Wood Modification Technologies

Principles, Sustainability, and the Need for Innovation

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Front cover: Lookout tower Pompejus in acetylated raiata pine, Fort De Roovere, Halsteren, The Netherlands.

The watchtower is also used as an open-air theatre, and an information point for tourists. The facade of the watchtower has been designed according to the principle of the Voronoi diagram, a mathematical design principle, which enables the triangles of the steel construction to be used to have windows and openings in the facade. By using this design principle, it allowed the facade to be divided into elements that could be prefabricated and transported, and at the same time, add an additional layer to the facade to break the dominance of the triangular steel structure.

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Preface

The forest sector and wood-based industries are challenged by changes in resource availability, energy supply and climate change. It is part of the continuing discussion between economics, ecology, and social welfare that can be summed up as sustainability. Wood is a natural, renewable, reusable and recyclable raw material that can play a major role in minimising the negative effects on the climate and environment, when it is sourced from sustainably managed forests. There is intense competition from the non-sustainably derived materials entering markets that have traditionally been dominated by timber products. Environmental concerns are leading to the phasing out of some traditional wood preservatives, which rely upon toxicity as their primary mode of action. Also, there are increasing quantities of plantation grown broad-leaved and conifer species being processed and such woods generally have inferior properties compared with timber sourced from natural forests. In order to maintain the competitiveness of wood, new approaches are needed. Research into wood modification is one way of meeting these challenges.

In his book *Wood Modification,* published in 2006, Professor Callum Hill gave a precise but also very general definition of the concept of wood modification:

> *Wood modification involves the action of a chemical, biological or physical agent upon the material, resulting in a desired property enhancement during the service life of the modified wood. The modified wood should itself be nontoxic under service conditions and, furthermore, there should be no release of any toxic substances during service life, or at the end of life following disposal or recycling of the modified wood. If the modification is intended for improved resistance to biological attack, then the mode of action should be non-biocidal (Hill, 2006, p. 20).*

This work, together with the work performed in the European Thematic Network for Wood Modification[1] culminating with its first conference in Ghent 2003 (Van Acker and Hill, 2003), brought together different research and industrial activities in Europe in the area of wood modification. For almost two decades, wood modification research activities have been increasing around Europe and beyond. Besides intensified research, there were also increased volumes and new products of modified wood on the market. Among these were thermally modified, furfurylated and acetylated timber. Several networking activities related to wood modification have also been executed. Specifically, two activities within the frame of COST—European Cooperation in Science and Technology—have been central: COST Action FP0904 "Thermo-hydro-mechanical wood behaviour and processing" that ran from 2010 to 2014, chaired by Professor Parviz Navi from Ecole Polytechnique Fédérale de Lausanne (EPFL, Switzerland) and vice chaired by Associate Professor Dennis Jones, and COST Action FP1407 "Understanding wood modification through an integrated scientific and environmental impact approach—ModWoodLife" running from 2015 to 2019, chaired by Professor Andreja Kutnar (University of Primorska, Slovenia) and vice chaired by Associate Professor Dennis Jones. Although a considerable amount of research and development activities have taken place in the past two decades, we are still just at the beginning of a new era regarding the use of modified wood. The volumes of modified wood in Europe, but also in the rest

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[1] A project entitled "Wood modification, the novel base, providing materials with superior qualities without toxic residue" funded by the European Commission, through its Fifth Framework programme.

of the world, is small compared to the volumes of wood products produced. A recent task within the framework of the COST Action FP1407 was to outline the current status of wood modification across Europe in terms of national inventories (Jones et al., 2019). Based on the reported production volumes and on subsequent investigations, it was estimated that in 2020 the annual production volume for modified wood in Europe will be slightly more than 700,000 m^3, which is, to say the least, a very modest volume.

However, modified wood, as defined by Hill, is not a new invention. Different modification methods can be traced back to ancient times (Navi and Sandberg, 2012; Ch. 2, Sandberg et al., 2017) and the industrial revolution, which resulted in highly industrialised production processes involving wood modification techniques for mass production of furniture, for example.

Wood modification is an all-encompassing term describing the application of chemical, mechanical, physical, or biological methods to alter the properties of the material. Such a definition of wood modification includes more or less everything that happens to the wood material after it has left the forest, and is more or less useless when one would like to simplify and give an overview of an area of interest. For this reason, the purpose of what would be achieved and the area in which the modifications are intended to be applied must be defined. This book focuses on industrial wood modification processes, i.e., processes that are applied on an industrial scale and deliver products to the market. Wood, is this case, is defined as solid wood and veneer, but of course, many of the processes described may also be applied to wood particles and fibres.

With this limitation in terms of wood modification, chemical treatments of wood that reach the core of cellulosic microfibrils, destroying the crystalline structure and eliminating most of the composite structure of wood, are in most cases, excluded from this book. Such subversive treatments radically modify the chemical components of the wood, and so the material produced lacks practically all the intrinsic characteristics of untreated natural wood. An example of a subversive treatment is the liquefaction of lignocelluloses (Jindal and Jha, 2016), typically to produce oil from biomass under very severe conditions.

There are already several good, fairly well-updated books in the field of wood modification, such as the already mentioned books by Hill, and by Navi and Sandberg, but also more chemistry-oriented presentations, such as *Chemical Modification of Wood* by a Professor at the University of Wisconsin (Madison, USA), Roger Rowell (Rowell, 2013). There are, however, only few, if any, overviews of the wood modification in the combined fields of sustainability, innovation, and industrialisation. This book aims to fill that gap.

The book is divided into six chapters. Chapter 1 gives an overview of wood modification with the aim of helping the reader to structure the field. A presentation of wood as a substrate for modification, and its response on a chemical and biological level, as well mechanical response upon loads before and after modification, is also presented within this chapter. For the reader that would need a deeper understanding in these fields, the chapter provides an extensive reference list of books covering the basic properties of wood as a material in detail. Wood modification has been divided into three main groups, each represented in their respective chapters (Chapters 2–4): (Chapter 2) chemical treatments, (Chapter 3) thermally-based treatments, and (Chapter 4) treatments with the use of electromagnetic irradiation, laser or plasma, for example. In Chapter 5, short presentations of different processes that are described in the literature or other places and that may be suitable to apply as wood modification processes are given. In general, these processes are not yet industrialised or may be well established in fields other than wood processing and modification. The intention with this chapter is to help the reader to sort out the potential of these processes, and find ways for further reading. The last chapter, *Modified wood beyond sustainability,* presents modified wood from a sustainability and circular economy perspective. Wood modification, its environmental impact and the use of modified wood in a healthy living environment are discussed. The potential role of wood and wood modification in achieving the ambitious targets of the European Green Deal are presented through discussion on recycling, up-cycling, the cradle to cradle paradigm, and end-of-life disposal options. The chapter concludes with the technical challenges identified in advancing Industry 4.0 in general and in wood modification processing.

The target audience for the book are students at high-school and university level, as well as researchers and people practiced in the industry. For the benefit of wood engineers and other people with an interest in this fascinating industry, we hope that the availability of this material as a printed book will provide an understanding of all the fundamentals involved in the processing of modified wood.

The authors hereby acknowledge COST—European Cooperation in Science and Technology—which enabled the authors of this book to meet many researchers from around Europe and develop great collaborations in the past several years. CT WOOD CoE at Luleå University of Technology and InnoReNew CoE are acknowledged for their financial support to the writing of this book. Furthermore, they give thanks to their supporters in the process of preparing this book, especially to Tatiana Abaurre Alencar Gavric, Gertrud Fábián and Chia-Feng Lin for helping them with the designs of the figures. A good graphic tells more than a hundred words.

Finally, they authors are dedicating this book to all young researchers from around the globe who are to take wood modification research to a new level, resulting in increased modified wood production and its use in the healthy built environments.

October 2020

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Contents

Chapter 1

Wood and Wood Modification

1.1 Wood modification

As a natural renewable resource, wood is generally a non-toxic, easily accessible and inexpensive biomass-derived material. Since ancient times, wood has been used by mankind due to its inherent properties, where a specific part of a tree of a particular species that could be found locally was utilised to achieve the best performance when used in construction, for different types of tools or for purposes not included in the practical tasks of life. Apart from drying, modification of timber has been rare in historical terms. Nevertheless, since wood is a natural product that originates from different individual trees, limits are imposed on its use, and the material may need to be transformed to acquire the desired functionality. This has become increasingly evident in the modern and highly industrial era. Modification is thus applied to overcome weaknesses in points of the wood material that are mainly related to moisture sensitivity, low dimensional stability, low hardness and wear resistance, low resistance to bio-deterioration against fungi, termites, marine borers, and low resistance to UV radiation.

Nowadays, wood modification is defined as a process adopted to improve the physical, mechanical, or aesthetic properties of sawn timber, veneer or wood particles used in the production of wood composites. This process produces a material that can be disposed of at the end of a product's life cycle without presenting any environmental hazards greater than those that are associated with the disposal of unmodified wood.

The wood modification industry is currently undergoing major developments, driven in part by environmental concerns regarding the use of wood treated with certain classes of preservatives. Several fairly new technologies, such as thermal modification, acetylation, furfurylation, and various impregnation processes, have been successfully introduced into the market and demonstrate the potential of these modern technologies.

The main reasons for the increased interest in wood modification during the last decades with regard to research, the industry, and society in general can be summarised as:

1) a change in wood properties as a result of changes in silvicultural practices and the ways of using wood,
2) awareness of the limited availability of rare species with outstanding properties for modern use, such as durability and appearance,
3) awareness and restrictions by law of the use of environmental non-friendly chemicals for increasing the durability and reducing the maintenance of wood products,
4) increased interest from the industry to add value to sustainably sourced local sawn timber and by-products from the sawmill and refining processes further along the value chain,

5) EU policies supporting the development of a sustainable society, and
6) the international concern for climate change and related activities mainly organised within the frame of the United Nations (UN), such as the Paris Agreement under the United Nations Framework Convention on Climate Change (UNFCCC).

What is wood modification?

Wood modification is an all-encompassing term describing the application of chemical, mechanical, physical, or biological methods to alter the properties of the material. This definition of wood modification includes almost everything that happens to the wood material after it has left the forest and is more or less useless for simplifying and giving an overview of an area of interest. For this reason, it is necessary to define the purpose and the area in which the modification is intended to be applied. Such a purpose may, of course, change over time.

In the field of wood technology, wood modification includes any method or process that tends towards a better performance of the wood, where the term *wood* refers to roundwood (e.g., round timber, logs or other log-like products such as pit props, pylons, etc.), hewn timber, sawn timber, veneer, strands, chips and other types of wood particles used in wood composite products. In modern wood technology, it has become desirable for the modified wood to be non-toxic in service and that disposal at the end of life does not result in the generation of any toxic residues. This means that chemical treatments of wood that reach the core of the cellulosic microfibrils, destroying the amorphous and crystalline structures and eliminating most of the composite structure of wood, are in most cases excluded. Such subversive treatments radically modify the chemical components of the wood, and the material produced consequently lacks practically all the intrinsic characteristics of untreated natural wood. An example of a subversive treatment is liquefaction of lignocelluloses (Yao et al., 1994), which is mainly adopted to produce oil from biomass under very severe conditions.

Hill (2006) has provided a well-accepted definition of wood modification: *"Wood modification involves the action of a chemical, biological or physical agent upon the material, resulting in a desired property enhancement during the service life of the modified wood. The modified wood should itself be non-toxic under service conditions, and furthermore, there should be no release of any toxic substances during service, or at end of life, following disposal or recycling of the modified wood. If the modification is intended for improved resistance to biological attack, then the mode of action should be non-biocidal"*.

It should be noted that the above does not necessarily exclude the use of a hazardous chemical in the preparation of modified wood, provided that no hazardous residues remain in the wood when the modification process is complete.

In this book, the focus is on methods that have introduced a modified solid wood or veneer product or are introduced in large-scale wood preservation projects. This means that processes that are producing or are very near to being involved in the production of modified products are dealt with. The book does not focus on the following areas, however some are briefly described in Chapter 5, and readers are referred to other sources for an introduction to the field:

- treatments aiming to improve wood properties such as fire/flame stability (cf. Lowden and Hull, 2013; Visakh and Arao, 2015),
- preservation of ancient small artefacts (cf. Unger et al., 2001),
- wood particles or disintegrated wood mixed with other polymeric material, such as wood-plastic composites (cf. Jawid et al., 2017), or
- modification and derivatisation of extensively mechanically and chemically degraded wood constituents (cf. Huang et al., 2019).

The modification of wood can involve active modification, which changes the chemical nature of the material, or passive modification, in which the properties are changes without any alteration in the chemistry of the material. Most active modification methods investigated to date have involved a chemical reaction with the cell-wall polymer hydroxyl groups. These hydroxyl groups play a key role in the wood-water interaction while simultaneously being the most reactive sites. In moist wood, the water molecules settle between the wood polymers, forming hydrogen bonds between the hydroxyl groups and individual water molecules. A change in the number of these water molecules results in shrinkage or swelling of the wood. All possible types of wood treatment affect the wood-water interaction mechanism. The main wood-treatment interaction mechanisms that may be responsible for new wood properties are summarised in Figure 1.1.

Several wood-treatment interaction mechanisms tend to occur at the same time. For example, in thermal modification, parts of the cell-wall polymers are altered, which may lead to cross-linking, reduction of hydroxyl groups, and undesired cleavage of the polymer chains.

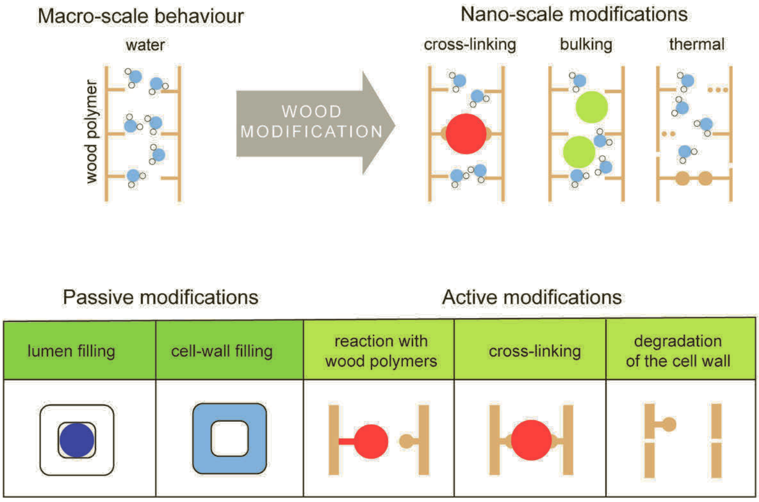

Figure 1.1 Schematic diagram illustrating the effect of active and passive modifications.

Most of the wood-modification processes that are being developed or under experimentation have full or partial origins in the pioneering research and seminal work of Alfred J. Stamm and his colleagues at the Forest Products Laboratory in Madison, Wisconsin, during the 1940s and 1950s (cf. Stamm, 1964). An early attempt to use chemical modification in industrial production was made by the Nobel family in their factory in St. Petersburg during the 1840s. To increase the durability and reduce the hygroscopicity of wood for wheel hubs, they impregnated the wood with a mixture of ferric sulphate and an acid, and dried it slowly in special boxes. Finally, they coated the hub with linseed oil and a varnish to further reduce moisture absorption. The trials were successful and they called the process "hardening wood". It was patented by Alfred Nobel's father Immanuel and his business partner Colonel Nikolai Aleksandrovich Ogarev, on 6 April 1844. The production was mechanised by a steam engine and 36 wheels a day were produced for the Russian army (Tolf, 1976; Meluna, 2009; Carlberg, 2019).

Wood modification processes[1]

To modify wood, four main types of process can be implemented: (1) chemical treatment, (2) thermally-based treatment, including thermo-hydro (TH) and thermo-hydro-mechanical (THM) treatment, (3) treatment with the use of electromagnetic irradiation, plasma or laser, and (4) other types of treatment, e.g., treatments based on biological processes (Figure 1.2).

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[1] In this book, the different processes are denoted by their scientific denominations. When a trademark of a process is used in the text, it is marked with the ™ sign in general, regardless if the trademark is registered or not.

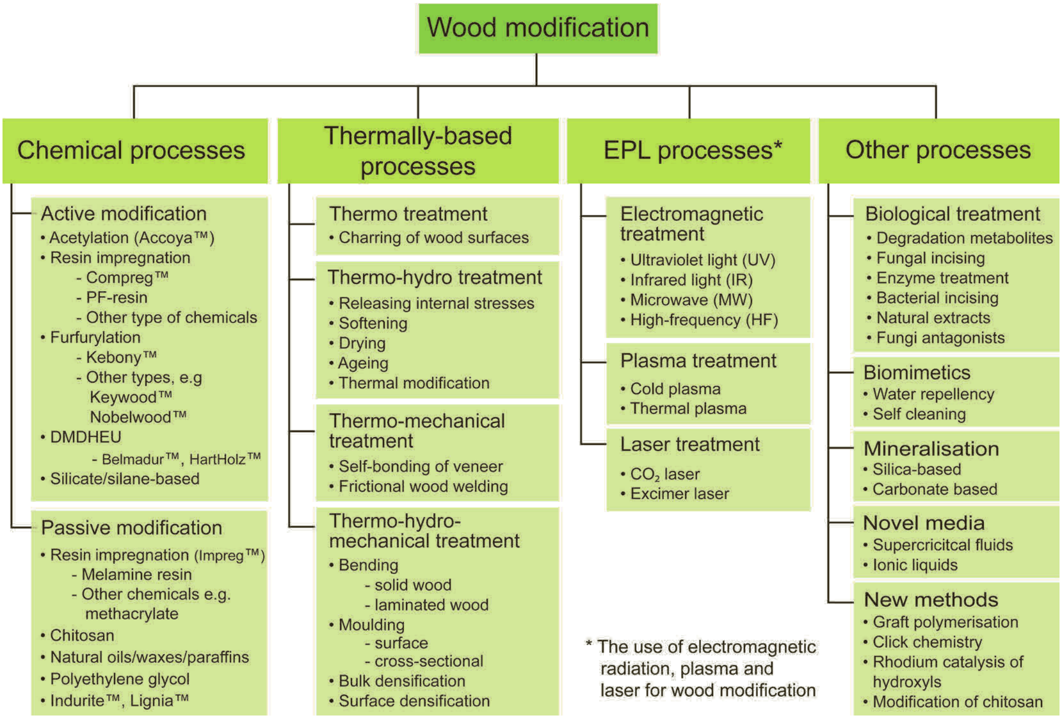

Figure 1.2 Classification of wood modification processes.

In the chemical modification of wood, a reagent reacts with a wood polymeric constituent (lignin, hemicelluloses, cellulose) to form a stable covalent bond between the reagent and the cell-wall polymer (Rowell, 1983). The reagent does not need to be environmental friendly itself, but the total process and the final product must be (i.e., the non-environmental reagent may just be used "indoors the process").

Chemical modification of wood can, therefore, in this sense be regarded as an active modification because it results in a chemical change in the cell-wall macromolecules. In this book, processes that only fill the lumen and/or cell wall are also included (Figure 1.1), as they follow the definition stated by Hill (2006). Currently, little is known about the general mode of action of chemically modified wood, but some hypotheses have been proposed by Hill (2006), specifically for biological degradation: (1) the equilibrium moisture content is lowered in modified wood, hence, it is more difficult for fungi to get the moisture required for growth and subsequent wood decay; (2) there is a physical blocking of the entrance of decay fungi to micro-pores in the cell walls; and/or (3) the action of specific enzymes is inhibited.

One group of emerging wood treatments involves the combined use of temperature and moisture through which force can be applied, i.e., thermo-hydro (TH) and thermo-hydro-mechanical (THM) processes (Navi and Sandberg 2012). In an orthodox definition, no additives are used in the processes except for water in combination with wood, heat, and, in THM, external forces to change wood properties or to shape the wood. Procedures including impregnation or gluing to lock a shape are usually also included in these modification processes.

In processes based on electromagnetic radiation, the aim will be to focus on processes that utilise the fact that wood at a high moisture content is capable of absorbing a large amount of electromagnetic energy. The amount of energy required to raise the temperature is determined by the specific heat capacity of the material. The specific heat capacity of wood is influenced by its moisture content, its dry density and the temperature. This type of modification process also includes changing the wood surfaces by laser or plasma treatment.

There is, of course, a wide range of processes which is not possible to describe in detail in this book, but which have been or may be of interest for the modification of wood. Some such processes are briefly described in Chapter 5, as a separate group of *other processes*. This chapter gives a short state-of-the-art description of processes that have been of interest in the wood industry but for some reason have not been further developed, or modification methods used (industrially or at least on a larger scale) in other industrial sectors than the wood sector (e.g., in the food or agricultural sectors, etc.) that may be of interest for the modification of wood.

The efficient modification of wood requires reactions at the molecular level and this requires an understanding of the fundamentals of organic chemistry which, together with other related areas of wood science, such as wood structure, wood-water relationship, wood biodegradation, weathering of wood, and stress-strain response of wood, are presented in this chapter.

1.2 Wood as a substrate for modification

A comprehensive knowledge of the characteristics of any material is essential for its optimal utilisation. This is especially true for wood because of its cellular nature, its complex cell-wall structure, and its variability. For the development and understanding of wood-modification processes, it is necessary to understand not only the principles of tree growth but also the macroscopic and microscopic features that determine wood quality in a broad sense. A wood modification process must, in general, be adapted to the species for which it is to be applied, and to the features and properties of the wood species in question.

Trees are major components of the biosphere and their wood is one of the most important renewable resources. Wood is the result of an evolutionary process over millions of years to perfectly meet three main functions: water transport, mechanical support, and storage of reserve nutrients for the living tree.

Wood is a material with a biological structure consisting of cellulose and lignin-based cells and the anatomy of a tree or a piece of wood requires a journey through several orders of magnitude. The structure of wood is multi-levelled and hierarchical, and it is possible to define more than ten different structural levels between the macroscopic (the trunk) and the molecular level, including cells and the grouping of cells into functional units and tissues of a characteristic size and shape. It is important to distinguish between the macro-structure, micro-structure and ultra-structure. The structure visible to the naked eye, or with a magnifying glass up to 10 times magnification is called the macro-structure, and macro-features such as grain and knots are readily apparent. A microscopic examination of the wood gives the overall information of the structure and the general character of the wood, showing the morphological characteristics of the woody plant. This is the most reliable method of wood identification, and microscopic examination is most important with conifers, where visual features of the wood are characterised more by similarities than differences. At the ultra-structure level, the cell-wall composition is in focus. Below this resolution lies the realm of individual chemicals. All these features must be considered when designing a wood modification process.

The tree

Everybody knows that a tree is a “large” plant with a stick (the trunk) in the middle to hold the crown with its foliage aloft. Trees generally produce wood during their growth, but do all trees produce wood? The answer is of a semantic nature and depends on how we define the concept of “a tree”, i.e., must the central stick be of wood? Trees are among the largest organisms that have ever lived. A redwood (giant sequoia) tree growing in North America can, for example, have a weight 10 times that of a full-grown blue whale, but there are also very small trees like the Artic willow that reach a height of only a few centimetres. Many trees grow big when conditions are favourable and stay small when they are not. Foresters and other people have insisted that plants with several supporting stems should be called shrubs, but in nature such a definition is not easily pinned down. For example, in Brazil one particular species grows single-trunked trees where there is good availability of water, e.g., along the riverbanks, but multi-stemmed short shrubs where it is drier (Tudge, 2005). Trees may grow from ground level with several solid trunks of equal magnitude and the size of, e.g., a Scots pine tree. To divide species into trees and shrubs may be practical, but it is not really a distinction based on genetic differences. Nature is not designed to be easy for biologists and foresters!

In fact, there are many lineages of trees, separate evolutionary lines that have nothing to do with each another except that they are all plants (Tudge, 2005). A tree is not a distinct category—it is just a way of being a plant. Trees have dominated dry land for over 300 million years, which is far longer than both mammals and dinosaurs, and today forests cover about a third of the world's dry land. Trees are extremely diverse, with more than 80,000 species all over the globe—some of them evergreen, while others are deciduous. The accepted definition of a tree is that it is a plant with a more or less permanent shoot system supported by a single *woody* trunk, i.e., a plant where the cambium cells have the ability to undergo *secondary thickening*, i.e., the ability to produce both xylem tissue (wood) and phloem (bark). By such a definition, true wood comes only from conifer and broad-leaved trees.

Thus, evolution has resulted in two categories of tree, conifers (softwood) and broad-leaved (hardwood) trees, both originating from the so-called seed plants (Figure 1.3). Many lineages of seed plants have appeared during the long time of evolution, and most have long been extinct. Timber merchants label all conifers *softwoods* and all broad-leaved trees *hardwoods*, even though some conifers are a lot harder than many hardwoods, and the softest woods of all are in fact hardwood.

