturated anhydrous ammonia vapour at 25 °C for 1 to 2 days has a higher density and takes longer to regain moisture and swell than the original sample (Pollisco et al. 1971).

Impregnation with some other chemicals could adversely affect some wood properties. Changes in the hemicelluloses are catalysed by cupric (and to a lesser extent aluminium) salts (Stamm 1964; Back 1967), which promote the formation of carboxylic radicals, and the cross-linking of these groups is catalysed under acidic conditions. These covalent linkages may increase tensile strength but, together with the cleavage of cellulose chains, also increase brittleness with loss of toughness and abrasion resistance (Anderson, Back 1975; Back, Stenberg 1976, 1977; Seburg et al. 1953; Stamm 1964; Stenberg 1980).

Conclusions

The stability of wood is affected by several chemical, ultrastructural and physical features, when it is felled, dried and put into use under different conditions. Moreover, those features differ according to the age of the tree when the wood is formed or to the conditions under which the tree grows. The stability of undried wood can be modified by exposure to chemicals or improved by heating at elevated

temperatures for different periods of time. Similarities exist between the principles underlying preparation of stabilized press-dried paper and the high-temperature drying of wood.

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