More than 360 million years ago, the first plants that reproduced not by spores but by seeds appeared. Seed plants, to which all trees belong, are divided into two categories: gymnosperms, i.e., plants with naked seeds, and angiosperms, i.e., plants with covered seeds. The gymnosperms consist of conifers, cycads (fern plants) and their relatives, ginkgos, and gnetales. Today, only one species of ginkgo remains: the maidenhair tree in western China. The gnetales group consists of about 70 distinct species, but the phylogenetic[2] position of the group is uncertain. It was sometimes placed close to the angiosperms, but is today associated with conifers.

The angiosperms include all other flowering plants. In contrast to the gymnosperms, the majority of angiosperm species are not trees but herbs. They are further divided into two categories: dicotyledons (dicots) and monocotyledons (monocots). The broad-leaved trees belong within the dicotyledon category. The most important structural difference between dicotyledons and monocotyledons is that the monocots have no ability to undergo secondary thickening, which means that monocot plants that look like trees, such as bamboo, palm trees and banana plants, are by definition not regarded as true trees.

The most important structural difference between gymnosperms and angiosperm trees is in their xylem fibre structure. Gymnosperm wood has only one type of cell, called tracheids, which

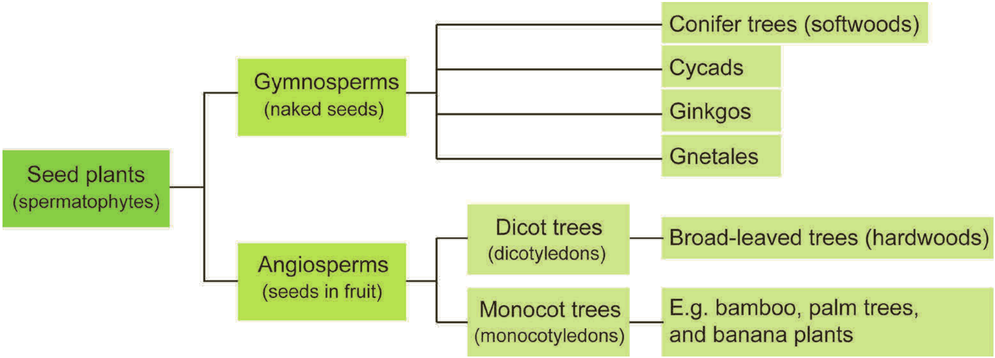

Figure 1.3 Conifer trees and broad-leaved trees are included in the botanical division of spermatophytes, i.e., seed plants.

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[2] Phylogenetics—the evolutionary history and relationships among individuals or groups of organisms, e.g., species, or populations.

transport water up to the leaves and strengthen the trunk. The angiosperm wood has two main types of xylem cell for these functions: vessels which pipe water through the trunk, and fibres (libriform fibres and fibre tracheids) for the strength of the trunk.

Conifer trees are characterised by their needle-like leaves, and such trees are commonly considered to be evergreens because most of them remain green all the year around and annually lose only a portion of their needles. Most conifers also bear scaly cones, inside which seeds are produced. In contrast to conifers, broad-leaved trees bear "broad leaves" which generally change colour and drop in the autumn in temperate zones, and produce seeds within acorns, pods, or other fruit bodies.

Only a small fraction of a tree's biomass is capable of producing the sugars it needs to grow, i.e., most of the energy created by photosynthesis in the leaves is used for building up xylem in the stem, branches and root system as the tree grows. This strategy has been shown to be very successful in competition with other plants for the light, water and nutrients needed for survival.

Trees compete for the "light"[3] by holding their canopy of leaves high above the ground, and the permanent structure (the trunk) above the ground makes trees successful in that sense compared to, for example, non-woody herbaceous plants which die back every year. The single stem of a tree makes it competitive also to plants which typically branch near the ground and so have several narrow stems rather than a single trunk, i.e., shrubs. As a group of trees grow in height, they cast such a dense shade by their collective canopy that most of other plants die out and the area will become a forest. The disadvantage of the single-trunk tree strategy is the high energetic cost for the construction of the tall trunk-branch system, resulting in a slow growth compared with that of herbaceous plants.

The structure of trees

The structure of wood is a result of the requirements of the living tree. Xylem is the part of the tree that in everyday speech is called wood and consists of hollow tubes (cells) with a length of a few millimetres connected to each other (Figure 1.4). The majority of these cells within the living tree are dead, i.e., the protoplasm is absent, leaving hollow cells with rigid walls. Protoplasm is the living content of a cell that is surrounded by a plasma membrane, i.e., a biological membrane that protects the cell from its environment. The only living cells in a tree are located in the cambium, in the sapwood rays, and in the inner bark (phloem). Their functions are related to the growth of the tree (cambium cells) and to the storage of the nutrients produced by photosynthesis.

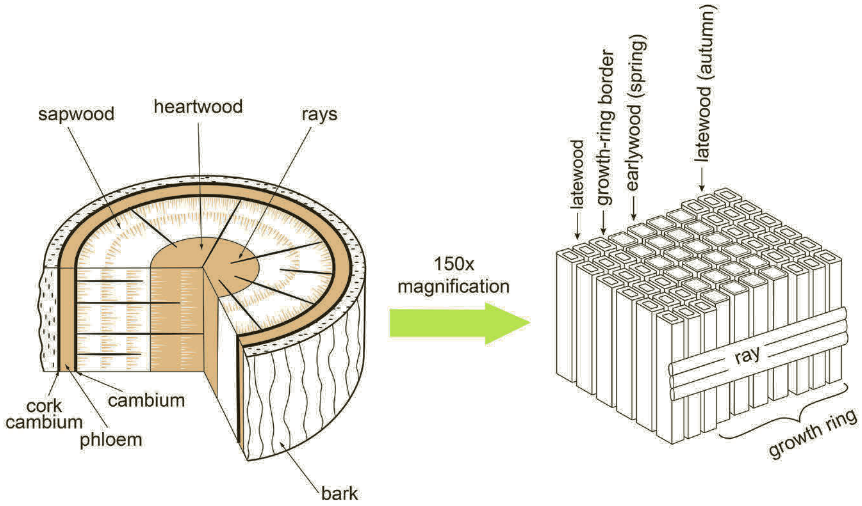

Figure 1.4 Major tissue types in the tree cross section (left), and enlargement of a growth ring of a typical conifer tree, showing the relative difference in size between earlywood and latewood cells (right).

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[3] Light is defined by CIE in relation to the sensitivity of the human eye to wavelengths between 400 and 700 nm.

The vascular cambium is the main growth tissue in the stem and roots of a tree, sheathing the xylem tissue from roots to leaves. It is a single cell layer that generates tissue on both the inside and outside—the process of *secondary thickening*—so that the tree grows thicker year by year, always with fresh xylem and phloem tissue coming on line and guaranteeing the function of the tree. The cambium lays down xylem on the inside to strengthen the stem and increase its water transport capability. On the outside, the cambium produces a thinner layer of phloem, a tissue that transports sugars created by photosynthesis in the leaves down to the roots for use in producing new cells and for storage in the trunk and bark nutrient-storing cells.

After cambial division, each successive xylem cell undergoes enlargement, wall thickening, and lignification. The cells lose their living cytoplasm and are left as dead cells of cellulose stiffened with lignin. The rate of cell division and the final size are thought to be largely influenced by growth-regulating hormones (auxins). As time passes, the xylem cells lose their function as the conducting tissue and the inner structural part loses its ability to transport water (heartwood formation). Xylem tissue is formed in an aqueous environment and exists in a living tree in the maximum swollen state—*the green state*.

The bark usually refers to tree tissue outside the cambium, but it can be simply divided into the inner bark (phloem) and the outer bark (cork). A disadvantage for the tree is having vital phloem tissue in the outer parts of the trunk where there is a greater risk of damage. The outer bark protects the tree from drying out and from extreme temperature fluctuations, mechanical injury, fire, etc. The outer bark is compounded from formerly functional phloem and custom-built cork, highly evolved and adapted. The oldest vessels of the phloem are crushed as new phloem tissue is laid down inside them, so that their functions become redundant. The crushed phloem is incorporated into the bark, providing essential protection. Many trees have a layer of secondary cambium (cork cambium) outside the principal cambium layer, with the specific task of producing cork. Cork cells (like xylem cells) are born to die, they finish up small, with thick, impermeable cell walls.

Within the cross section of a stem, there is often a visible difference in colour between two broad divisions known as sapwood and heartwood. The sapwood portion of the tree is physiologically active, i.e., the sapwood is involved to the normal functions of the living tree, and the ray cells of the sapwood are in continuous communication with the cambium and the inner living bark (Figure 1.4). The sapwood acts as a nutrient- and water-storage reservoir and provides the function of sap conduction. Heartwood is usually found in the centre lower portion of mature stems and all its cells are dead. At one time, heartwood was sapwood, but it no longer has a physiological function.

The water-transport system in a tree

The conduction role of the tree starts with taking up water and minerals from the ground by the root system and continues up the xylem tissue, i.e., through the roots, stem and branches, to the leaves, where photosynthesis takes place. Sugars produced by photosynthesis are in return transported down the tree along the phloem tissue. Some trees may also take up water from the air; the redwoods of California get about a third of their water from the morning fogs that sweep in from the Pacific.

Over 90% of the wood cells are arranged along the axis of the trunk or branches. In conifers, the vast majority of these cells are the long (up to 10 mm) and narrow (about 30 μm) tracheid cells, which have the dual functions of water conduction and supporting the stem (see Table 1.1 for proportions and functions of different cell types). This dual function makes the tracheid cell non-optimal in its function; the water-transport is improved by having large-diameter cells, i.e., wide cells, whereas strength is improved by having long and narrow cells. The shape of the cells is a compromise between these two. Broad-leaved trees have long, narrow fibres (libriform fibres and fibre tracheids) which support the trunk, but also very wide and thin-walled vessel cells, which only have the function of water transport (Figure 1.5). The larger diameter of the vessels dramatically decreases the resistance to water flow through them. Broad-leaved trees therefore have a much better water transport than conifers, even though the vessels constitute only a small proportion of the total number of cells.

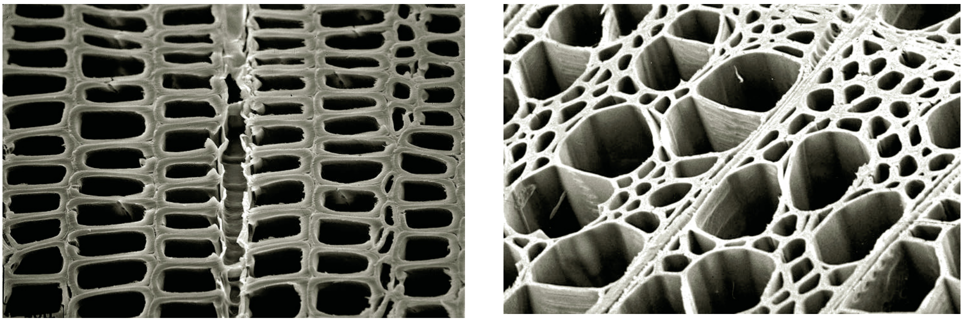

Figure 1.5 Cross-section view of tracheid cells in a conifer species, Swiss pine (left), and in a broad-leaved species, magnolia, containing both fibres and large-diameter vessels (right).

If a large air bubble gets into a wood vessel, the water column is broken by the tension in the water above and below it. The whole vessel then fills with air, forming what is called an embolism (Ennos, 2016). Once an embolism has formed, it is prevented from spreading through the whole length of the tree by sieve-like plates along the wood vessels which trap the air bubble. However, unless water is actively forced into the vessel again, the vessel remains empty and loses its conducting activity. Very dry conditions, but especially cold conditions increase the risk for embolism. Embolism is not common in conifers as the tracheid cells are much narrower than the vessels.

Regardless of the type of water-conducting cells in the tree, it is spectacular that water can rise from the root system to a height of 100 metres, to reach the leaves in their canopy. Several theories have been put forward over the years, such as the *positive root-pressure theory*, the *capillary force theory*, the *suction theory* and the *cohesion theory*. The positive root-pressure theory suggests that water is pumped up the trunk by the roots based on the osmosis effect, but it has only been possible to demonstrate this effect in birches and maple, and only in early spring (Ennos, 2016).

The capillary forces alone would be able to draw water to a height of only about 0.5 m in wood based on the dimensions of the different wood tissues, while the maximum height caused by the atmospheric pressure allows this capillary transfer to be only 10 metres or so. Cohesion theory, as defined by Boehm (1893) and Dixon and Joly (1894), is today the most accepted explanation for the water transport in trees. It is based on the fact that water is pulled up under tension when water is lost from the leaves by transpiration. If water is held in a narrow tube it can withstand large stretching forces without breaking. The cohesion-tension principles have been reviewed, particularly in terms of non-destructive testing (Bentrup, 2016). The strength of the water is due to cohesion between its molecules, and these forces can theoretically hold up a column of water nearly three kilometres high. These tensile forces acting upon the tree stem due to the water cohesion are sufficient to cause a measurable deformation in the shape of the stem.

The principal direction in trees and of wood

When discussing the structural features of wood, it is important to indicate which surface or direction is being referred to. Three distinctive planes exposing different views of the wood structure can be noted. A cut perpendicular to the longitudinal direction of stem is called a transverse or cross section, a cut in the radial plan is called a radial section and a longitudinal cut tangent to the growth rings is termed a tangential section. It is also necessary to distinguish between the three principal directions in wood; longitudinal or axial (L), radial (R) and tangential (T), which are the local directions responsible for wood anisotropy,[4] for its mechanical, physical and technological properties. Figure 1.6 illustrates schematically the principal axes and the corresponding radial, tangential and cross sections.

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[4] Anisotropy is the property of being directionally dependent, which implies different properties in different directions, as opposed to isotropy. It can be defined as a difference along different axes in a material's physical or mechanical properties.

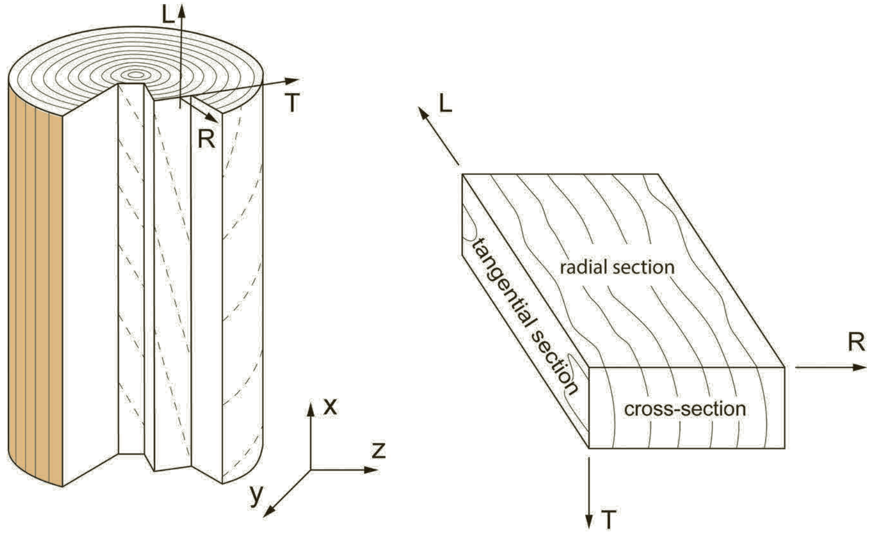


Figure 1.6 Schematic representation of the three principal axes and sections in wood. The stem section to the left show the principal axes, viz. longitudinal (L), radial (R) and tangential (T), and the rectangular piece of wood to the right shows the definition of the principal sections in sawn timber.

The macrostructure of wood

The main macroscopic parts of wood are, as shown in Figure 1.7:

- growth rings, earlywood and latewood,
- bark,
- pith,
- rays,
- resin canals of conifer wood,
- vessels of broad-leaved wood,
- sapwood and heartwood,
- knots,
- reaction wood,
- juvenile wood, and
- texture, colour and scent of wood.

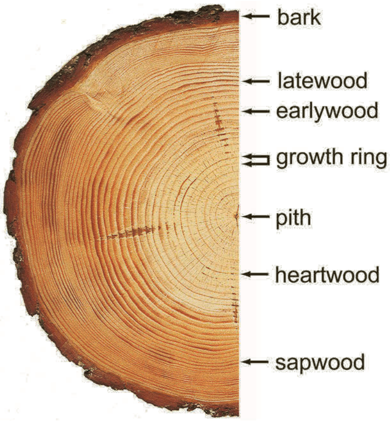


Figure 1.7 The macrostructure of wood visible in the cross section (un-dried disc) of Scots pine.

It is difficult to distinguish *juvenile wood* from *normal wood* by the naked eye, although it is here considered as a macroscopic feature due to its great importance for wood in use. For the same reason, features like texture, colour and scent of wood are briefly described in this section.

Growth rings, earlywood and latewood

In trees that grows seasonally, the addition of xylem and phloem is intermittent. In a typical tree growing in the temperate zone, the new xylem laid down in spring is wide but thin walled, while the later xylem is narrower but thick-walled. The difference can be seen in the cross section of a trunk as the presence of concentric layers. The layered arrangement of the xylem tissue in these growth rings is probably the most characteristic feature of wood. *False growth rings* may occur, and in some cases certain rings may be locally discontinuous. The growth rate and periodicity of trees growing in tropical forests—the forests located around the equator—are not the same as those of trees in temperate forests. Tropical trees in places where there are distinct wet and dry seasons also show growth rings, but trees growing where the climate is constant do not show growth rings. For these reasons, the term *growth ring* is preferred over *annual ring*.

In temperate regions, growth starts at the beginning of spring, continues in the summer and stops in the autumn. The part produced in the spring is called earlywood and that in the summer is called latewood. At the beginning of this vegetative growth, trees form a new layer of wood between the existing wood and the bark, the branches and the roots. The growth rings can often be easily distinguished because of differences in structure and colour between the earlywood and latewood. It is customary to divide the growth rings into three classes, conifers, ring-porous and diffuse-porous broad-leaved trees (Figure 1.8).

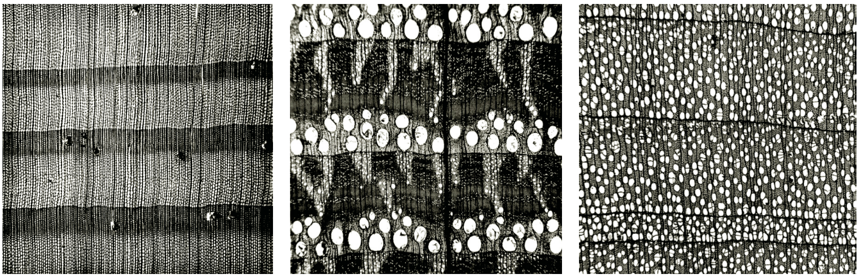


Figure 1.8 Cross-section view of a conifers species, Scots pine (left), a ring-porous broad-leaved species, oak (middle), and a diffuse-porous broad-leaved species, goat willow (right).

In temperate conifer woods, there is often a marked difference between latewood and earlywood (i.e., the growth-ring border), the latewood being denser than the earlywood, cf. Figure 1.9. The earlywood cells have thin walls (approximately 2 μm) and are mainly lumen, whereas the latewood cells have thicker walls (approximately 5 μm) with narrower lumen. The strength of the wood is in the walls, not the cavities. Hence, the greater the proportion of latewood, the greater is the density and strength. The width of a growth ring is less important for the density of a conifers as the proportion and nature of the latewood in the growth ring.

In contrast to the growth-ring border, the border between earlywood and latewood is diffuse, and the change in density and cell-wall thickness, for example, is gradual through this transition zone. There are numerous definitions to distinguish between earlywood and latewood, the most universally accepted definition being the one proposed by Mork (1928), who suggested that cells are classified as latewood when double the wall thickness is greater than the lumen diameter. Since then, more accurate definitions have been proposed, e.g., by Phillips et al. (1962) based on a β-particle method, and by Jagels and Dyer (1983) based on a digital image analysis of the shape of the cell cross section.

In ring-porous woods, each season's growth is always well defined, because the large vessels formed early in the season are on the denser tissue of the previous year, cf. Figure 1.8 middle.

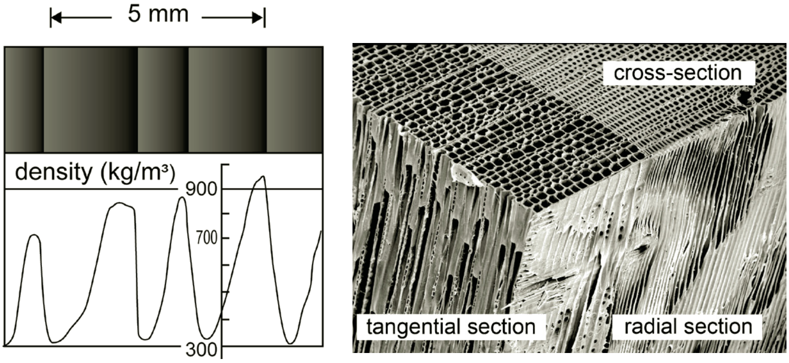


Figure 1.9 Density variation between earlywood and latewood in longleaf pine (left) after Phillips et al. (1962), and a 3D scanning electronic micrograph of Norway spruce (right).

In the case of the ring-porous broad-leaved woods, there seems to be a definite relationship between the rate of growth of timber and its properties. This may be briefly summed up in the general statement that the more rapid the growth or the wider the rings of growth, the heavier, harder, stronger, and stiffer is the wood. This, however, only applies to ring-porous woods, such as oak, ash, hickory, and others of the same group, and it is, of course, subject to exceptions and limitations.

In ring-porous woods of good growth, the thick-walled, strength-giving fibres are usually most abundant in the latewood. As the width of the ring diminishes, this latewood is reduced so that very slow growth produces relatively light, porous wood composed of thin-walled vessels and wood parenchyma. In good oak, these large vessels of the earlywood occupy 6 to 10% of the volume of the tree, whereas in inferior material they may make up 25% or more. The latewood of good oak is dark coloured, firm and consists mostly of thick-walled fibres which form one half or more of the wood. In inferior oak, this latewood is greatly reduced in both quantity and quality. Such a variation is largely due to the rate of growth.

In the diffuse-porous woods, the demarcation between growth rings is not always so clear and, in some cases, it is almost (if not completely) invisible to the naked eye. Conversely, when there is a clear demarcation there may be no noticeable difference in structure within the growth ring, cf. Figure 1.8 right.

In diffuse-porous woods, the vessels are uniform in size, so that the water-conducting capability is scattered throughout the ring instead of being concentrated in the earlywood. The effect of the rate of growth is not, therefore, the same as in the ring-porous woods, nearly approaching the conditions in the conifers. In general, it may be stated that woods give a stronger material when they grow at a medium rate than when they grow very rapidly or very slowly. In many uses of wood, total strength is not the main consideration. If ease of working is a requirement, wood should be chosen with regard to its uniformity of texture and straightness of grain, and this will, in most cases, occur when there is little contrast between the latewood of one season's growth and the earlywood of the next.

Radial growth begins first near the top of the tree and proceeds gradually downward in the stem, resulting in more earlywood and wider growth rings near the pith in the upper crown region. Transition to latewood occurs first near the base. Farthest from the source of the growth regulating hormones (auxins) and proceeds upwards. The density of an individual wood fibre is, therefore, determined by its position relative to the live crown and by the time of its formation.

Bark

The trunk has an outer covering, called bark, which protects the wood from extremes of temperature, drought, and mechanical injury. Bark constitutes, on average, about 10% of the volume of a tree, but this figure varies depending on tree species and age. The bark usually refers to tree tissues outside the cambium. It includes a number of different tissues, but bark can simply be divided into the inner bark (phloem and cork cambium) and the outer bark (cork layer). The relatively light-coloured inner bark is living tissue that conducts sugars downwards from the leaves. The dark-coloured and dry outer bark includes only dead tissue and is more or less impermeable to water and gases with an insulating function. The cell walls in the cork layer contain *suberin*, a waxy substance which protects the stem against water loss and the invasion of insects into the stem, and prevents infection by bacteria and fungal spores. The cork produced by the cork cambium is normally only one cell layer thick and it divides periclinally (parallel to the tissue surface) to the outside, producing cork. Like wood, bark is anisotropic with regard to dimensional stability and strength. Its thermal properties and heating value are similar to those of wood.

Pith

In the centre of the wood is the pith, which is formed during the first year of growth and becomes a storage area for impurities that are deposited from the active xylem during the growth of the tree. Pith consists of soft, spongy *parenchyma* cells, and is located in the centres of the stem, branches and roots. In some plants, the pith is solid, but in most cases it is soft. The shape of the pith varies between species and it varies in diameter from about 0.5 mm to 8 mm. Freshly grown pith in young shoots is typically white or pale brown, but it usually darkens with age. It may be inconspicuous, but it is always present at the centre of a trunk or branch. The roots have little or no pith and the anatomical structure is more variable.

Rays

Wood rays extend in the transverse direction from the bark toward the centre of the tree at a right angle to the growth rings. The first formed rays extend from the bark to the pith and are called primary rays, others extend from the bark to some later-formed growth ring outside the pith and are called secondary rays.

All the transverse cells found in any given wood are included in the wood rays, ribbon-like aggregates of horizontally oriented cells. The rays are formed by the cambium and extend in the radial direction in the xylem, cf. Figure 1.4. Rays consist of nutrient-storing cells and provide a route by which sap can be transported horizontally either to or from the inner bark (phloem).

Rays may contain ray parenchyma, ray tracheids and ray epithelial cells, but rays are usually composed predominantly of ray parenchyma cells, with ray tracheids forming one or more marginal rows of cells and an occasional row of cells in the body of a ray. When transverse resin canals are present, rows of epithelial cells and the resin canal cavity are also included in the ray.

The size of the rays is very different in different species. They can vary from being slightly visible to completely invisible to the naked eye. The variation between different species is great, and this means that they are useful for identification. In conifers, rays are usually one-cell or a maximum of two-cells wide in the tangential direction and 1 to 20 and sometimes up to 60 cells high. The rays in broad-leaved woods vary between one and several cells, depending on the species. These rays can always be observed in the tangential, radial and transverse sections.

Rays have a major influence on wood properties, not least the strength. The rays are the weakest zones in the wood, and they easily cause the wood to split. Rays are also one of the main causes of transverse hygroexpansion anisotropy, i.e., the rays restrain dimensional changes in the radial direction, and their presence is partially responsible for the fact that when wood is dried it shrinks less radially than tangentially. The mechanism of differential transverse hygroexpansion has been the subject of considerable controversy in the literature on wood science for many years, see for instance Skaar (1988).

Resin canals in conifer wood

A characteristic feature of some conifer woods is their resin content, which is often sufficient to give them a clear fragrance and make newly sawn timber sticky. Resin canals or resin ducts are tube-like intercellular spaces, which transport resin in both the longitudinal and horizontal directions

(Figure 1.10). The vertical and horizontal resin canals are interconnected and form a uniform network in the tree (Ilvessalo-Pfäffli, 1995). Transverse resin canals, that are located inside the rays, are seen in the tangential section. Resin is formed in epithelial parenchyma cells and can in some species be stored in special resin canal cavities, called resin pockets. These cells supply resin to the channels and pockets.

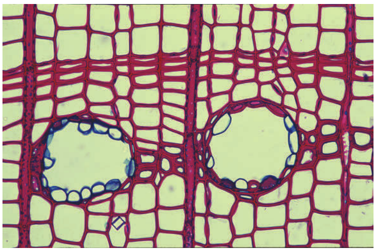


Figure 1.10 Cross-section view of two resin canals in Norway spruce.

Vessel elements in wood from broad-leaved trees

The fundamental anatomical difference between wood from conifer and wood from broad-leaved trees is that broad-leaved woods contain specialised conducting cells called vessel elements. These vessel elements are generally much larger in diameter than other types of longitudinal cells and the vessels are in general shorter than both broad-leaved and conifer fibres, cf. Table 1.1. A number of vessel elements are linked end-to-end along the stem to form long tube-like structures. Both the size and arrangement of the vessels in the cross-section of a wood sample are used to classify wood from different broad-leaved trees, cf. Figure 1.8.

Sapwood and heartwood

In most species, the difference in properties between sapwood and heartwood are substantial, especially regarding water and moisture transport, and they must be taken into consideration for most types of wood modification process. Some species, e.g., aspen, birch, beech, hornbeam and maple, do not normally develop heartwood, but these tree species may have discolouration around the pith due to microorganisms or frost, so-called *red core* or *false heartwood*. There are also differences in heartwood formation between species, especially between conifer and broad-leaved trees.

The sapwood is the outer, water-conducting part of the trunk that, in the living tree, contains living cells for the storage of reserve material (see rays). Young trees have only sapwood, but as they mature and no longer need the whole cross section of the xylem part of the trunk for fluid transport, they develop heartwood, i.e., the water-conducting function ceases, the remaining living wood cells die, and the cell walls are preserved and help to support the tree for many years to come. Heartwood is the inner and central part of the trunk, which, in the living tree, contains only dead and non-water-transporting cells and in which the reserve materials have been removed or converted into extractives. Heartwood can also be found in the roots of many species, especially in the region near the stem (Hillis, 1987). The new wood cells thus created are added to the sapwood, while the older cells adjacent to the heartwood gradually change to form new heartwood. The proportions of sapwood and heartwood vary according to species, the age of the tree, the position in the tree, the rate of growth, and the environment.

In some species, a zone usually comprising 1–3 growth rings can occur between the sapwood and the heartwood for a short period of time. This transition zone is described by Hillis (1987) as a narrow, pale-coloured zone surrounding some heartwood or injured regions, often containing living cells, usually devoid of starch, often impermeable to liquids, with a moisture content lower than that of the sapwood and sometimes also lower than that of the heartwood.

The volume percentage of living cells (parenchyma cells) in the sapwood varies between 5 and 40% of the total tissue volume (Hillis, 1987). The death of these cells and the transition of the sapwood to heartwood are accompanied by the secretion of oxidised phenols, which are often the origin of the pigmentation of heartwood. In trees in which heartwood and sapwood have the same colour, the death of these cells does not lead to pigmentation. The substances secreted by the trees are called extractives. They are typically toxic to wood-decaying organisms and help the wood to resist fungi and insects.

Figure 1.11 shows the difference in colour between sapwood and heartwood. Sapwood often has a clearer colour than the heartwood, but in many species this distinction between the sapwood and heartwood does not exist in colour but only in function and moisture content.

In contrast to heartwood, sapwood in the living tree has a very high moisture content. In Scots pine, there is also a large variation in moisture content in the sapwood between the earlywood and latewood, where the moisture is found mainly in the earlywood. In the heartwood, there is no difference in moisture content between earlywood and latewood (Figure 1.12).

During heartwood formation in a number of broad-leaved species, the vessels are filled with outgrowths of parenchyma cells called tyloses into the hollows of vessels, cf. Figure 1.13.

Tyloses are growths that partially or completely block the vessels in which they occur, a situation that can be either detrimental or beneficial depending upon the use to which the wood is put (Bosshard, 1974). The existence of tyloses in the heartwood vessels of white oak, and the relative

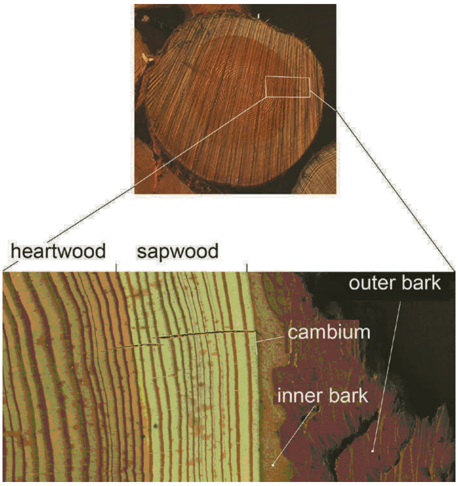


Figure 1.11 Cross section of a Scots pine log (upper) and a magnification of a portion of the same cross section, showing the inner bark, outer bark, cambium, and the sapwood/heartwood border (lower). The small dark dots in the lower image are resin, which has flowed out when resin canals were cut.

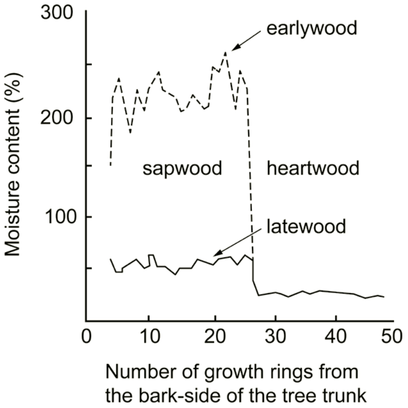


Figure 1.12 Moisture content of earlywood and latewood in sapwood and heartwood of Scots pine (Vintila 1939).

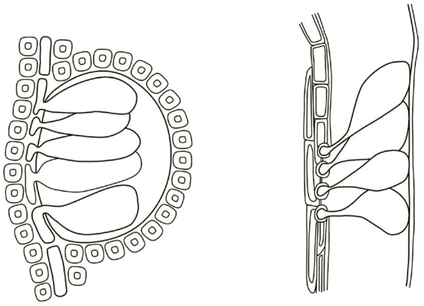


Figure 1.13 Tyloses in a broad-leaved tree vessel in transverse (left) and longitudinal (right) sections. The tyloses effectively prevent water transportation in the vessel.

lack of them in red oak, is the reason why white oak is preferred in the manufacture of barrels, casks and tanks for the storage of liquids. In contrast to this beneficial feature of tyloses, wood in which they are well developed may be difficult to dry or impregnate with chemicals (Bowyer et al., 2007).

Knots

As the trunk grows, old and new branches form junctions called knots. Where the cambium is alive at these points, there is a continuity of growth combined with a change in orientation and the knot is termed green or alive. On lower branches, the cambium is frequently dead, and the trunk grows around the branch enclosing its bark. These "black" or "dead" knots, are liable to fall out of sawn timber during sawing or during further processing. An important feature of knots is their deviant fibre orientation around and in the knot itself, which clearly affects both the appearance and properties of sawn timber.

Reaction wood

When a tree is growing on a sloping land surface or is exposed to a dominant wind direction, the load on the stem is unbalanced. The tree then starts to produce abnormal wood known as reaction wood to compensate for the unbalanced load. The formation of reaction wood is related to the process of straightening of leaning stems and the same happens in the branches and in the area where the branches join the stem.

Conifers and broad-leaved trees have adopted different strategies for the formation of reaction wood. The reaction wood of conifers is called *compression wood*, because it forms on the compression-stress side of leaning stems. In broad-leaved trees the reaction wood is called *tension wood* because the increased growth takes place on the upper or tension-stress side of the leaning tree, as shown in Figure 1.14.

In both conifer and broad-leaved trees, the wood formed on the side of the stem or branch opposite to the reaction wood is known as *opposite wood*, while that lying between the reaction wood and the opposite wood, is referred to as *lateral wood*. In comparison with wood production in a vertically growing stem with almost perfectly circular growth rings, compression wood and tension wood are usually produced in larger quantities, giving the stem a cam-shaped cross section with pronounced eccentricity with respect to the pith (Barnett and Jeronimidis, 2003).

Reaction wood has physical and mechanical properties different from those of normal wood and some of these properties are worth mentioning. The compressive strength of compression wood is greater than that of normal wood, however, compression wood is consequently very brittle. This brittleness can be a problem if the wood is subjected to bending. The tensile strength and Young's modulus of tension wood are greater than those of normal wood, and it also has a higher fracture toughness and impact resistance. Reaction woods have different shrinkage characteristics from those of the adjacent normal wood, due to a deviation in the micro-fibril orientation in the S_2 cell-wall layer, and this can result in warping and cracking of the wood during drying.

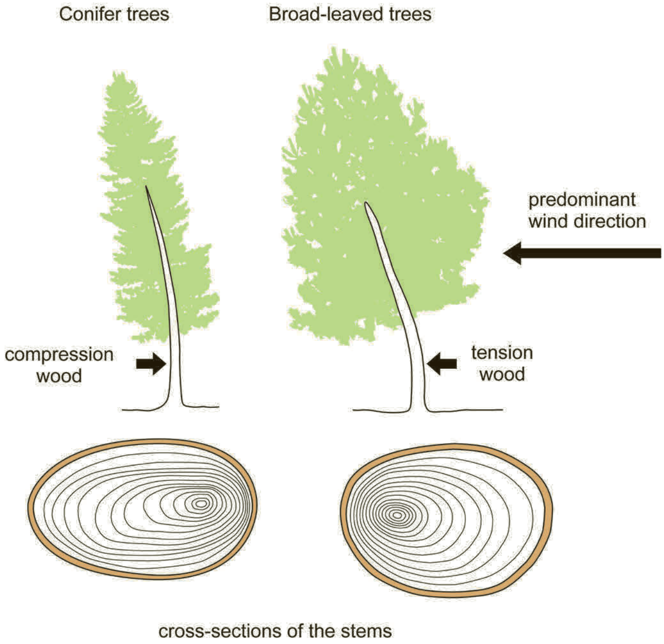


Figure 1.14 Formation of reaction wood in wood in conifer and broad-leaved trees as a result of a predominant wind direction over a long time.

Although it has not been generally recognised, reaction wood has many characteristics similar to those of juvenile wood (Zobel and Sprauge, 1998). In conifer wood, both juvenile wood and compression wood have short cells with flat micro-fibrillar angles and often a high lignin content; in broad-leaved woods, juvenile fibres of both the diffuse- and ring-porous wood types are short and broad-leaved woods have tension wood with a high cellulose content. When a tree is producing juvenile wood, it is especially susceptible to environmental forces that lead to the formation of reaction wood (Zobel and Sprauge, 1998).

Juvenile wood

Although it is not visible in the trunk cross-section, there is an important pith-to-bark gradient in density that is unique for each species. The fact that a relatively pronounced change in density often occurs in conifers during the first 15 to 30 years of growth gave rise to the term *juvenile wood* (Zobel and Sprauge, 1998). This term can lead to confusion, because this wood is found not just in young (juvenile) trees, but near the pith in every tree, regardless of age. However, the juvenile wood first laid down by the cambium near the centre of the tree has characteristics that differ from the wood formed at a large number of growth rings from the pith. This juvenile wood is sometimes referred to as *core-wood or crown-formed wood* and the mature wood as *outer-wood*. Although juvenile wood occurs in both conifer and broad-leaved trees, it is usually much less evident in broad-leaved woods. The source of juvenile wood is primarily in young plantations, thinnings, top wood, plywood cores and the harvesting of young stands.

Unlike heartwood that evolves in the lower parts of the trunk upwards, juvenile wood is formed nearest to the pith at all heights in young tress, and only in the top regions of mature trees (Figure 1.15). The most common characteristics used to identify the juvenile zone are density and cell length, although several other characteristics are also used. Each has a different curve of development from the pith outward so that the definition of the juvenile zone depends on the characteristic used. Bendtsen (1978) showed for hard pines (subgenus *Pinus*; soft pines has subgenus *Strobus*) that density, strength, cell length, cell-wall thickness, transverse shrinkage and latewood percentage increase from pith to bark, whereas the fibril angle, longitudinal shrinkage and moisture content

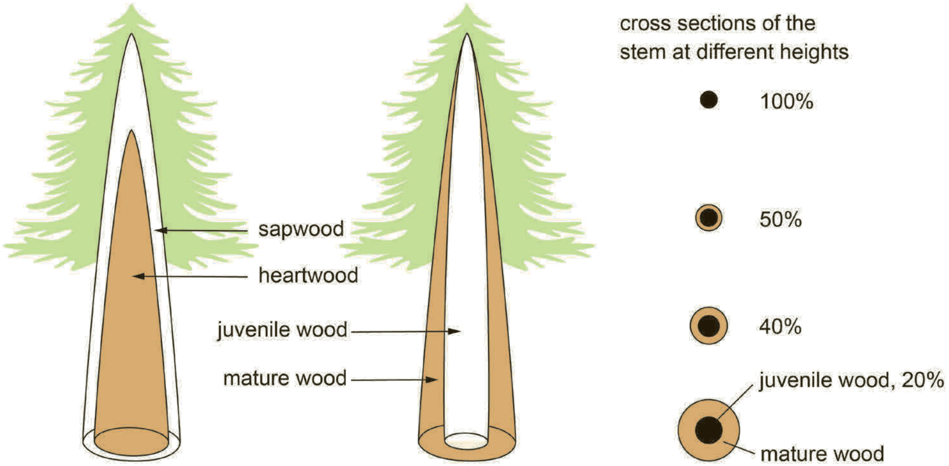


Figure 1.15 Juvenile wood occurs around the pith and roughly forms a cylinder up the tree. In contrast to heartwood, the proportion of juvenile wood increases towards the top of the tree.

of the same species decrease from pith to bark. Zobel and Talbert (1984) also conclude that the chemical composition of juvenile wood differs from that of mature wood. In most conifer woods, the lignin content is higher and the cellulose content lower in the juvenile wood. In wood from broad-leaved trees, Zobel and Talbert (1984) say that the proportion of and the chemical make-up of cellulose and lignin differ and that the holocellulose content is higher in juvenile wood than in mature wood.

The texture, colour and scent of wood

The texture of wood is material-dependent, i.e., the texture depends on the type of wood and on how the wood is built up. A piece of wood can show a great variation in hue depending, for example, on the type of wood, the content of extractive substances, heartwood or sapwood, and age. For most types of wood, the growth-ring orientation in the cross-section of the wood is important for the texture. A tangential surface with horizontal growth rings becomes mottled, whereas the radial surface with vertical growth rings has an even and harmonious pattern, as shown in Figure 1.16. Special patterns may result from uneven heartwood pigmentation, irregular growth-ring formation, deviations in cell and grain direction, or any combination of these.

Figure 1.16 Influence of growth-ring orientation on the texture of the flat-side surface of Scots pine tangential section with horizontal growth rings (left), and radial section with vertical growth rings (right).

Colour is one of the most conspicuous characteristics of wood and, although quite variable, it is one of the important features used in identification as well as adding aesthetic value. Basic wood substances, i.e., cellulose and lignin, have little colour of their own, so any distinctive colour is associated with heartwood (Hoadley, 1990). A dark colour always indicates heartwood, whereas a light colour can be either heartwood or sapwood. Some wood also undergoes considerable colour change with age or on exposure to UV-radiation.

Certain woods have distinctive odours. Many conifer woods, as well as numerous tropical woods, are known for their aromatic character. The odour is due to volatile extractives or resins in the wood.

The microstructure of wood

The cellular structure of wood is generally classified as a *microstructure* because the structural units are on a millimetre to micrometre scale (Table 1.1), and some type of magnifying tool, such as a microscope, is needed for the study.

Table 1.1 Density, dimensions and volume percentages of various cells in different tree species from the temperate zones (Fengel and Wegener, 1984).

	Conifer wood			**Broad-leaved wood**		
	Fir	**Nor. spruce**	**Scots pine**	**Eur. beech**	**Eur. oak**	**Poplar**
Density [kg/m^3]						
Minimum	320	300	300	490	390	
Average	410	430	490	680	650	400
Maximum	710	640	860	880	930	
Fibre[1] length (mm)						
Minimum	3.4	1.7	1.4	0.6	0.6	0.7
Average	4.3	2.9	3.1			
Maximum	4.6	3.7	4.4	1.3	1.6	1.6
Fibre[1] diameter (µm)						
Minimum	25	20	10	15	10	20
Average	50	30	30			
Maximum	65	40	50	20	30	40
Vessels length (mm)				0.3–0.7	0.1–0.4	0.5
Vessels diameter (µm)				5–100	10–400	20–150
Cell percentage (average values on volume)						
Tracheids	90	95	93	38	44/58	62
Vessels				31	40	27
Parenchyma	scarce	1.4–5.8	1.4–5.8	4.6	4.9	scarce
Ray cells	9.6	4.7	5.5	27	16.2/29.3	11.3

[1] Fibre – tracheids or libriform fibres. The following terminology misuse was pointed out by Zobel and Buijtenen (1989). The term “fibre” is commonly used for both the true fibres of broad-leaved woods and the tracheids of conifer woods. Although this is botanically incorrect, the general use of the term fibre must be recognised, since numerous publications refer to the fibre characteristics of conifer woods as well as to the real fibres of broad-leaved woods.

Microstructure of conifer wood

The wood of conifer trees consists of two types of cell: longitudinal tracheids and ray parenchyma, oriented both axially and horizontally. Most of the tracheids are longitudinal, while the parenchyma cells have a radial orientation. In addition to these two types of cell, other elements, such as epithelial cells, constitute longitudinal and horizontal resin canals. The transverse tracheids are not present in all species. The various types of conifer wood cells are presented in Table 1.2 and Figure 1.17.

The longitudinal or vertical tracheids constitute about 90–95% of the volume of conifer woods (Ilvessalo-Pfäffli, 1995). These are long, narrow cells with closed ends and bordered pits (Figure 1.18). The length of a tracheid varies from 2 to 6 mm and the width from 0.014 to 0.060 mm. The tracheids of latewood have a thick wall and a small lumen and are more suited to provide mechanical support than the tracheids of earlywood, whose function is mainly to conduct sap.

The length of a longitudinal tracheid, which is a closed unit, is very small compared to the height of the tree. To ensure the conduction of sap within the tree, it is thus necessary for each

Table 1.2 Different cell types and their functions in conifer wood.

Cell type	Function
Longitudinal tracheids	Support, conduction
Parenchyma	
Ray parenchyma	Storage
Longitudinal parenchyma	Storage
Epithelial parenchyma	Secretion of resin
Short tracheids	
Ray tracheids	Conduction
Strand tracheids	Conduction

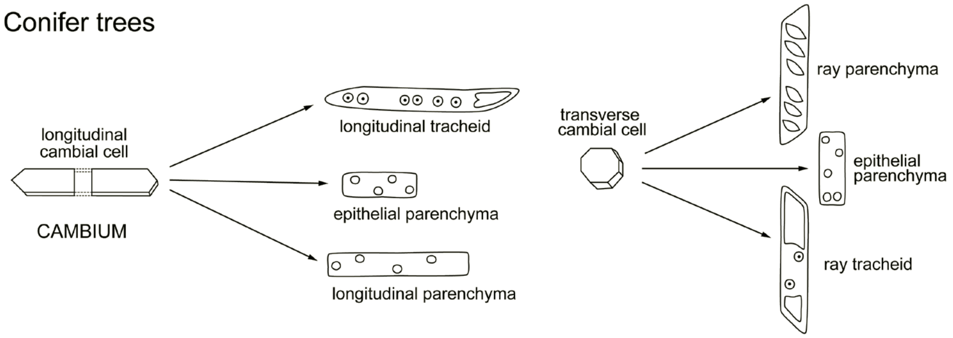


Figure 1.17 Diagram showing the anatomical elements in wood from conifer trees.

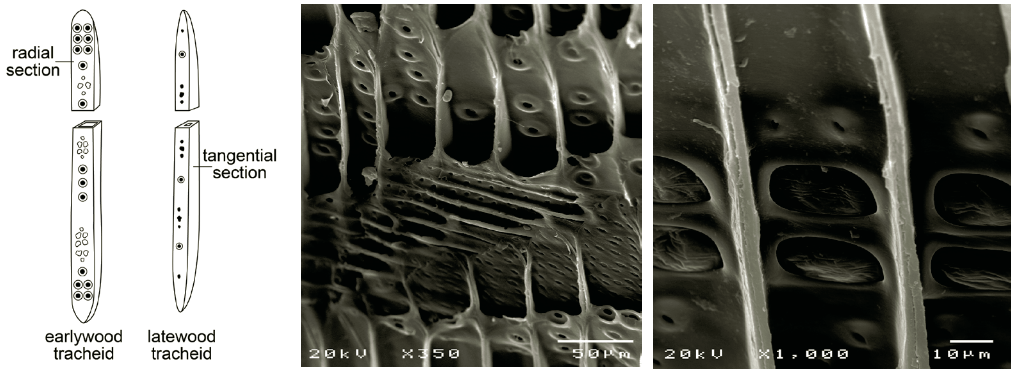


Figure 1.18 Diagrammatic representation of an earlywood and a latewood tracheid (left), and micrographs of the radial walls of Scots pine tracheids presenting bordered pits (middle), and (to the right) simple pits between tracheids and ray parenchyma cells (in the centre), and small bordered pits between tracheids and ray tracheids (above and below).

tracheid to be functionally connected to other tracheids. The conduction between tracheids, in both the lateral and vertical directions, takes place through pits, most of which are located in the radial walls. Pits also exist in the tangential walls of the tracheids, but they are much less numerous.

The pits of earlywood tracheids are large and circular, averaging about 200 pits per tracheid, whereas latewood tracheids have rather small, slit-like pits, and only 10 to 50 per tracheid (Trendelenburg and Mayer-Wegelin, 1955).

Pits have two essential parts, the pit *cavity* and the pit *membrane*, the cavity being open internally towards the lumen of the cell and closed by the pit membrane. Pits are of many shapes and sizes, but they are generally reduced to two basic types based on the shape of the cavity, viz. the *simple pit* and the *bordered pit* (Ilvessalo-Pfäffli, 1995).

In the simple pit, the cavity is almost straight-walled and only gradually widens or narrows toward the cell lumen. The lumen end of the cavity is known as the pit aperture. In the bordered pit, the cavity is constricted towards the lumen, forming a dome-shaped chamber, which is overarched by the pit border. The pits of adjacent cells are usually paired, forming three types of pairs (Figure 1.19).

A *simple pit pair* consist of two simple pits between parenchyma cells and in broad-leaved woods also between vessel elements and parenchyma cells. A *bordered pit pair* consists of two bordered pits between tracheids in conifers and between vessel elements in broad-leaved woods. A *half-bordered pit pair* consists of a bordered pit and a simple pit in the contact zone between the longitudinal tracheids and the rays and is, therefore, also called a *cross-field pit*. Their size, shape, and arrangement vary according to species and cross-field pitting is the most important feature in the identification of conifer species on a micro-structural level. Half-bordered pit pairs are also found between a vessel element and a parenchyma cell in broad-leaved woods.

In the bordered pit pairs of most conifer woods, the membrane has a thickening in the central zone called the *torus*, which is somewhat larger in diameter than the aperture and is impermeable to water. The membrane around the torus, the *margo*, is porous (Figure 1.20). When the torus is pressed against one of the apertures, the passage of water is prevented. The result of this phenomenon is called an *aspirated pit*, and it occurs when sapwood is transformed into heartwood or when the wood is dried. In heartwood, the pits are definitively blocked in this position (Figure 1.21).

The rays consist of radially oriented, brick-like and thin-walled parenchyma cells. The rays of conifers are composed either of parenchyma cells alone or of parenchyma cells and ray tracheids. In conifer trees, less than 25 parenchyma cells usually pile up to form a ray.

The ray tracheids are about the same size as the ray parenchyma cells. They are dead cells with small bordered pits leading to other ray cells and to longitudinal tracheids. The ray tracheids seem to be functionally limited to the occurrence of resin canals. Their functions are conduction, and the accumulation and storage of water and other substances in the radial direction.

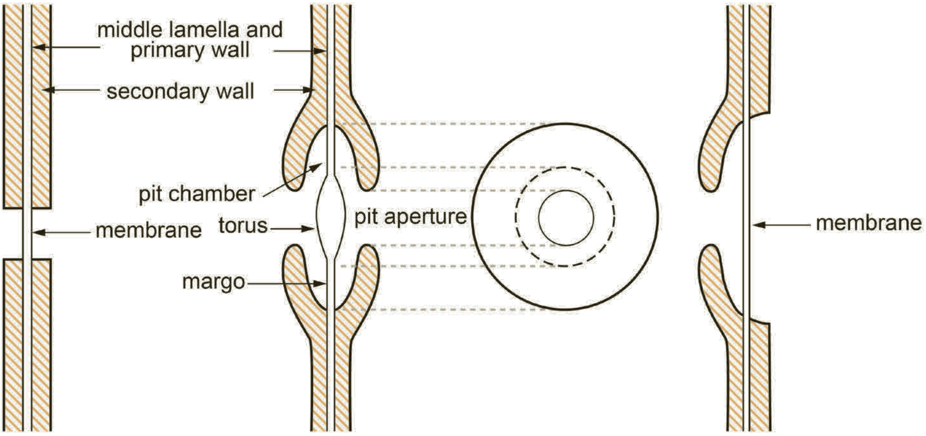


Figure 1.19 Three types of pit pair; simple pit pair (left), bordered pit pair (middle), and semi-bordered pit pair (right).

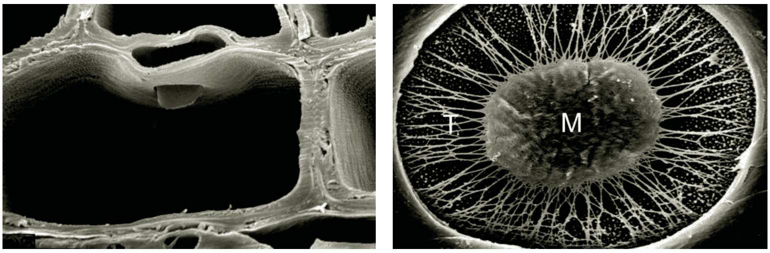


Figure 1.20 Cross-section view through a cell wall of European silver fir containing a pit (left), and membrane of a bordered pit showing the torus (T) and the margo (M) through which water passes from one cell to the next (right).

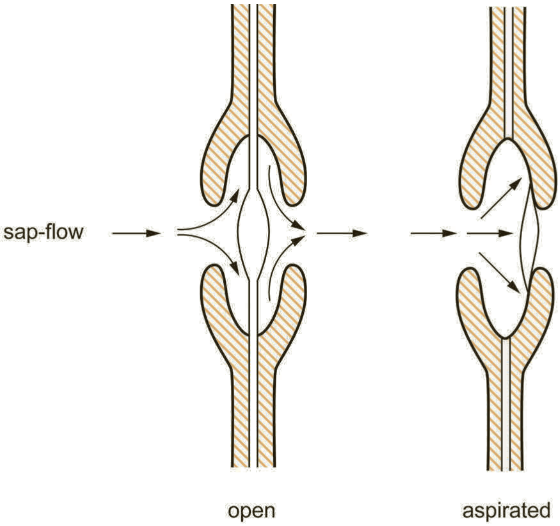


Figure 1.21 Aspiration of pits is involved in heartwood formation in conifers and may also occur when drying the sapwood.

Microstructure of wood from broad-leaved trees

The structure of broad-leaved wood is more complex than that of conifer wood and, during their evolution, broad-leaved wood have developed special types of cell from the tracheid: vessel elements for conduction and fibres for support. Wood from broad-leaved trees is made up of various types of cells which are very variable in dimension and form. The different types of cells constituting broad-leaved timber are presented in Table 1.3 and in Figure 1.22. Practically all broad-leaved

Table 1.3 Different cell types and their functions in wood from broad-leaved trees.

Cell type	Function
Vessel elements	Conduction
Fibres:	
Libriform fibres	Support
Fibre tracheids	Support
Parenchyma:	
Ray parenchyma	Storage
Longitudinal parenchyma	Storage
Tracheids:	
Vascular tracheids	Conduction
Vasicentric tracheids	Conduction

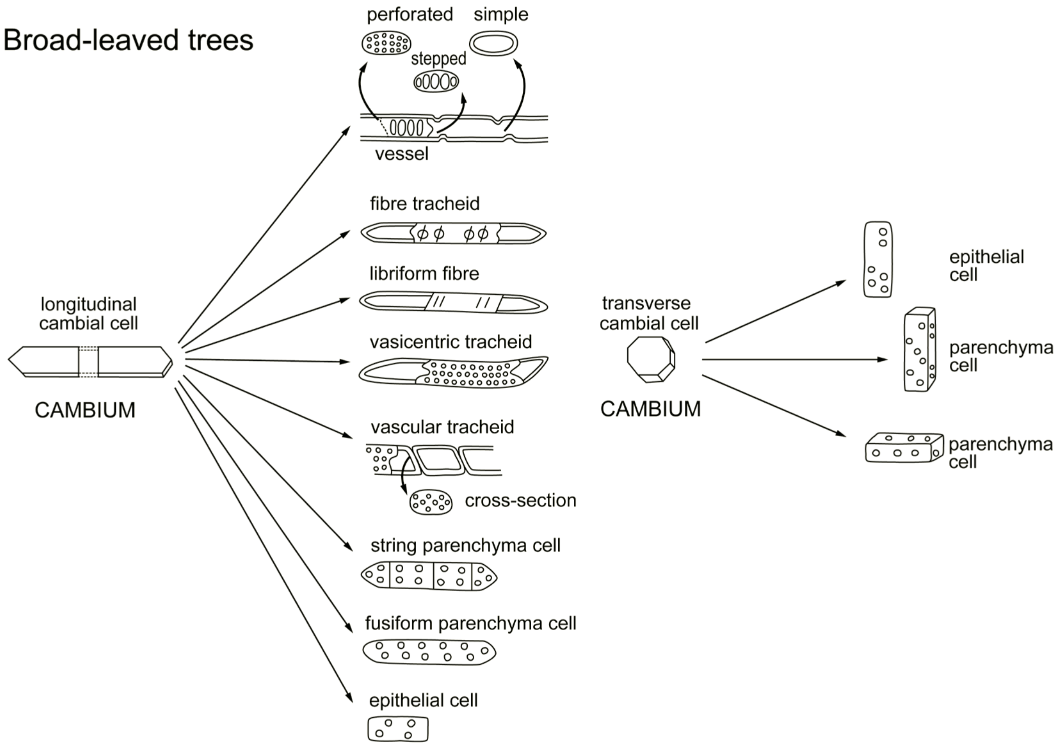


Figure 1.22 Diagram showing the anatomical elements in wood from broad-leaved trees.

wood contains longitudinal vessels, longitudinal fibres, and longitudinal parenchyma cell, as well as ray parenchyma cells. The broad-leaved wood rays, unlike those in conifers, consist exclusively of parenchyma cells.

Longitudinal cells

The longitudinal cells in broad-leaved trees consist of vessels, the tracheids, axial fibres, and axial parenchyma (Figure 1.22).

A vessel is a tube made of successive cell elements connected to form long continuous tubes in the tree. The volume of vessels in broad-leaved timber varies between 6 and 55%. Their diameter varies between 20 and 300 µm, and the passage of sap in the longitudinal direction is made possible by wide openings (perforations) at each end of the vessel elements. In addition, water and sap can transfer to adjacent vessels laterally through small pits in the vessel walls. The pits connecting two laterally adjacent vessels are different from the bordered pits, since they are primarily simple pits without a "torus" (Figure 1.19 left).

The fibres

The role of longitudinal cells is to provide mechanical support for the wood. They are long cells with thick and rigid walls varying between 0.8 and 2.3 mm. In wood from broad-leaved trees, the volume percentage of the fibres varies between 25 and 75%. Figure 1.23 shows a micrograph of wood from a broad-leaved tree with fibres, vessels and rays.

Wood from broad-leaved trees usually contains a greater volume percentage of longitudinal parenchyma than conifer wood. These cells fulfil a storage function for nutrients. The rays in broad-leaved woods consist of two or up to 40 radial cells in height, one to more than 20 in width, and sometimes in so great a number that the rays are visible to the naked eye.

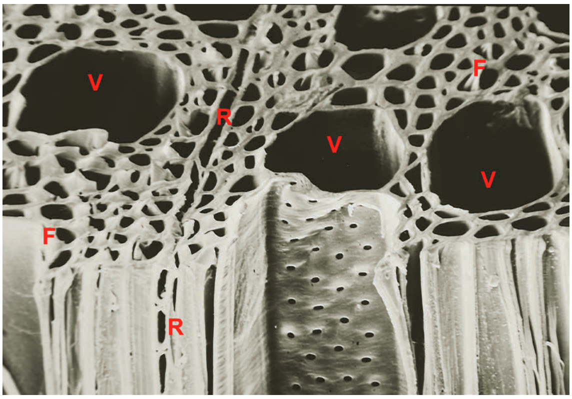


Figure 1.23 3D SEM micrograph of a broad-leaved wood structure showing vessels (V), fibres (F) and rays (R).

In summary, wood from broad-leaved trees is characterised by the presence of vessels, tracheids, fibres, longitudinal parenchyma and ray parenchyma. The vessels fulfil the role of conduction and the fibres with their thick walls ensure the flexible rigidity and mechanical support of the tree and often constitute most of wood volume, up to 60%. The radial and longitudinal parenchyma cells ensure that there is a reserve of nutrient substances in the tree.

Wood cell-wall structure and ultrastructure

With the help of a polarised, optical or electronic microscope, the various layers which form the cell wall can be observed. This wall consists of the primary wall (P) and the secondary wall (S). The *middle lamella* (M) is not an integral part of the cell wall but it interconnects the cells. The *middle lamella* and primary wall are however frequently treated as a single entity called the *compound middle lamella*. Figure 1.24 shows a representation of a segment of a longitudinal cell (tracheid) surrounded by other cells. The secondary wall is made up of three distinct layers: S_1, S_2 and S_3.

The middle lamella appears after the division of cambial cells and varies between 0.5 μm and 1.5 μm in thickness. The optical microscope shows the existence of an important quantity of lignin in this layer. This layer joins the cells together. To separate the cells (e.g., for anatomical study or for the manufacture of paper pulp), techniques involving maceration or chemical attack are used. These destroy the middle lamella and allow cells to be separated.

The primary wall is very thin and measures approximately 0.1 μm in thickness. Like the middle lamella, it contains a large quantity of lignin, but very little cellulose. It is often difficult to distinguish the primary wall from the middle lamella.

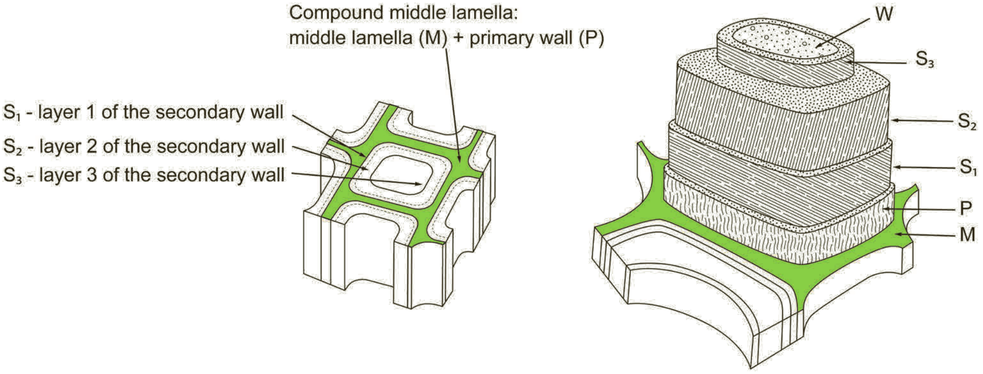


Figure 1.24 Diagrammatic representation of a longitudinal cell (tracheid) surrounded by six other cells (left), and representation of the various layers of the cell wall (right). M: middle lamella, P: primary wall, S: secondary wall with its S_1, S_2 and S_3 layers, W: warts layer.

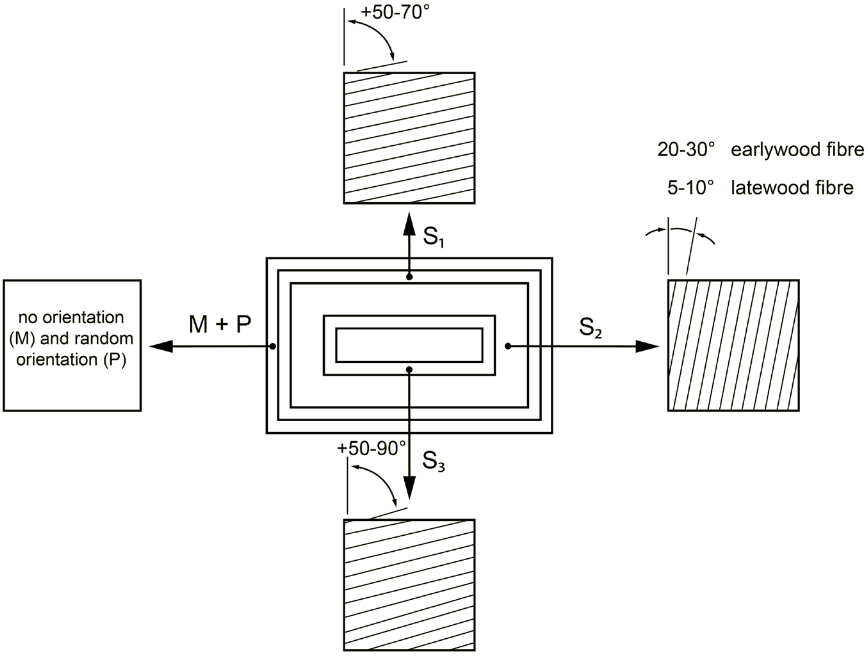


Figure 1.25 Schematic diagram of the microfibril-angle arrangements within the S_1, S_2 and S_3 layers. The angles are given with the longitudinal axis of the fibre as a reference.

A polarising microscope reveals that the secondary wall is made up of three layers (S_1, S_2 and S_3). In the latewood, the S_2 layer is the thickest part of the tracheid wall and there is little difference in thickness between the S_1 and S_3 layers. These layers consist of cellulose fibrils and microfibrils of different angles to the fibre axis.

The microfibril angle of the S_2 layer ranges between 5–10° (latewood) and 20–30° (earlywood), that of the S_1 between 50 and 70° and that of the S_3 layer between 50 and 90° (Figure 1.25). These layers, on the other hand, consist of concentric parallel laminae. The S_2 layer consists of 30 to 40 laminae in the cells of earlywood and more than 150 laminae in those of latewood. The S_2 layer is significantly thicker than its neighbours and, hence, contributes in a dominant way to the mechanical and physical properties of the cell wall.

1.3 The chemical composition of wood

Having established the basic structures within wood, it is necessary to consider their chemical composition. Wood consists mostly of carbon (50−53% in conifer wood and 47−50% in wood from broad-leaved trees) and oxygen (40−44%) together with 6% hydrogen, small amounts of nitrogen and other elements bonded together forming compounds with different elemental compositions, structures and weights (Tillman et al., 1981).

The main chemical components of wood are *cellulose, hemicelluloses* and *lignin*. In addition, there are other components called *extractives*, which, e.g., are deposited in the cell wall during the formation of heartwood. Table 1.4 gives the volumetric percentage of each chemical component, and its polymeric nature, degree of polymerisation and function, and Figure 1.26 shows their variation within different layers of the cell wall.

Brief introduction to covalent bonds

In any chemical compound, atoms strive towards distribution of valence (outermost) electrons corresponding to a stable noble gas state. Noble gases in group 8A in the periodic table of chemical

Table 1.4 Chemical components of wood, their polymeric natures and functions.

Component	Composition (% volume)	Polymeric nature	Degree of polymerisation	Basic monomer	Function
Cellulose	45–50	Linear molecule semi-crystalline	5,000–10,000	Glucose	Fibre
Hemicelluloses	20–25	Ramified amorphous molecule	150–200	Essential sugars, excluding glucose	Matrix
Lignin	20–30	Three-dimensional amorphous-bonded	-	Phenylpropane	Matrix
Extractives	0–10	Polymerised molecule	-	Polyphenol	A protection element
Ash	0–5	Minerals	-	-	-

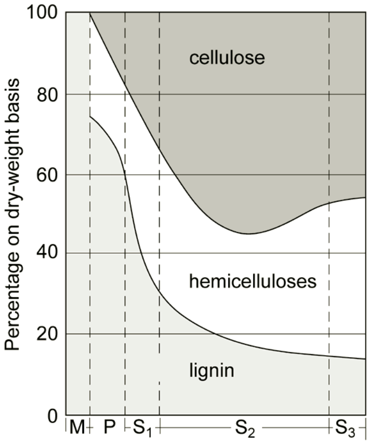
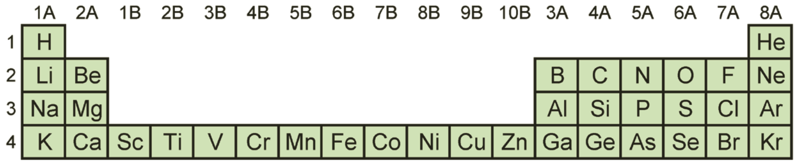


Figure 1.26 Distribution of cellulose, lignin and hemicelluloses within the cell-wall layers (after Panshin et al., 1964). For details of the different cell-wall layers, see Figure 1.24.

elements have this stable distribution of valence electrons and do not normally form chemical compounds (Figure 1.27). Most other atoms are satisfied with eight valence electrons and only the small hydrogen atom is satisfied with two valence electrons. Atoms in the upper left of the periodic table (Figure 1.27) are the most electropositive and wish to get rid of (donate) electrons, whereas those in the upper right corner are the most electronegative and wish take up (accept) electrons (except noble gases).

The position in the vertical group tells us how many electrons an atom can take up to achieve noble gas character; elements in groups 1A, 2A, 3A may lose one, two and three electrons, respectively, whereas elements in group 5A, 6A and 7A may take up three, two and one electrons, respectively. Adding electrons to such electronegative atom or withdrawal of electrons from electropositive atom leads to the formation of negative anions and positive cations, respectively. Ions with different charges are strongly attached to each other by electrostatic forces forming ionic bonds without sharing electrons, like the sodium cation (Na^+) and chloride anion (Cl^-) in table salt



	1A	2A	1B	2B	3B	4B	5B	6B	7B	8B	9B	10B	3A	4A	5A	6A	7A	8A
1	H																	He
2	Li	Be											B	C	N	O	F	Ne
3	Na	Mg											Al	Si	P	S	Cl	Ar
4	K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr

Figure 1.27 Selected parts of the periodic table.

(NaCl). Since a carbon atom has four valence electrons (group 4A in the periodic table) and is neither electropositive nor electronegative, it can achieve noble gas character instead by sharing electron pairs with at most four atoms, whereas the small hydrogen atom (group 1A in the periodic table) can bind only one atom. Methane (CH_4) is the simplest stable organic molecule (neither electronegative nor electropositive) in which carbon is covalently bonded to four hydrogen atoms. Oxygen has six valence electrons (group 6A in the periodic table) and can share electron pairs with at most two atoms, which complete the octet of electrons in an oxygen atom. Multiple covalent bonds are not planar but tetrahedral to achieve a molecule with the lowest energy, so that molecules with such bonds have a three-dimensional structure. Single bonds are written as straight lines but to be able to draw such structures on paper, it is customary to print bonds pointing out of the paper plane as bold and those pointing back into the paper as dashed. Those in the plane of paper are printed as solid lines (Figure 1.28).

Single bonds such as the carbon-oxygen bond in the alcohol in Figure 1.28 can rotate. The hydrogen bonded to the oxygen atom can, therefore, point in the direction judged by the extent of rotation of the C-O bond. Rotation can, however, be restricted in more complicated structures due to interactions with other atoms within or between other molecules and rotation does not normally occur in double-bonded structures, as will be discussed later.

In dimethyl ether (CH_3-O-CH_3), the two carbon substituents called methyl groups (CH_3) are bonded covalently to the electronegative oxygen. The difference in electronegativity between those constituents is not so high that the bonding electron is not completely withdrawn towards the oxygen atom forming ions, but only polarises the bond so that the oxygen is partly negatively charged and the two carbon atoms partly positively charged (Figure 1.29). This leads to the formation of a dipole so that the angle between the two C-O bonds is tetrahedral, meaning that the dimethyl ether molecule has a net polar character. Note that compounds like tetrachloromethane are non-polar as the centre of gravity of charges coalesce at the same spot.

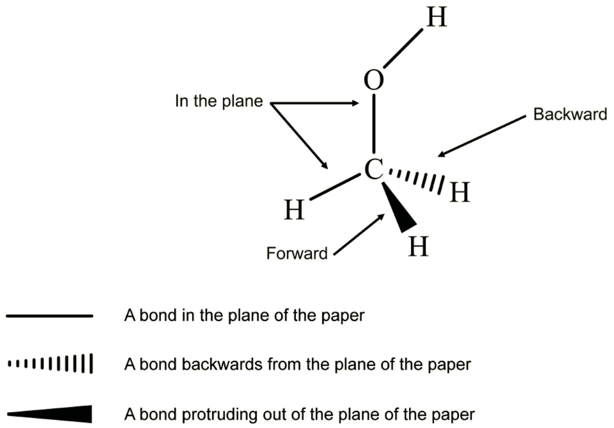


Figure 1.28 Drawing of molecules illustrating the three-dimensional structure exemplified with methanol.

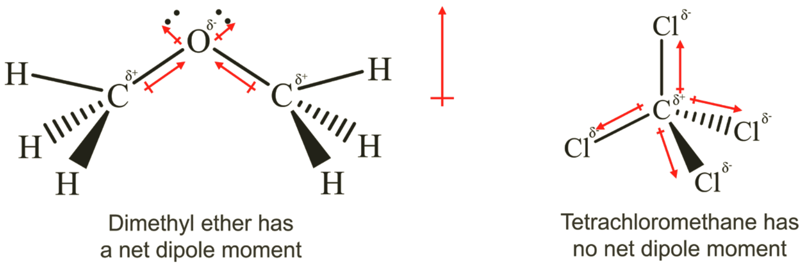
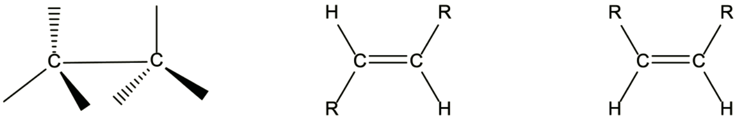


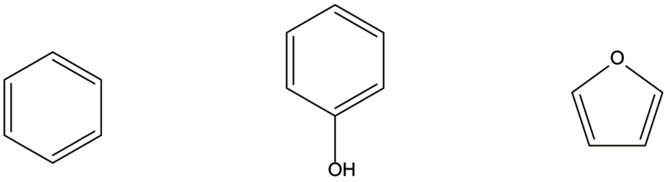
Figure 1.29 Polar dimethyl ether and non-polar tetrachloromethane.

In wood, carbon and oxygen atoms can share not only one but two electron pairs (denoted as one or two solid lines in Figure 1.30), while nitrogen can share up to three electron pairs with carbon, resulting in a number of ways of forming different functional groups. Note that to simplify the drawings carbons are usually omitted in the phenolic rings and single bonds are not drawn between oxygen and hydrogen in hydroxyl groups. R denotes a compound group of C and H atoms.

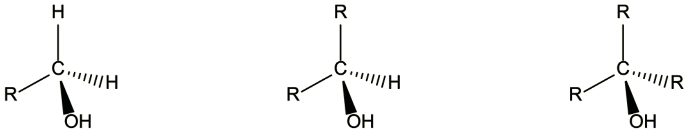
The distance between the two hydrogen atoms in a hydrogen molecule (H_2) is 0.084 nm but the distance is greater between heavier atoms; 0.11 nm for a carbon-hydrogen bond and 0.15 nm for a carbon-carbon bond. Double bonds are shorter than single bonds; 0.13 nm for a carbon-carbon double bond in an alkene (Figure 1.30). A carbon-carbon double bond is stronger than a carbon-carbon single bond. The binding electrons are distributed equally between the two bonding atoms in the product and to homolytically cleave the bond requires 2.85 MJ/mol for CH_2=CH_2 and 1.59 MJ/mol for CH_3-CH_3 (Hart et al., 2003). A chemical structure with no carbon-carbon double bonds is saturated, but a structure with a carbon-carbon double bond is unsaturated. An



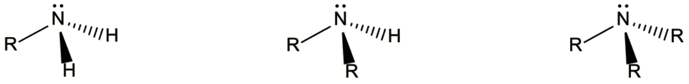
alkane *trans*-alkene *cis*-alkene



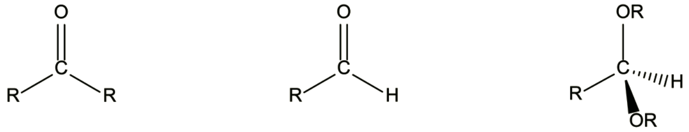
arene phenol furan



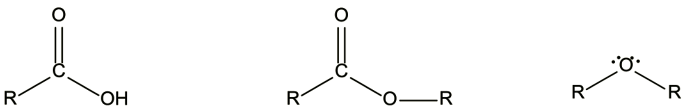
primary alcohol secondary alcohol tertiary alcohol



Primary amine secondary amine tertiary amine



ketone aldehyde acetal



carboxylic acid carboxylic ester ether

Figure 1.30 Functional groups in organic wood constituents. R represents a hydrocarbon substituent.

alkene in an unsaturated fat becomes saturated by reaction with hydrogen gas (-CH=CH- + H_2 → -CH_2-CH_2-). In the case of alternating single and double carbon-carbon bonds (so-called conjugated double bonds), the bond distance between carbons is between those of single and double bonds. In the symmetric arene in Figure 1.30, for example, the bond distance between the carbon atoms is the same, giving a stable aromatic character. Conjugated double bonds are thermodynamically more stable than isolated double bonds, since the electron cloud is distributed over a wider space, increasing the number of degrees of freedom and lowering the entropy of the system. The double-bonded structures are planar, i.e., not tetrahedral, and substituents are separated by 120 degrees, forming a flat structure.

The presence of functional groups influences the structure and the chemical, physical and biological properties of the compound. Important features of molecules are how they interact with other molecules by electrostatic forces or bonds. Such bonds are much weaker than covalent bonds and are usually written as dotted lines. These secondary forces are non-polar, polar and hydrogen bonds. Non-polar bonds act between non-polar compounds and are weaker (2−8 KJ/mol) than polar bonds (6−12 KJ/mol) which exist between polar compounds. Hydrogen bonds are the strongest secondary bonds (12−28 KJ/mol) existing between a hydrogen atom bonded to an electronegative atom (in wood oxygen or nitrogen) and another electronegative atom. Although these secondary forces are much weaker than covalent bonds, they are still of significant importance for the physical properties of the molecules. Non-polar bonds exist mainly between hydrocarbon structures such as non-polar alkanes, alkenes and arenes, whereas polar bonds exist between structures where carbons are bonded to oxygen, nitrogen or other strongly electronegative atoms, as in groups such as ethers, aldehydes, ketones, acetals, esters, amines and amides. Hydrogen bonds are found in alcohols, phenols, carboxylic acids, amines and amides. The hydrocarbon ethane (C_2H_6) which is a non-polar compound exists in a gaseous state at room temperature, whereas methanol (CH_3-OH), which has a molecular weight similar to that of ethane, is a liquid due to the formation of hydrogen bonds between the hydroxyl groups in the alcohol. Methanol can also form hydrogen bonds with water (H-OH) and is, therefore, miscible with water. The solubility of *n*-pentanol ($CH_3(CH_2)_3CH_2$-OH), however, is low. Alcohols with long hydrocarbon chains are insoluble in water because the weaker non-polar forces formed between long hydrocarbon chains in the alcohols counteract the forces between the hydroxyl groups of water and the alcohol, so that the alcohols mix with themselves instead, leading to separation of the mixture into two phases. The rule of thumb that “similar dissolves/interacts with similar” can often be used to roughly evaluate whether substances interact or are miscible with each other.

Chemical reactions of organic compounds

The polarisation of bonds and accessibility to bonds with high energy may indicate chemical reactions involving the breaking and formation of bonds. Those are mostly of a heterogeneous character, where electron pairs are involved, but reactions with single electrons are possible, such as when wood is subjected to UV-radiation and heating at high temperatures. In this section, we focus on reactions involving the heterogeneous breaking and formation of covalent bonds. Alkanes are non-polar and fairly unreactive, whereas one bond in the double bond in alkenes has higher energy and is more reactive than the other bond; ethene (H_2C=CH_2) can add water (H_2O) to form ethanol (CH_3-CH_2OH). The reaction rate increases in the presence of an acid catalyst, such as hydrochloric acid, which is totally dissociated into protonated water (H_3O^+) and a chloride anion. The proton will bind with the double bond in a first step, resulting in a high energy and reactive carbocation intermediate. A lone pair of electrons from the electronegative oxygen in water adds to the positively charged carbon, followed by a rapid deprotonation to give the alcohol, and the proton can be re-used as a catalyst in the first step of the reaction (Figure 1.31).

However, the reaction is reversible, which means that under certain conditions alkene can be formed as a result of the dehydration of the alcohol. Removal of the alcohol during hydration of the alkene favours the formation of new alcohol molecules in order to maintain the equilibrium, whereas

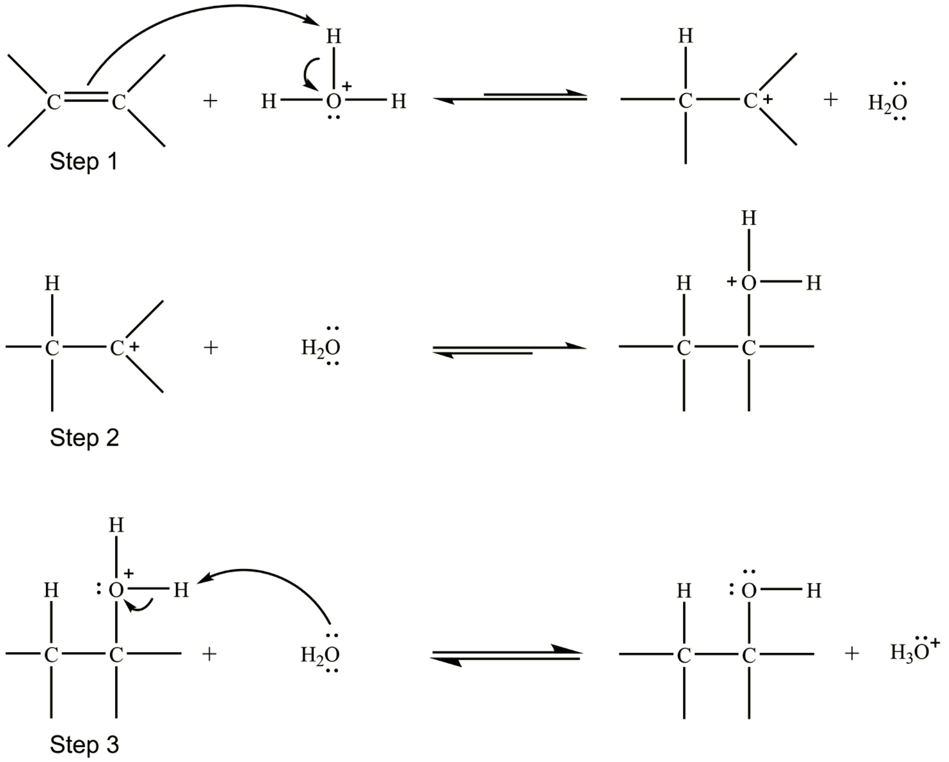


Figure 1.31 Hydration of alkene into alcohol catalysed by an acid (proton): Step 1: Protonation of the double bond to form a carbocation, Step 2: Nucleophilic attack by water, and Step 3: Deprotonation to form an alcohol.

removal of water favours the formation of alkene from alcohol. The stability and concentration of the reactants and products, their physical state (solid, liquid or gas) and the reaction temperature are also important for the outcome of the reactions and, in some cases, the reaction may take place in one direction. The reaction rates are generally lower for large and solid compounds than for small compounds in a liquid phase. The formation of ethers by condensation of two alcohol molecules is usually less favourable, but will result if the product is stabilised, as is the case when furfurals are formed by the heating of monosaccharides. Ethers (R-O-R) are fairly stable and need strong acids to be cleaved, but this may occur under milder acid conditions when the ether is activated, such as with the β-ether bond in the phenolic arylglycerol-β-aryl ether in some lignins (Figure 1.38).

Another reaction of importance in wood chemistry is the formation of acetals by the reaction of an aldehyde and two alcohol molecules catalysed by an acid (Hart et al., 2003). The lone electron pair from the oxygen atom in the alcohol (R^2OH) will bond to the electropositive carbon in the aldehyde ($R^1HC{=}O$) giving a hemiacetal which is normally not very stable and could reverse to the initial compound even under non-catalysed conditions. In the presence of an acid catalyst a new alcohol could react with the formed hydroxy group in the hemiacetal to give an acetal and water (Figure 1.32). As these reactions are reversible under those conditions, the removal of water moves the reaction towards the acetal, whereas the addition of water moves the reaction back towards the starting materials. Acetals are stable in strong alkaline conditions, as in the presence of NaOH (aqueous), whereas hemiacetals are not.

Ketals and hemiketals are formed in an analogous way by reaction with ketones ($R_2C{=}O$) and alcohols.

Aldehydes can also react with phenolic compounds, but the reaction with the phenolic hydroxyl group (PhOH, where Ph refers to the phenyl ring) results in fairly unstable products. The electron pair in the aromatic ring can instead bond with the electropositive carbon in the aldehyde by substituting

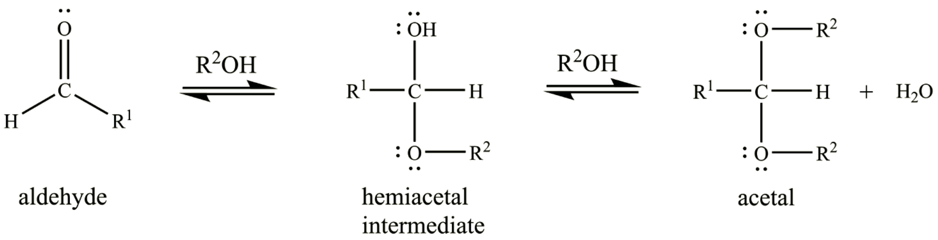


Figure 1.32 Reversibility of acid catalysed acetal formation (R^1 = H or hydrocarbon substituent, R^2 = hydrocarbon substituent).

with a hydrogen bonded to the ring (electrophilic aromatic substitution) (Hart et al., 2003). The reaction can be catalysed by both acid and alkali and a phenolic methylol ($HOPhCH_2OH$) group is formed by the reaction with formaldehyde (Figure 1.33).

As the aromatic structure is re-formed in the methylolated phenol (Hart et al., 2003), further reactions of formaldehyde with the methylolated phenol, unless blocked by other groups, may continue up to a maximum of three methylol groups. The phenolic methylol group ($HOPhCH_2OH$) can also react with the double bond in a vacant position in another phenolic unit, forming a more stable methylene bridge ($HOPhCH_2PhOH$). Examples of various coupling patterns are shown in Figure 1.33 (bottom). The reactions are condensation reactions, which means that a higher molecular weight compound and water is formed. In Figure 1.33, three possible dimers are shown and they

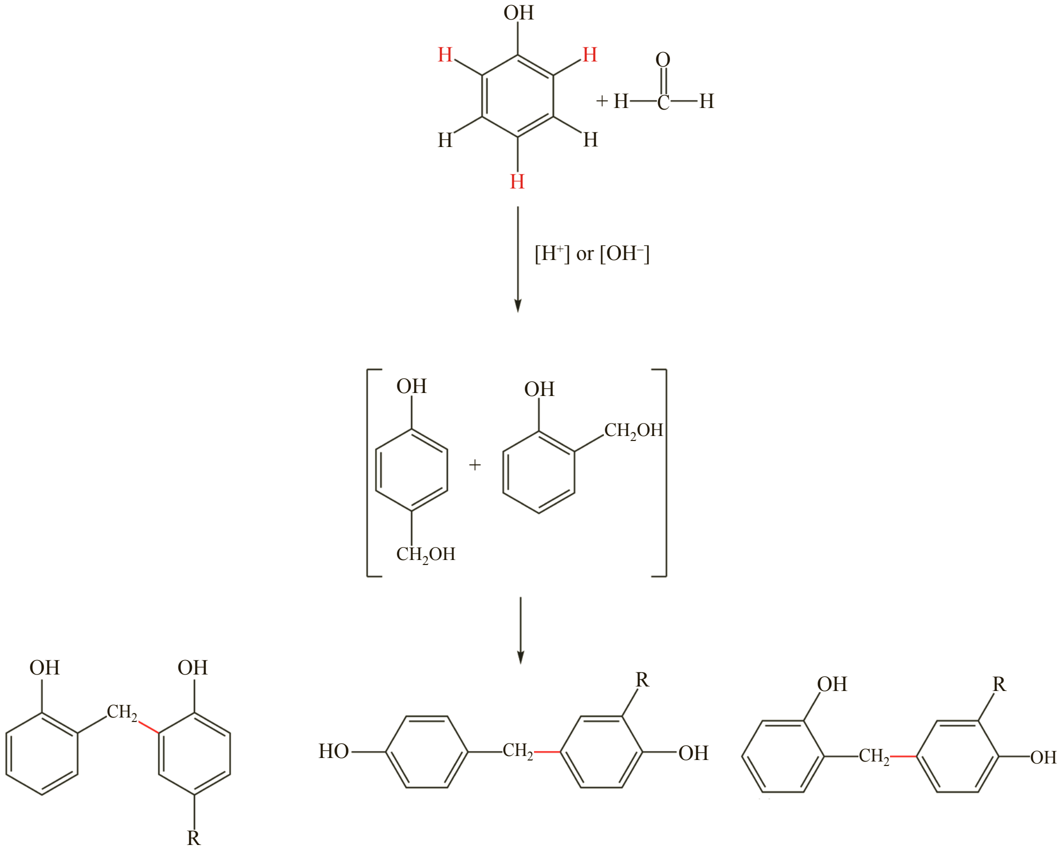


Figure 1.33 Examples of acid- and base-catalysed reactions of phenol and formaldehyde.

can continue to form higher molecular weight polymeric resol-type phenolic compounds when base catalysed via reactions with more phenolic methylol groups. During acid catalysis, novolac structures are formed with no methylol groups and formaldehyde needs to be added for achieving further condensation reactions. As a result of reactions with more extensively methylolated phenols, a less flexible cross-linked network structure can be obtained.

Structure and reactions of wood constituents

Almost all compounds in wood are organic molecules made up of carbon-based skeletons with straight, branched and ring structures in which atoms are held together by sharing electron pairs to form covalent bonds. Wood consists mainly of carbohydrates which basically consist of monosaccharides. In monosaccharides, hydrates (or hydroxyl groups) are covalently bonded to each carbon in a five- or six-carbon membered chain in which at least one of the hydroxyls is in the oxidised form, often as a terminal group (-CHO). Monosaccharides exist in the form of ring structures, such as β-D-glucose in the upper right part in Figure 1.34, essentially a ring-formed hemiacetal (Figure 1.32) in equilibrium with the open aldehyde form but also with the α-form of glucose in which the hydroxyl group at C1 points in the axial direction (upper left part in Figure 1.34). Glucose can be found mostly in the cambium where the biosynthesis of wood occurs. Nearly all carbohydrates in wood (except some mono- and oligosaccharides) are polymers consisting of covalently bonded monomers (monosaccharides or monosaccharide acids) forming linear and branched chains. Cellulose is the most abundant carbohydrate in wood, constituting 40–45% of the total wood mass in which the monomers (β-D-glucoside) are bonded in the same way; carbon 1 in one unit to carbon 4 in another unit via a glucosidic bond (1→4) into an extremely long straight back-bone chain, 10,000–15,000 monosaccharide units in the cell wall as shown in the lower part of Figure 1.34 (Goring and Timell, 1962). A glucoside is about 0.5 nm in size, so the maximal length of a cellulose chain is 5–7 μm, which is about the same as the cell wall thickness (Fengel and Wegner, 1989).

Water is formed in connection with formation of glucosidic bonds together with the growing chain during cellulose biosynthesis. However, the glucosidic bonds formed can be cleaved by reaction with water in the presence of a cellulose-degrading enzyme (cellulase), even under ambient

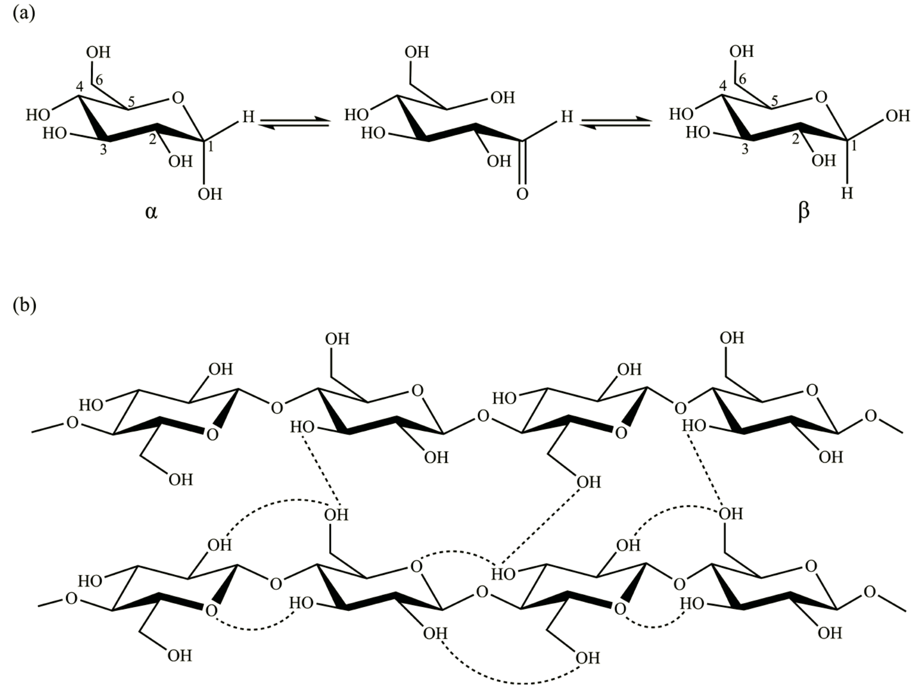


Figure 1.34 Equilibrium between β-D-glucose, open form and α-D-glucose (upper), and parts of two cellulose chains displaying hydrogen bonds within and between the chains (lower).

conditions. Acid catalysed hydrolysis (also consuming water) of the glucosidic bond in cellulose requires strong acid conditions and heat in contrast to the hydrolysis of many other polysaccharides (Figure 1.35). Homolytic cleavage of the glucosidic bond may take place, especially when wood is heated at high temperatures under neutral and dry conditions.

Why are such strong conditions needed when the formation of cellulose in the cell wall takes place under much milder conditions? This is because the cellulose chains in wood exist mostly in stable crystalline structures with a crystallinity of about 60% (Newman and Hemmingson, 1990). These crystalline structures are stabilised by forming hydrogen bonds (dotted lines in Figure 1.34) between hydroxyl groups and between hydroxyl groups and oxygen in neighbouring rings. Hydrogen bonds also exist between cellulose chains which are efficiently packed into three-dimensional crystalline fibrils joined into microfibrils 2–4 nm in width, as shown in Figure 1.36 (Donaldson, 2007). At the microcrystal surface, less ordered paracrystalline cellulose chains exist. Other molecules, such as hemicelluloses, water or similar polar compounds (preferentially those that can form hydrogen bonds) can also interact at the cellulose surfaces. Microfibrils are linked by more sensitive amorphous regions and aligned into thicker macrofibrils that can be up to 40 µm long, achieved by overlapping cellulose chains (Ek et al., 2009). Fibrils are oriented in various directions depending on their position in the cell wall. In the S_2 layers, the fibrils are almost aligned in the fibre direction, but they have other directions in other cell-wall layers (Figure 1.25 and 1.36).

Hemicelluloses are heterogeneous polysaccharides constituting 15−30% of the mass in wood and consist mainly of monosaccharides linked to each other by glucosidic bonds (Ek et al., 2009). The backbone chain in hemicelluloses consists of monosaccharides (β-D-glucose and β-D-

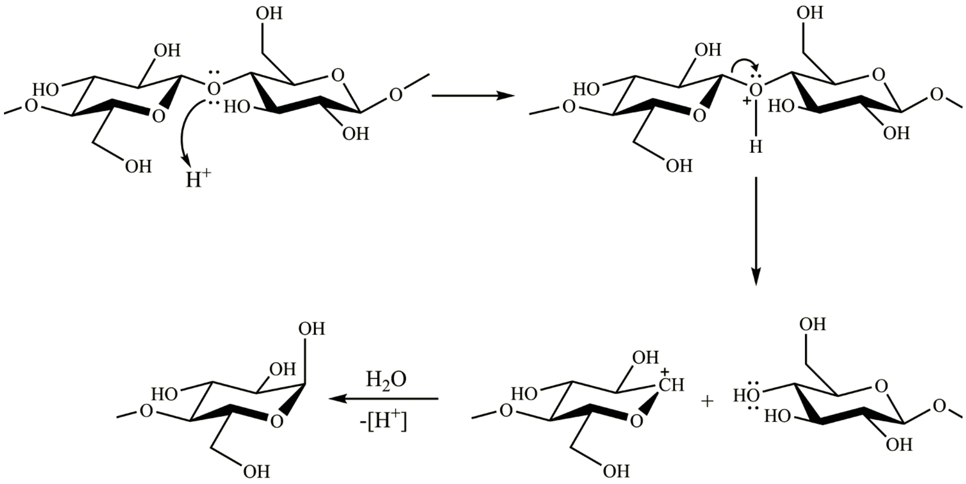


Figure 1.35 Acid catalysed hydrolysis of glucosidic bond in cellulose.

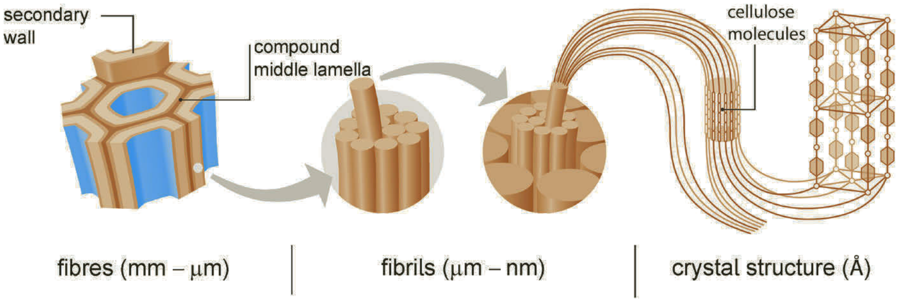
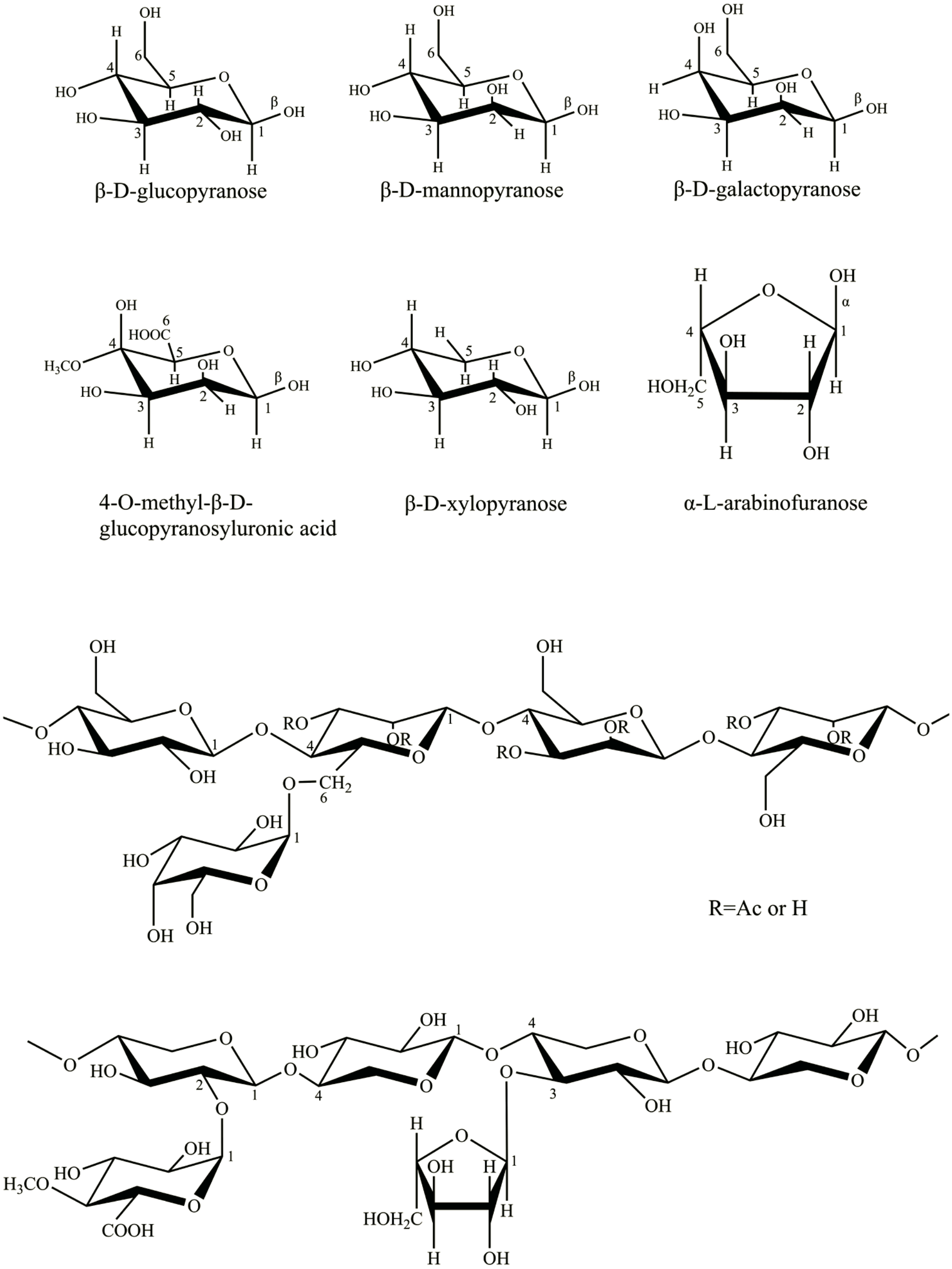


Figure 1.36 Elementary fibril, micro- and macrofibril of cellulose and their orientation in the cell-wall layers (Zimmermann et al., 2004).

mannose in glucomannans and β-D-xylose in xylans) with side groups (β-D-galactose in conifer glucomannans, α-L-arabinose in conifer xylans and acetyl groups in broad-leaved xylans as well as in conifer glucomannan) and ionising groups (4-O-methylglucuronic acid in xylans). The backbone is much shorter than the cellulose chains and consists of 100–200 monomer units (Figure 1.37).

This heterogeneous structure makes hemicelluloses amorphous with hydroxyl groups freer and more accessible to form hydrogen bonds with other compounds such as water. When wood is exposed to humid conditions, water molecules substitute with the hydrogen bonds mostly between


β-D-glucopyranose

β-D-mannopyranose

β-D-galactopyranose

4-O-methyl-β-D-glucopyranosyluronic acid

β-D-xylopyranose

α-L-arabinofuranose

Figure 1.37 Hemicellulose structures: monosaccharides and uronic acid in hemicelluloses (upper two rows), galactoglucomannan in conifers (middle), and conifer xylan (lower).

carbohydrate chains and, therefore, require more space, resulting in a swelling of the cell wall up to a moisture content of 25–30% when the cell wall becomes saturated (at the so-called fibre-saturation point, FSP). The cell wall swells mostly outwards and the result is a macroscopic swelling of the wood, particularly in the transverse direction. Swelling also makes the wood more reactive and susceptible to microbial or other attacks. The wood constituents are also softened by the increase in moisture content and this occurs to a greater extent in the hemicelluloses than in the cellulose and lignin, especially at lower temperatures (Back and Salmén, 1982). To be able to selectively separate wood fibres during mechanical pulping, for example, softening at a high moisture content and sufficient high temperature of the lignin-rich middle lamella is necessary.

Polysaccharides are polymeric carbohydrates that are non-structural, but they still have important functions in wood, such as starch working as a nutrient, pectins which are constituents of the primary wall and pit membrane and galactan in tension wood. Further information on structures of polysaccharides has been published elsewhere (cf. Ek et al., 2009). Due to their carbohydrate origin, they are sensitive to acidic, alkaline and microbiological attacks and the pit membranes can be degraded by bacteria when wood is stored in water.

Lignin is also a polymeric compound, constituting 20–30% of the mass in wood. It has a higher C:O ratio and a higher calorific value than the carbohydrates and interacts with water to a lesser extent than hemicelluloses. The relative content of lignin is high in cell corners and the middle lamella but it is also present in the cell wall and assists in binding the wood cell together and in making the cell wall less sensitive to moisture. Lignins have a phenolic origin and, in wood, they are made up essentially of three phenylpropane units: H-type (*p*-hydroxyphenylpropane), G-type (guaiacylpropane) with one methoxyl ($-OCH_3$) group bonded to the aromatic unit dominant in conifers and S-type (syringylpropane) with two methoxyl groups. These last two types form the predominant GS-type of lignin in broad-leaved woods (Figure 1.38).

The presence of these units differs, depending on their position in the tree; compression wood has a higher lignin content and a higher content of H-units than the lignin in clear wood (Westermark, 1985). The middle lamella binds the wood cells together and has relatively more H-units than the secondary wall. The phenyl propane units in lignin polymers are linked to each other by ether and carbon-carbon covalent bonds in at least 10 different ways, the beta-ether bond (β-O-4) being the most common (Figure 1.38). The branching and phenolic content in cell-wall lignin are fairly low, but a lower content of uncondensed β-ether structures in the middle lamella has been suggested (Westermark, 1985) and this indicates more branching. In broad-leaved species, GS-lignin has more β-ether bonds than G-type lignin in conifers. The β-ether bonds are sensitive both to heating and to acidic and alkaline conditions (Westermark et al., 1995) and this is important for the delignification processes (Gierer, 1980). Such reaction will lower molecular weight and increase the phenolic content of lignin. Methoxyl groups bonded to aromatic rings are more stable than β-ether bonds under hydrolytic conditions. However, methoxyl groups in lignin could be degraded oxidatively by outdoor exposures (see Section 1.6). Thermal modification could lead to cleavage of β-ether bonds (Tjeerdsma et al., 1998; Windeisen et al., 2007). Most inter-unit carbon-carbon bonds are stable under hydrolytic conditions, but formaldehyde can be formed by splitting off the terminal methylol group in the propane chain during such heating. These reactions take place under acid-catalysed conditions at lower temperatures than thermal modification (i.e., below the threshold temperature of 150°C), especially in the presence of sulphuric acid (Lundquist, 1992). Under certain conditions, more stable condensed carbon-carbon structures may be formed (Figure 1.41). Lignin is, to some extent, mainly covalently bonded to hemicelluloses such as α-ether bonds (Karlsson et al., 2004; Balakshin et al., 2011). Galactoglucomannan in the lignin-hemicellulose matrix is considered to interact with cellulose fibril surfaces by hydrogen bonds (Ek et al., 2009). Thus, the ultrastructure of a conifer cell wall may be regarded as a series of interconnected layers consisting of cellulose, galactoglucomannan, lignin-xylan, galactoglucomannan and cellulose. Lignin-carbohydrate bonds can contribute to the (wet) stability of the matrix and explain the oxidative bleaching conditions needed to selectively remove residual lignin from pulps.

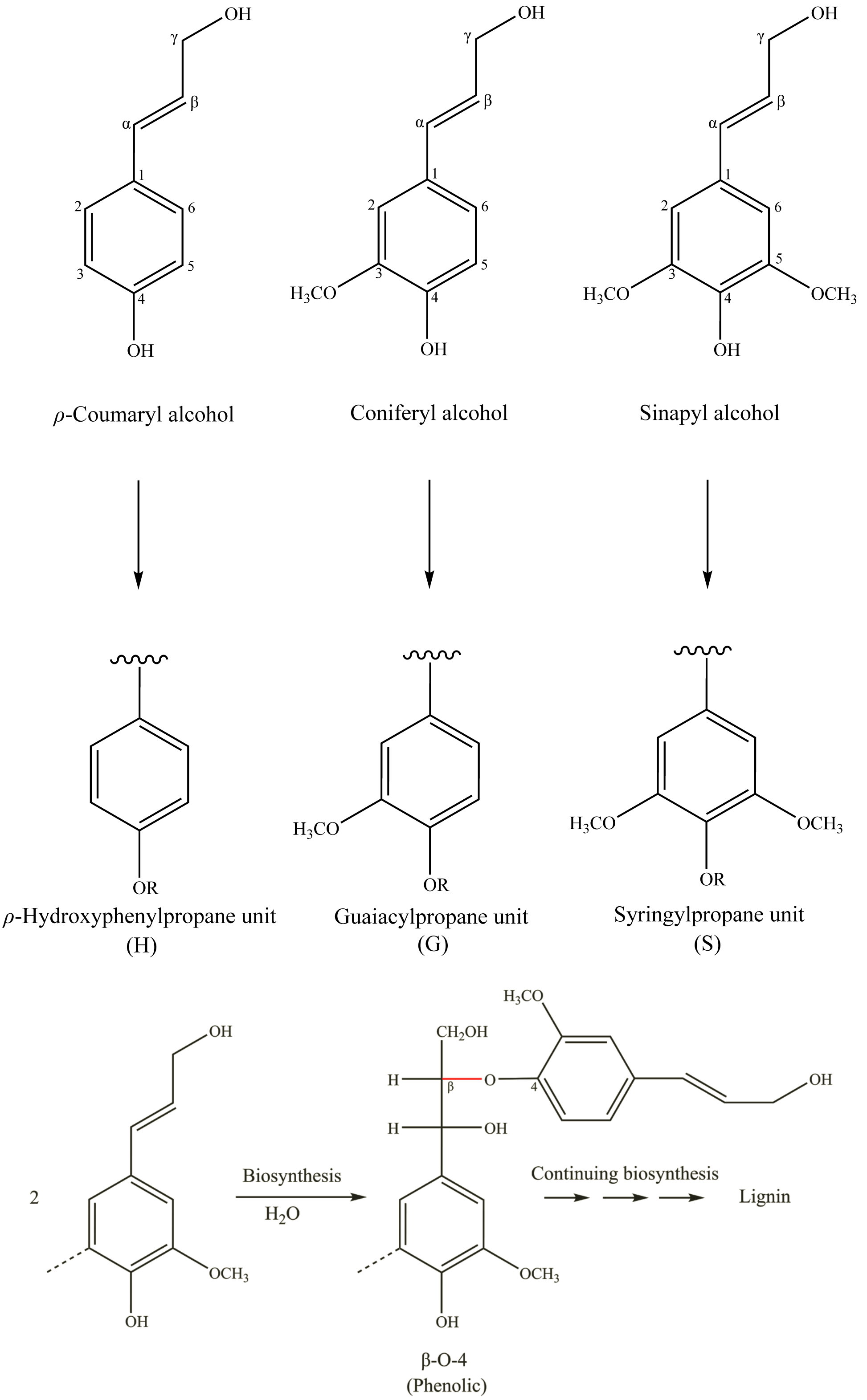


Figure 1.38 Building units in lignin and formation of arylglycerol-β-aryl ether (β-ether).

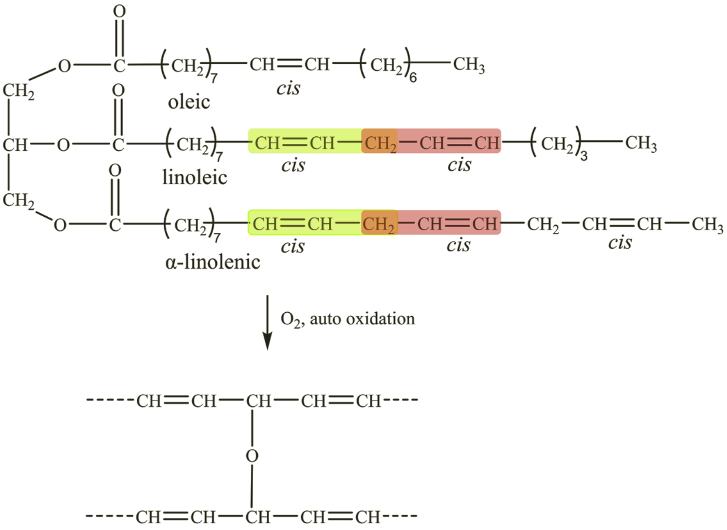


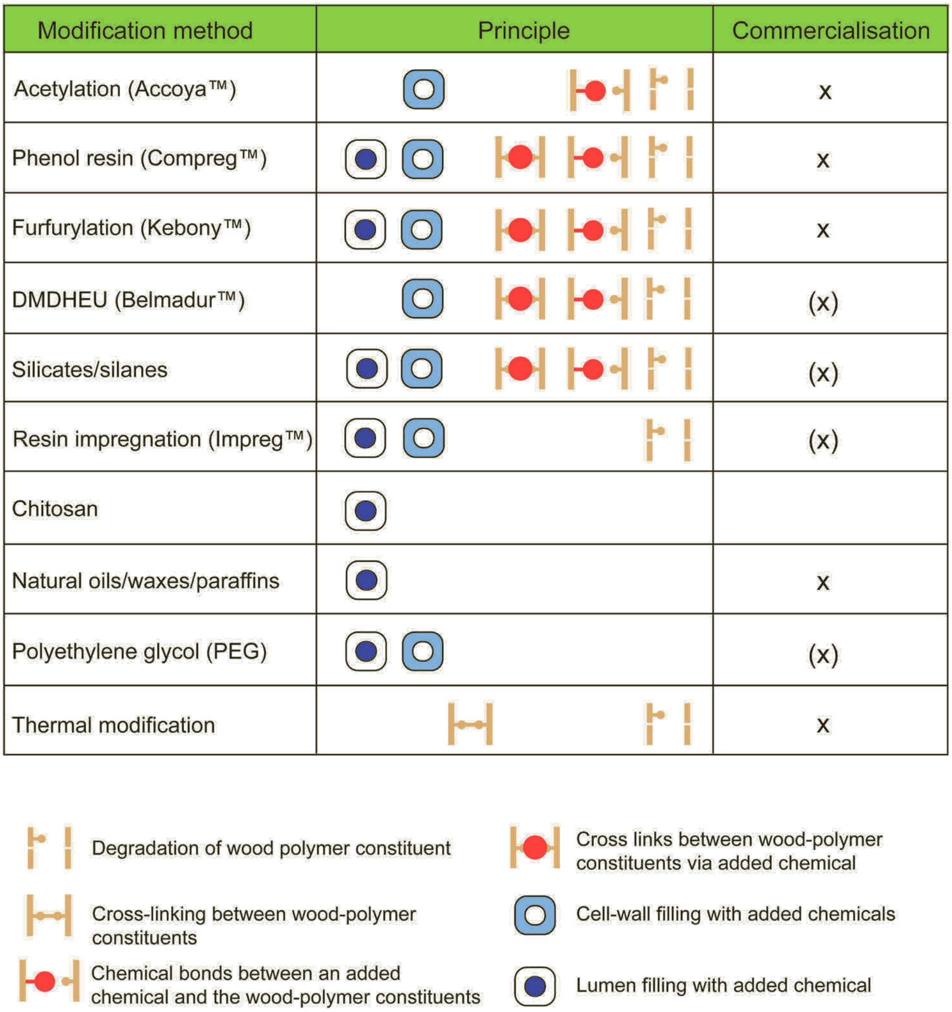
Figure 1.39 Reactions of unsaturated triglycerides during the curing of drying oils.

The non-structural compounds usually constitute a minor amount of the mass of wood and typically consist of low molecular hydrophobic extractives of various types and various solubilities, mono-, oligo- and polysaccharides, inorganic salts and small amounts of proteins and peptides. Basically, extractives obtained by extraction with organic solvents, such as acetone, are fats, waxes, fatty acids, terpenoids (resin acids, monoterpenes and sesquiterpenes, steroids) and stearyl esters, and phenols (lignans, tannins). They are considered to be used by the wood for protection (phenols, resin acids and terpenes, fatty acids), as nutrients (fats and sugars) and during biosynthesis (steroids, proteins, inorganics). These non-structural constituents have relatively little effect on the strength properties and the equilibrium moisture content (EMC) of wood, but they may consume some of the modifying reagents by reactions with reactive groups, such as hydroxyl groups in carbohydrates and phenolic compounds in tannins and lignans, etc. They may also hinder the efficient uptake and contact with the wood polymers by oxidative drying of migrated unsaturated oily resins in aspirated pits, resulting in ether cross-linking of the side-chains (Figure 1.39). Under alkaline conditions, ester bonds in fats, stearyl esters and waxes can be saponified, forming corresponding alcohols and ionisable fatty carboxylic groups which can have surfactant properties. Cleavage of ester bonds may also take place during acid conditions as well as by heating.

The chemistry of wood modification

When wood is modified, the aim is primarily to reduce the moisture uptake and thereby hinder biological degradation by changing the chemical structure instead of adding toxic substances (biocides) or using durable trees from, e.g., endangered tropical resources. The modification should be undertaken in a way to ensure there is no release of hazardous chemicals as a result of the treatment or during the service life of the product. Often, the modification results in a reduction in the interaction of the treated wood with moisture and water, so reducing its ability to swell and shrink, making the wood more dimensionally stable. Wood modification can be divided into two principles: *passive* and *active* modification (Hill, 2006). In Figure 1.40, various modification methods and their mechanisms of wood protection are presented.

The degradation of wood-polymer constituents involves a lot of possible reaction and reaction routes, some of which have already been presented. Degradation by the splitting of sensitive covalent bonds (glucosidic) in carbohydrate polymers increases with increasing temperature and, if water is present, it can be consumed during the splitting of these bonds (hydrolysis) catalysed by acid (cf.



Modification method	Principle	Commercialisation
Acetylation (Accoya™)	◎ ⊢●·⊣ ⊦· ¦	x
Phenol resin (Compreg™)	◉ ◎ ⊢⬤⊣ ⊢●·⊣ ⊦· ¦	x
Furfurylation (Kebony™)	◉ ◎ ⊢⬤⊣ ⊢●·⊣ ⊦· ¦	x
DMDHEU (Belmadur™)	◎ ⊢⬤⊣ ⊢●·⊣ ⊦· ¦	(x)
Silicates/silanes	◉ ◎ ⊢⬤⊣ ⊢●·⊣ ⊦· ¦	(x)
Resin impregnation (Impreg™)	◉ ◎ ⊦· ¦	(x)
Chitosan	◉	
Natural oils/waxes/paraffins	◉	x
Polyethylene glycol (PEG)	◉ ◎	(x)
Thermal modification	⊢··⊣ ⊦· ¦	x

⊦· ¦ Degradation of wood polymer constituent

⊢⬤⊣ Cross links between wood-polymer constituents via added chemical

⊢··⊣ Cross-linking between wood-polymer constituents

◎ Cell-wall filling with added chemicals

⊢●·⊣ Chemical bonds between an added chemical and the wood-polymer constituents

◉ Lumen filling with added chemical

Figure 1.40 Chemical- and thermal-modification methods, commercial and principle. X − available product, (X) − introduced or used in large-scale experiments.

Figure 1.35). Glucoside bonds in hemicelluloses are degraded more easily than cellulose during heating and by hydrolysis in the presence of acid. Bonds to hemicellulose side groups, like arabinose, are more sensitive to acid hydrolysis than the backbone chain as suggested by Mäki-Arvela et al., 2011 (Figure 1.37). Greater losses of carbohydrates were observed with birch than with pine even at a slightly lower temperature during thermal modification in open systems (Zaman et al., 2000). The formation of soluble xylose-rich carbohydrates during thermal modification was greater in closed systems saturated with steam than in an open system (Karlsson et al., 2012). The degradation of wood polymers leads to shrinkage of the wood cell wall and, thus, to the dimensions of the wood and a small reduction in wood density occurs when degraded materials are removed during the heating. Under high pressure conditions, the steam pressure had a greater influence than the peak treatment temperature on the degradation of European beech wood (Willems et al., 2015). During thermal modification, which is considered to start at ca. 150°C, the degradation reactions are dehydration of hydroxyl groups, increasing C:O ratio and the hydrophobicity of the material, although some volatile dehydration products, such as furfurals, may be removed during the thermal treatment (Figure 1.41). Acetic acid, formed by the hydrolysis of acetyl groups of acetylgalactoglucomannans in conifers, for example, together with other acids lower the pH and may further catalyse degradation reactions.

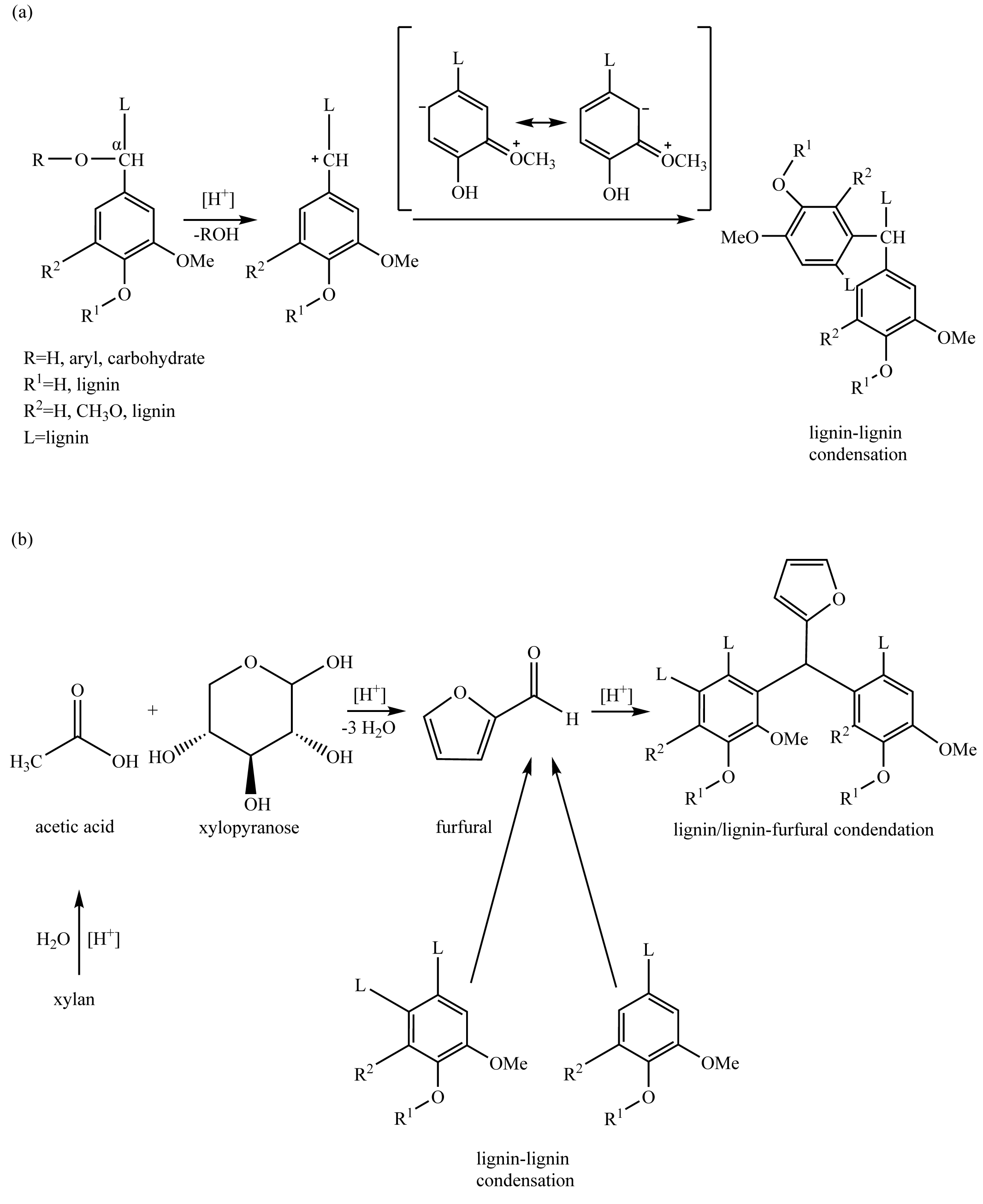


Figure 1.41 Examples of possible condensation products from lignin (upper), and degradation products of hemicelluloses during heating (lower).

Thermal modification under closed water-saturated conditions leads to an even lower pH, as has been found with birch sp., which has an acetyl content of about 3% (Torniainen et al., 2011; Rowell, 2012). As a consequence of the formation of more acidic modified-wood products, acid-resistant fasteners have to be used in constructions. It appears that there is a difference in the moisture uptake capacity of thermally modified wood during remoistening cycles, depending on whether the modification has been carried out in the presence of absence of water (Obataya and Tomita, 2002) and it has been suggested that this is due to the formation of reversible hydrogen bonds between hemicelluloses in the dry systems (Willems et al., 2020). The degradation can lead not only to a decrease in strength properties in wood but also to a less elastic material when the amorphous and flexible carbohydrate polymers are degraded.

The colour intensity increases with the intensity of treatment, this may be due to degraded lignin and also to lignin-like material formed from carbohydrate degradation products. Particularly in closed processes, moisture and acids can catalyse the modification reactions and a lower treatment temperature can be chosen than in an open process, such as the ThermoWood™ process, to get similar browning, although there being some differences in the reactions which occur. High strength and durability cannot be achieved by these processes. The material is quite durable but the treated wood is more sensitive to in-ground contact than wood from the acetylation and furfurylation processes.

In the presence of oxygen, the rate of degradation of wood is increased and alkali-labile oxidation structures may form. Sensitive bonds in lignin, such as β-ether bonds, may be cleaved, increasing the amount of phenols (Windeisen et al., 2007), while α-ether bonds (which are more sensitive than β-ether bonds) may result in free carbohydrates as a result of cleavage of lignin-carbohydrate structures. During heating, the formation of combustible gases may be sufficient to start a chain reaction and initiate combustion of the material. However, oxidative conditions are believed to have minor importance during the thermal modification of wood due to the inert atmosphere existing during the treatments.

Cross-links may occur between wood polymers when wood is heated (Tjeerdsma et al., 1998; Boonstra and Tjeerdsma, 2006). Condensation of lignin with itself (upper reaction in Figure 1.41) and together with some dehydrated carbohydrate compounds like furfural seems possible (lower reaction in Figure 1.41) especially under acidic conditions. Other aldehydes, such as formaldehyde, may lead to condensation products with lignin.

Since reactions with wood constituents on the cell-wall surface of the lumen or with pit membranes do not, in the long run, protect moisture from entering into and swelling the cell wall, the modifying chemicals need pores in the wood in order to be able to reach inner parts of the cell wall (Hill, 2006). Nano-pores exist in the cell wall when the wood is in a swollen state, but it is uncertain whether they are present in dry cell walls. They are either absent or very small. The modifying chemical and any solvent used must be able to find or create voids large enough to permit entry into the cell wall, and the covalently bonded penetrated material with low attained solubility still requires a greater volume when the solvents and reaction by-products have been removed at the end of process. This increases the cell-wall dimensions in the dry state and also the volume of the wood. Since wood swelling then becomes more restricted, the cell wall retains less water than untreated wood and this lowers the equilibrium moisture content in modified wood and reduces the dimensional changes upon drying and wetting. Under certain conditions, however, the swelling during treatment is so great that the wood starts to undergo irreversible cracking.

The acetylation of wood (Figure 1.42) is a chemical modification process in which the electrophilic reagent (acetic anhydride) is forced by the application of an external pressure to migrate through the wood pits, to react with accessible nucleophilic hydroxyl groups in the wood and to diffuse and react deeper into the cell wall (Rowell, 1983). Thus, bulking of the cell wall and loss of hydrophilic hydroxyl groups reduces the moisture uptake, and increases the resistance to swelling and the decay of wood (Hill and Jones, 1996; Hill, 2006). So far, radiata pine has mostly been used commercially due to its low density and open pore structure, but fibres in acetylated fibreboards can be more easily reacted than the solid wood products, and this can favour the use of other species.

As the anhydride is reactive with water, any moisture present will consume the reagent. The reactivity of the resulting acetic acid is not high and it will not further react with hydroxyl groups to any significant extent although it may work as a solvent (Rowell, 2012) and also assist in the degradation of wood constituents, especially if the temperature becomes too high due to the exorthermic nature of the acetylation reaction. Removal of the residual reaction solution at the end of the process leaves a bulked cell wall with increased wood dimensions, but also an essentially

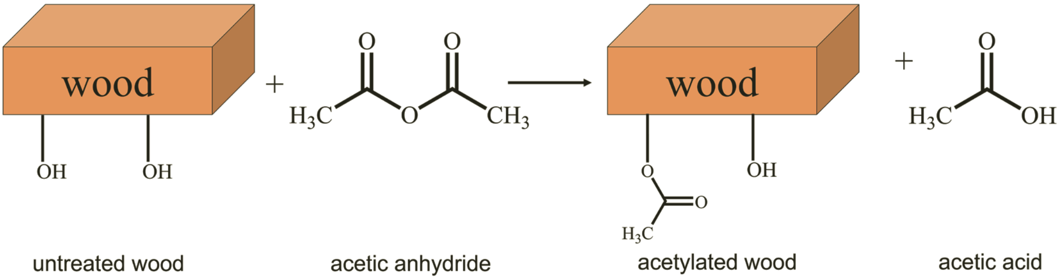


Figure 1.42 Acetylation of wood by acetic anhydride.

empty lumen with only small amounts of residual acetic acid. This and the extent of acetylation depend on several factors and is discussed together with other aspects of acetylation in Section 2.2.

The cross-linking of wood constituents by forming a bridge structure with the added chemical may also restrict the uptake of water by the cell wall, so that the total volume of the swollen wood cell wall at a given relative humidity is lowered (Figure 1.43 and Figure 1.44). Such reactions may be difficult to verify, especially if they are less prevalent, for example, during furfurylation (Figure 2.21). The formation of cross-linked structures can be supported if the modifying chemical contains more than one functionality or reactive group, if there is sufficient reactivity to form stable bonds with wood, if the size of the molecule is suitable for cross-linking and if the polarity of the molecule is appropriate for it to be mixed with the wood polymers.

Formaldehyde (HCHO) is known to react with phenols (Figure 1.33) and also with urea and melamine, forming synthetic adhesives. It seems also to react with wood polymers, especially in the presence of strong acid catalysts (Tarkow and Stamm, 1953) as the treatment gives a material with high resistance to swelling at low percentage weight gain. Formaldehyde itself has a low molecular weight, however, and a fairer comparison of the degree of reaction might be to consider the number of moles reacted. Nevertheless, formaldehyde may work as a cross-linker between lignin units but to a lower extent than when it reacts with phenols to form phenol-formaldehyde (PF) resin. The small formaldehyde molecule is not as flexible as larger aldehydes, such as glutaraldehyde, and this results in a brittle material. Formaldehyde itself is a gas and is seldom used due to its hazardous effects on human health. When used in the formation of PF and other formaldehyde-containing resin systems, it is applied as formalin (typically a 40% solution of formaldehyde in water).

Phenolic or resorcinol (PF or RF respectively) resins, formed from phenols or resorcinol (1,3-benzenediol) and formaldehyde, can be used as adhesives for wood products and also to modify and stabilise wood if an appropriately low molecular weight resin is used (Stamm and Seborg, 1939).

As they fill up lumen, the resin reacts/condenses with itself into larger more stable thermoset fragments when heated under alkaline conditions (Figure 1.43).

When an aqueous resin with relatively low viscosity and low molecular weight is used, a bulking effect is usually noted, and impregnation of the cell wall leads to a softening of the wood (Shams and Yano, 2011). Chemical bridges (like stable methylene) can form between reactive sites (activated aromatic carbons) in lignin and methylol groups (-CH_2OH) in PF-resin when wood is treated and cured with such a resin (Yelle and Ralph, 2016). The treatment leads to an increase of density which can be further increased by the application of pressure and heat during the curing of the softened wood in the Compreg™ process. Phenolic reagents may need careful handling and are based on fossil fuels, but they are included in this presentation as phenols may be replaced by bioderived alternatives.

An attractive wood modifier is 1,3-dimethylol-4,5-dihydroxyethyleneurea (DMDHEU) originally developed as a stable anti-wrinkling agent for fabrics, it has more recently been proposed and tested as an agent for wood modification. A stable bulking effect is seen when permeable wood

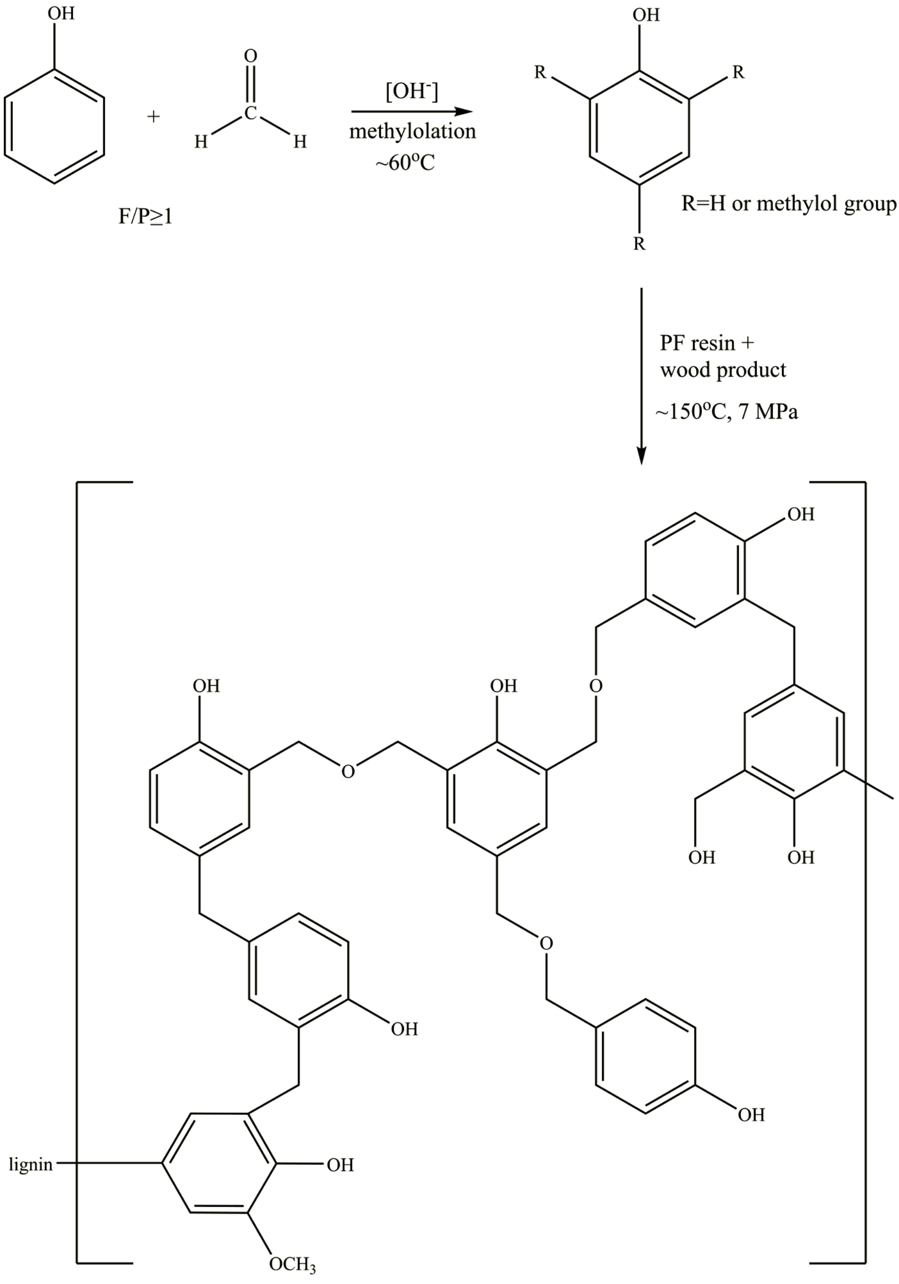


Figure 1.43 Curing of phenolic resin.

species are treated with aqueous DMDHEU (Militz, 1993; Emmerich et al., 2019). It may react with itself forming larger condensation products and at high treatment levels it can start to fill the lumen.

DMDHEU can also react with hydroxyl groups in wood constituents in the cell wall, forming new chemical bonds to these constituents (Figure 1.44). DMDHEU has two or more reactive functional groups and it has been suggested that cross-linked structures are formed in wood (Emmerich, 2019). Wood constituents may degrade due to high contents of added Lewis acid catalysts, and unreacted DMDHEU may lose formaldehyde during the process.

Treatment containing silicate/silane agents involves several types of more or less reactive compounds which may penetrate and swell the cell wall by reacting with hydroxyl groups in the

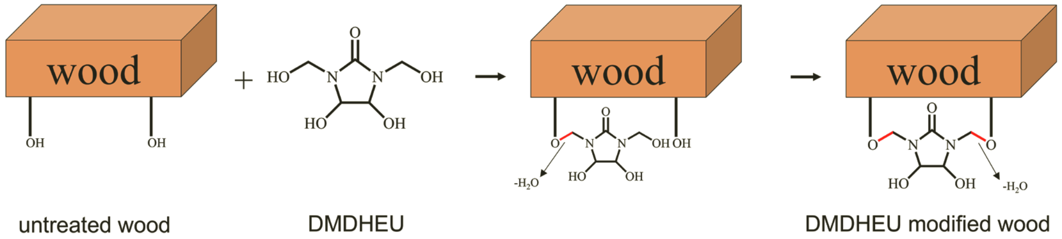


Figure 1.44 Reaction of dimethylol dihydroxy ethylene urea (DMDHEU) during wood modification.

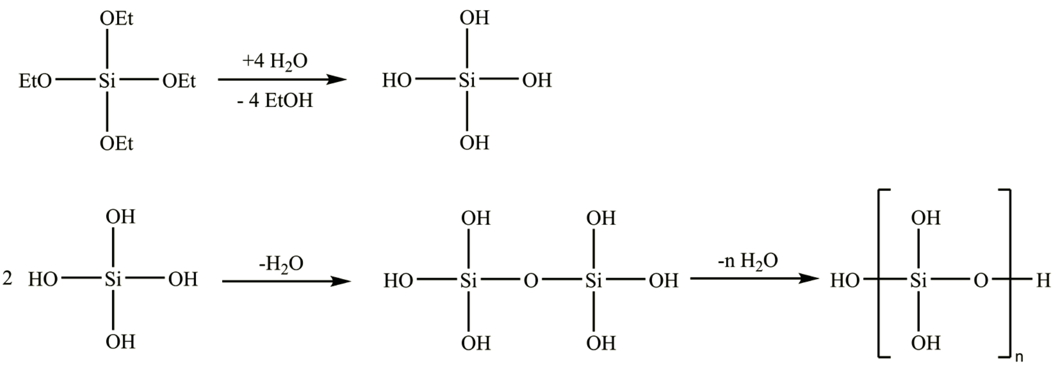


Figure 1.45 Reactions of silyl ether, tetraethyl-orthosilicate (TEOS) in presence of water (Mai et al., 2003).

wood constituents or by filling up the lumen in a more passive way, involving polymerisation reactions with the reagent itself. Treatment with water glass (sodium silicate) involves penetration into the wood but not into the wood cell wall, as bulking does not normally occur. Such alkaline silicates may need to be fixed to the wood to avoid leaching when the wood is exposed to water. There are numerous silanes and many of them are silyl ethers which will be hydrolysed in the presence of moisture to a silanol releasing the corresponding alcohol before reacting with itself (Figure 1.45) or with hydroxyl groups in the wood constituents (Mai et al., 2003).

The stability of the formed products may be influenced by the reacted silica-based compound itself, as well as by conditions such as the presence of acid together with moisture and heat.

Furfuryl alcohol or a similar agent reacts via condensation reactions, forming methylene and ether bridges, usually in the presence of an organic acid catalyst (Figure 1.45). Formaldehyde may be released during the treatments but could condense leading to a branched and cross-linked structure (Figures 1.46c and 1.46e, respectively).

Based on studies using model compounds of lignin, it has been suggested that bonds are formed between the furfuryl alcohol and the lignin but this is probably only to a minor extent (see the furfurylation section in Chapter 2). The cell wall is bulked by treatment and the percentage weight gain after hardening of the alcohol can be quite high. The lumen will, therefore, also start to be filled and that leads to the second type of treatment – *passive modification*.

Passive modification involves lumen filling and/or cell wall filling without a reaction with the wood constituents. Oils, waxes and paraffins can penetrate, if the viscosity is suitable through wood pits and more or less fill the larger pores (lumen) in the wood. Oils and waxes are mostly non-polar fatty esters and paraffins (Figure 1.47) and the latter may be heated to achieve a suitable viscosity. It may be more difficult for products like solid *carnuba oil* to penetrate into wood, but if they can be made less viscous (by heating or as an emulsion) they can be favourable because of less exudation during use.

The hydrophobic nature and fairly large molecular size of compounds, such as waxes and triglycerides, are often cited as the reasons for their not entering the hydrophilic cell wall when

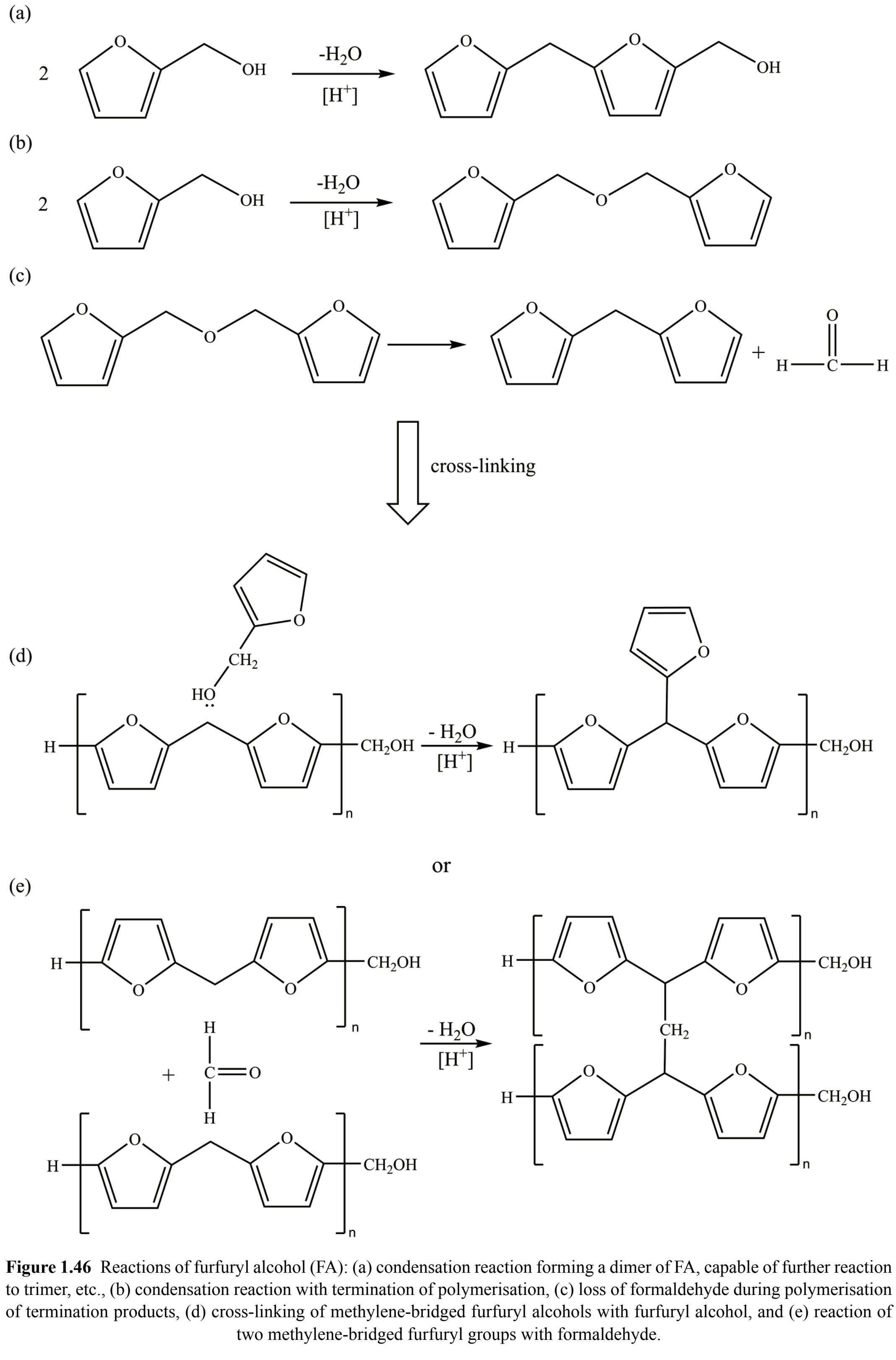


Figure 1.46 Reactions of furfuryl alcohol (FA): (a) condensation reaction forming a dimer of FA, capable of further reaction to trimer, etc., (b) condensation reaction with termination of polymerisation, (c) loss of formaldehyde during polymerisation of termination products, (d) cross-linking of methylene-bridged furfuryl alcohols with furfuryl alcohol, and (e) reaction of two methylene-bridged furfuryl groups with formaldehyde.

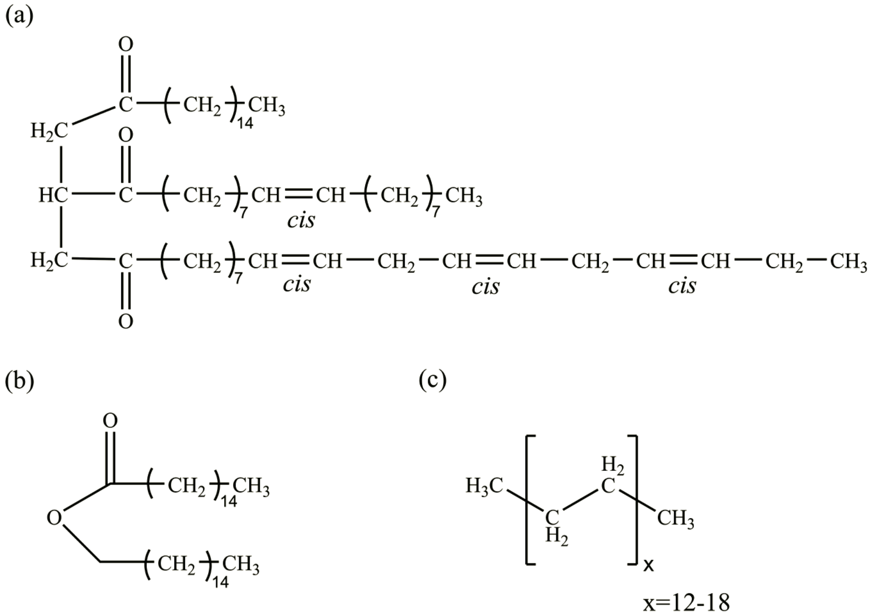


Figure 1.47 Chemical structure of: (a) triglycerides, (b) waxes, and (c) paraffins.

wood is exposed to such agents. Monomers, such as styrene and methyl methacrylate (often used in the plastic industries), may not efficiently penetrate into the cell wall either, despite their small molecular size, probably due to their more hydrophobic nature (Ermeyadan et al., 2014). Exudation from wood impregnated with oily substances may be a problem, especially when they are exposed to varying temperature conditions. This is because the drying/curing of oils (the formation of a cross-linked covalently bonded network between unsaturated hydrocarbon chains as shown in Figure 1.39) is slow due to limited access to oxygen inside the wood, as well as the presence of lignin (Salehi, 2012). Ester bonds are sensitive to both alkaline and acid conditions and to enzymes. Fats are known to be consumed under mild thermal wood modification conditions (Nuopponen et al., 2004). Creosote oils are coal distillates containing polyaromatic hydrocarbons and phenols, and they have been used to impregnate wooden poles and railway sleepers, but they will be phased out in many countries in the coming years due to their toxicity and are not considered in this book.

Polyethylene glycol (PEG) $(\text{-CH}_2\text{CH}_2\text{O-})_n$ is basically a back-bone chain condensation product of ethylene glycol ($OHCH_2CH_2OH$) with various aliphatic chain lengths, producing a range of hydrophilic characters and solubilities. If a suitable molecular size of PEG is chosen, the cell wall can be impregnated. However, as it is applied in a water-soluble form, it will be leached out when exposed to weathering or other water-treatment conditions (Wahlström and Lindberg, 1999).

Chitosan is a polymeric product formed by de-acetylation and partially hydrolysis of the glucosidic bond of chitin, which is found in the shells of crabs, crayfish, shrimps and prawns. Chitosan consists of glucose amine and varying amounts of N-acetyl glucose amine units glucosidically bonded to each other $\beta(1\rightarrow4)$ (Figure 1.48).

The maximum size of a particle able to pass through a pit membrane is about 200 nm and into a cell wall 100 times smaller when the cell wall is in the swollen state. Commercial preparations of chitosan have a size in the range of 10–60 nm and it is therefore assumed that chitosan is deposited mostly in the lumen. The material has only a minor influence on wood swelling but it has fungicidal properties. It is possible that, when dissolved under acidic water conditions, the resulting protonated amino groups (-NH_3^+) in the chitosan will more easily interact with the cell walls of fungi, but also attach with ionising groups (carboxylic) in the wood constituents.

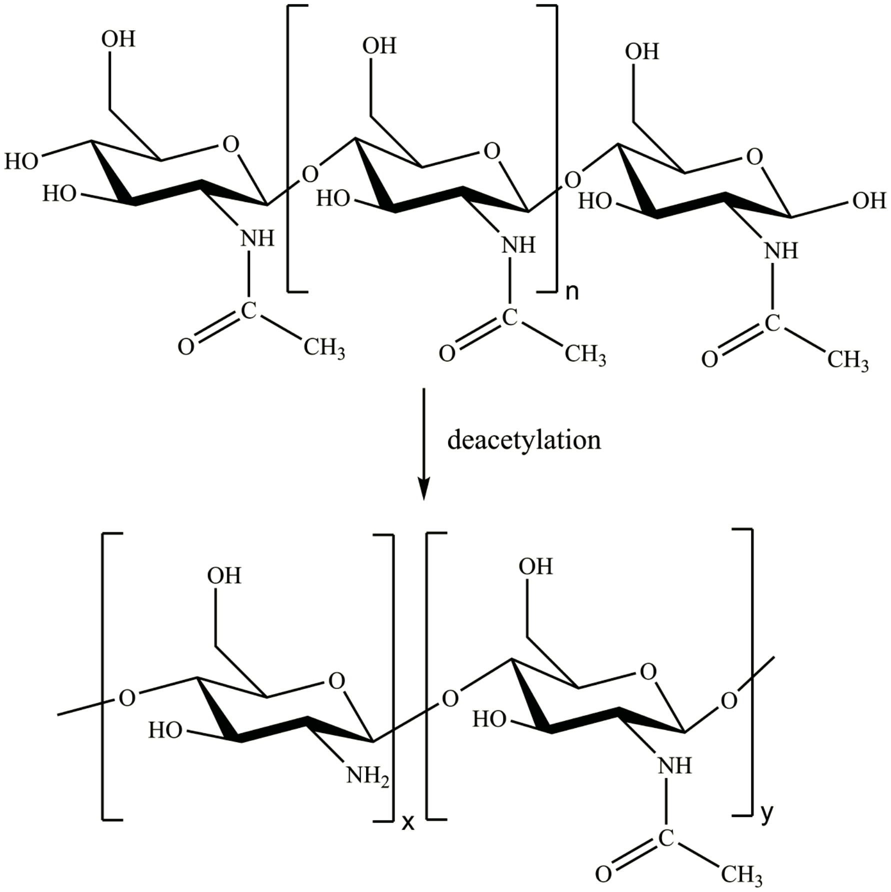


Figure 1.48 Deacetylation of chitin to chitosan.

Water-borne amino-plastic resins, such as melamine-urea-formaldehyde (MUF), can be used as adhesives but also as modifying agents in wood products (Hansmann et al., 2006), especially with low molecular MF-resin, such as metholylated melamines (Figure 1.49).

During impregnation, the resin fills the lumen and other larger pores and, if it is not too strongly cured, it can penetrate into the bulk of the cell wall where it can condense with itself forming a three-dimensional hardened structure (Norimoto and Gril, 1993; Lukowsky, 1999). In contrast to phenolic resins, melamine-urea formaldehyde (MUF) resin is not considered to react with wood components but it gives a rather brittle modified product. Careful control of the curing conditions is necessary to avoid the emission of formaldehyde.

Another commercial process, the Indurite™ process, involved the treatment of wood (radiata pine) with a mixture of starch and an amino-plastic resin. It can be considered to have been a passive process, although it was suggested that small amounts of cross-linker, such as formaldehyde or the dialdehyde glutaraldehyde ($OHC-(CH_2)_3-CHO$), needed to be added in order to improve the wood properties.

1.4 The wood-water relationship

As a biological material, a tree has an inherent need for water to facilitate its growth and sustenance. The same is true of wood-destroying mechanisms, which also depend on the presence of sufficient quantities of water to allow their bio-degradative mechanisms (Engelund Thybring et al., 2018). It is when felled timber is processed for use in construction that its usual natural parameters are changed.

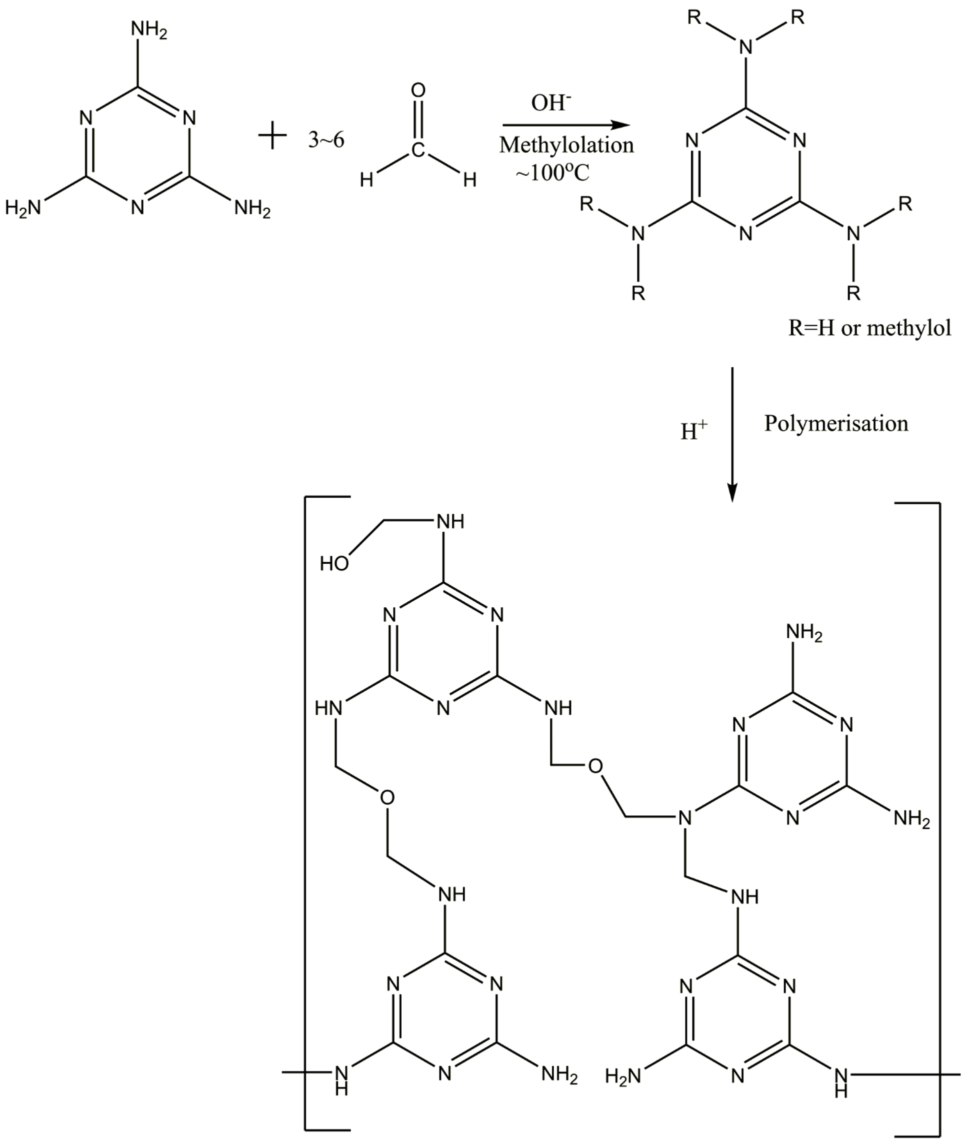


Figure 1.49 Melamine resin formation.

This section considers the relationship between wood and water in its natural and processed states, and how the performance of wood can be compromised through poor design. Methods for assessing the amounts of water present are also considered, together with the effects of wood modification on moisture levels.

Water in wood

The interaction of wood and water was clearly described by Skaar (1988):

> *Wood is a hygroscopic material, and its mass, dimensions and density, as well as its mechanical, elastic, electrical, thermal, and transport properties are affected by its moisture content. Wood is formed in a water-saturated environment in the living tree, but most of the water is removed prior to use. In use its moisture content and dependent properties change with changes in ambient conditions, particularly relative humidity. Wood is anisotropic with respect to most of its physical*

properties. The thermodynamics of moisture sorption, including enthalpy, free energy and entropy changes, are moisture dependent. Water sorption by wood is treated in terms of both surface and solution theories. Moisture transport in wood is also treated, particularly in relation to drying.

Within wood science, the moisture content is most commonly determined by the ratio:

$$\omega = \frac{m_w}{m_{dry}} \tag{1.1}$$

where ω ($kg \cdot kg^{-1}$ or often in %) is the moisture content, m_w is the mass of water, and m_{dry} is the dry mass. Since water in wood can be present both in cell walls and in the macro-void structure (i.e., mainly lumen), the maximum moisture content is the sum of the amounts of water present in cell walls and macro-voids.

Freshly cut and never-dried wood is usually known as "green" wood. In this state, the cell walls are water saturated and, in addition, water is found as a liquid, liquid–vapour mixture or vapour in cell lumens (Figure 1.50). The moisture state of green wood is not a unique quantity; it varies between tree species, within the tree, for example in the sapwood or heartwood, between seasons (if in a temporal zone) and possibly also with the time of day. This is clearly shown in Table 1.5, which gives an overview of a range of broad-leaved and conifer tree species (Glass and Zelinka, 2010).

The moisture content of the heartwood in conifers is typically much lower than that in sapwood. This is not always the case in broad-leaved trees, where the relation between moisture content in heartwood and sapwood depends not only on the species but on the season (Pallardy and Kozlowski, 2008).

As shown in Figure 1.50, when wood is dried, there is a reduction in the moisture content, and the cell walls eventually reach an unsaturated state. The moisture content at the transition point from saturated to unsaturated state, when all of the free water has evaporated is defined as the fibre saturation point (FSP). As the drying proceeds, the wood shrinks, and the reverse occurs if the wood is rewetted. The degrees of shrinkage depend upon the direction in relation to the direction of the grain, the shrinkage (swelling) being greatest in the tangential direction of the growth rings, and about half as much in the radial direction across the rings. The swelling is very low in the longitudinal direction. Tables of dimensional stability are available in the scientific literature (e.g., Glass and Zelinka, 2010), whilst reviews of moisture in wood (e.g., Engelund Thybring et al., 2013) provide details of studies and current understanding.

It is well known that the components of wood are capable of interacting with water through hydrogen bonding, the levels of which have been studied by several groups and compiled in Table 1.6 (Engelund Thybring et al., 2013; Engelund Thybring et al., 2020). The potential availability of OH groups is shown schematically in Figure 1.51, although maximum availability can never be achieved, especially when in situ within the wood cellular structure.

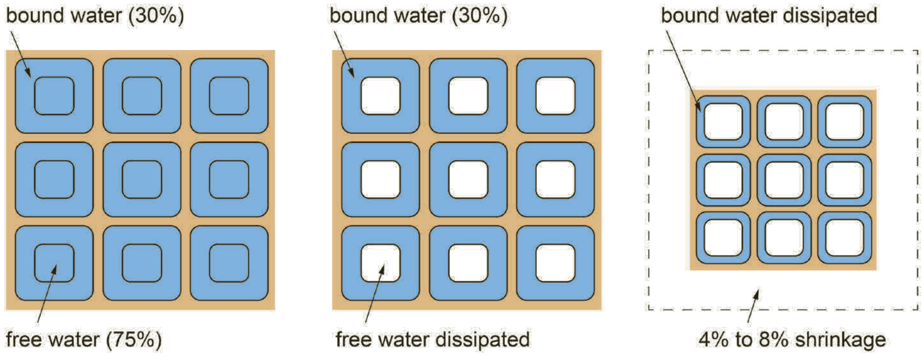


Figure 1.50 Diagram showing the drying process of green wood (green wood to the left, dry wood to the right).

Table 1.5 Average moisture contents of green wood mainly from North American species (based on data from: Glass and Zelinka, 2010). MC − moisture content, HW − heartwood, SW − sapwood.

Species	**Broad-leaved trees**	**MC (%) in:**		**Species**	**Conifer trees**	**MC (%) in:**	
		HW	**SW**			**HW**	**SW**
Alder	Red alder	-	97	Cedar	Eastern red cedar	33	-
Apple	Apple	81	74		Incense cedar	40	213
Ash	Black ash	95	-		Port Orford-cedar	50	98
	Green ash	-	58		Western red cedar	58	249
	White ash	46	44		Yellow cedar	32	166
Aspen	Aspen (Europe)	-	110	Cypress	Bald cypress	121	171
	Aspen (USA)	95	113	Douglas fir	Douglas fir (coast)	37	115
Basswood	American basswood	81	133	Fir	Balsam fir	88	173
Beech	American beech	55	72		Grand fir	91	136
Birch	Paper birch	89	72		Noble fir	34	115
	Silver birch	-	70		Pacific silver fir	55	164
	Sweet birch	75	70		White fir	98	160
	Yellow birch	74	72	Hemlock	Eastern hemlock	97	119
Cherry	Black cherry	58	-		Western hemlock	85	170
Chestnut	American chestnut	120	-	Larch	Tamarack	49	-
Cottonwood	Cottonwood	162	146		Western larch	54	119
Elm	American elm	95	92	Pine	Loblolly pine	33	110
	Cedar elm	66	61		Lodgepole pine	41	120
	Rock elm	44	57		Longleaf pine	31	106
Hackberry	Hackberry	61	65		Ponderosa pine	40	148
Hickory	Bitternut hickory	80	54		Red pine	32	134
	Mockernut hickory	70	52		Scots pine	35	134
	Pignut hickory	71	49		Shortleaf pine	32	122
	Red hickory	69	52		Sugar pine	98	219
	Sand hickory	68	50		Western white pine	62	148
	Water hickory	97	60	Redwood	Redwood	86	210
Magnolia	Magnolia	80	104	Spruce	Black spruce	52	113
Maple	Silver maple	58	97		Engelmann spruce	51	173
	Sugar maple	65	72		Norway spruce	37	133
Oak	California black oak	76	75		Sitka spruce	41	142
	Northern red oak	80	69				
	Southern red oak	83	75				
	Water oak	81	81				
	White oak	64	78				
	Willow oak	82	74				
Sweetgum	Sweetgum	79	137				
Sycamore	American sycamore	114	130				
Tulip tree	Yellow poplar	83	106				
Tupelo	Black tupelo	87	115				
	Swamp tupelo	101	108				
	Water tupelo	150	116				
Walnut	Black walnut	90	73				

Table 1.6 OH groups present in different wood polymeric components (Engelund Thybring et al., 2013).

Wood polymer	Formula unit	Molecular mass (g/mol)	OH-groups present	OH concentration (mmol/g)
Cellulose	$C_{12}H_{20}O_{10}$	324	6[f]	18.5[f]
Xylan[a]	$C_{33}H_{52}O_{24}$	833	12	14.4
Glucomannan[b]	$C_{30}H_{44}O_{24}R_6$[g]	795–1,049	9–15	8.6–18.8
Lignin[c]	$C_{160}H_{178}O_{58}$	3,029	24	7.9
Lignin[d]	$C_{278}H_{300}O_{96}$	5,177	29	7.5
Lignin[e]	$C_{301}H_{335}O_{110}$	5,713	45	7.9

Formulae derived from (a) and (b) Sjöström, 1993, (c) Adler, 1977, (d) Sakakibara, 1980, and (e) Reid, 1995

[f] only 33% of cellulose OH groups are accessible to water. This gives a water-accessible OH concentration *in situ* of cellulose of 6.1 mmol/g

[g] R = CH_3CO or H

Moisture of wood in service

Moisture is more or less omnipresent in wood since water molecules can be absorbed from the surrounding air, but the relative humidity of the air may be affected *inter alia* by the season, daily temperature variations, heating regimes within buildings, etc. Wood in use can also become wet in direct contact with liquid water, as typified by precipitation in various forms, such as rain, hail or snow. Wood used outdoors without shelter is, therefore, frequently exposed to precipitation, which can be further intensified in the form of wind-driven rain (Nore et al., 2007; Abuku et al., 2009; Barreira and de Freitas, 2013) and splash water (Glass and TenWolde, 2007; Bornemann et al., 2014).

There is almost permanent wetting if the material is in direct contact with freshwater, sea water or moist soil. The European Use Class (UC) system according to EN 335 (CEN 2013) does not distinguish between exposure to fresh water and soil contact, although the Use Classes are defined by moisture conditions and potential degrading organisms in a specific use condition. According to EN 335, the Use Classes are defined as shown in Table 1.7.

Based on the definitions in Table 1.7 and experience in monitoring timber in use, Niklewski et al. (2017) suggested the following moisture content limitations for various Use Classes:

- UC2: the moisture content is occasionally > 20%, with a median of < 17.5%
- UC3.1: the moisture content is frequently > 20%, with a median of 20 ± 2.5%
- UC3.2: the moisture content is frequently > 20%, with a median of 25 ± 2.5%
- UC4: the moisture content is rarely or never < 20%, with a median > 27.5%

Decay risk

It is well known that the continued exposure of wood to high levels of moisture can increase the risk of biological decay, particularly due to fungal attack. Niklewski et al. (2017), showed that the decay risk for wood in different Use Classes can alter the overall performance of the wood component and risk its premature failure. The Use Classes, as defined by EN 335, refer to the decay risks associated with different wood-destroying organisms, and should be used in conjunction with Service Class definitions in EN 1995-1-1, i.e., Eurocode 5 (CEN, 2010), which define strength values and can be used to calculate deformations of structural timber members under defined environmental conditions, as shown in Table 1.8.

The decay risk is significantly affected by the macro- and micro-climate. The local influences can be seen in the Use Classes determined for various parts of a road bridge in Spain, showing how localised cover can have a significant effect on the observed Use Class (Figure 1.52). This study by Lorenzo (2016) indicated the variation in performance on either side of the bridge, emphasising the north-south variation in material service life.

Early work into better understanding these effects focused on conditions in the USA (Scheffer, 1971), where four different sites were initially evaluated in terms of the effects of temperature and

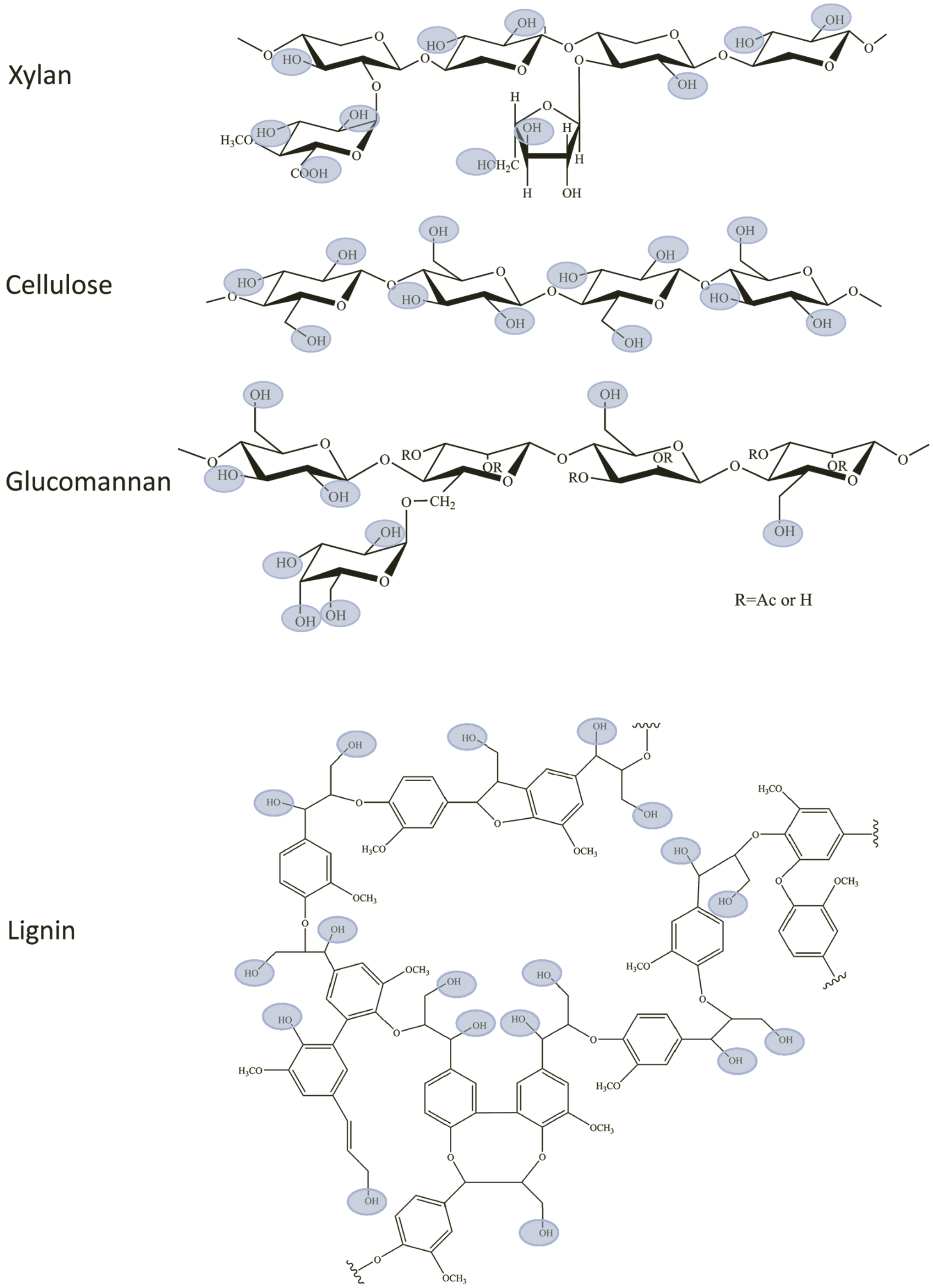


Figure 1.51 Overview of potentially available OH groups (Engelund Thybring, 2014).

local rainfall on the hazard potential of timber and classified according to the Scheffer's Climate Index:

$$\text{Climate index} = \frac{\Sigma_{Dec}^{Jan}[(T-35)(D-3)]}{30} \tag{1.2}$$

Table 1.7 Overview of Use Class classifications according to EN 335 (CEN, 2013).

Use Class	Definition according to EN 335
1	Situations in which the wood or wood-based product is inside a construction, not exposed to the weather and wetting. The attack by disfiguring fungi or wood-destroying fungi is insignificant and always accidental. Attack by wood-boring insects, including termites, is possible although the frequency and importance of the insect occurrence depends on the geographical region.
2	Situations in which the wood or wood-based product is under cover and not exposed to the weather (particularly rain and driven rain) but where occasional, but not persistent, wetting can occur. In this Use Class, condensation of water on the surface of wood and wood-based products may occur. Attack by disfiguring fungi and wood-destroying fungi is possible. Attack by wood-boring insects, including termites, is possible although the frequency and importance of the insect risk depends on the geographical region.
3	Situations in which the wood or wood-based product is above ground and exposed to the weather (particularly rain). Attack by disfiguring fungi and wood-destroying fungi is possible. Attack by wood-boring insects, including termites, is possible although the frequency and importance of the insect risk depends on the geographical region1). A large variety of in-use situations exist and, when relevant, Use Class 3 may be divided into two sub-classes Use Class 3.1 and Use Class 3.2.
3.1	In this situation, the wood and wood-based products will not remain wet for long periods. Water will not accumulate.
3.2	In this situation, the wood and wood-based products will remain wet for long periods. Water may accumulate.
4	A situation in which the wood or wood-based product is in direct contact with ground and/or fresh water. Attack by disfiguring fungi and wood-destroying fungi is possible. Attack by wood-boring insects, including termites, is possible although the frequency and importance of the insect occurrence depends on the geographical region.
5	A situation in which the wood or wood-based product is permanently or regularly submerged in salt water (i.e., sea water and brackish water). Attack by invertebrate marine organisms is the principal problem, particularly the warmer waters where organisms such as *Limnoria* spp., *Teredo* spp. and *Pholads* can cause significant damage. Attack by wood-destroying fungi and growth of surface moulds and staining fungi is also possible. The portion of certain components above water, for example harbour piles, can be exposed to wood-boring insects.

Table 1.8 Service Classes according to EN 1995-1-1 (CEN, 2010).

Service Class	Description
1	Characterised by a moisture content in the materials corresponding to a temperature of 20°C and the relative humidity of the surrounding air exceeding 65% for only a few weeks per year
2	Characterised by a moisture content in the materials corresponding to a temperature of 20°C and the relative humidity of the surrounding air exceeding 85% for only a few weeks per year
3	Characterised by climatic conditions leading to higher moisture contents than in Service Class 2

where T is the mean day temperature of the month (in Fahrenheit) and D is the number of days with more than 2.5 mm (0.1 inch) of rain per month. The original data showed that the decay hazard ranged from 0.0 for Yuma, Arizona to 137.5 for West Palm Beach, Florida, and that within continental USA three distinct climate zones were noted, indicating three levels of above-ground decay potential. The concept of the Scheffer’s Climate Index has been further applied to a range of regions, including Europe, as shown in Figure 1.53.

As these studies have developed and expanded, the effect of wind-driven rain has become more apparent and this has led to its inclusion in hazard assessment. As a result, the relationships between total rain fall, rain fall intensity, wind speed, and wind direction have been assessed to

Figure 1.52 Examples of Use Classes from a road bridge in Spain (courtesy of David Lorenzo).

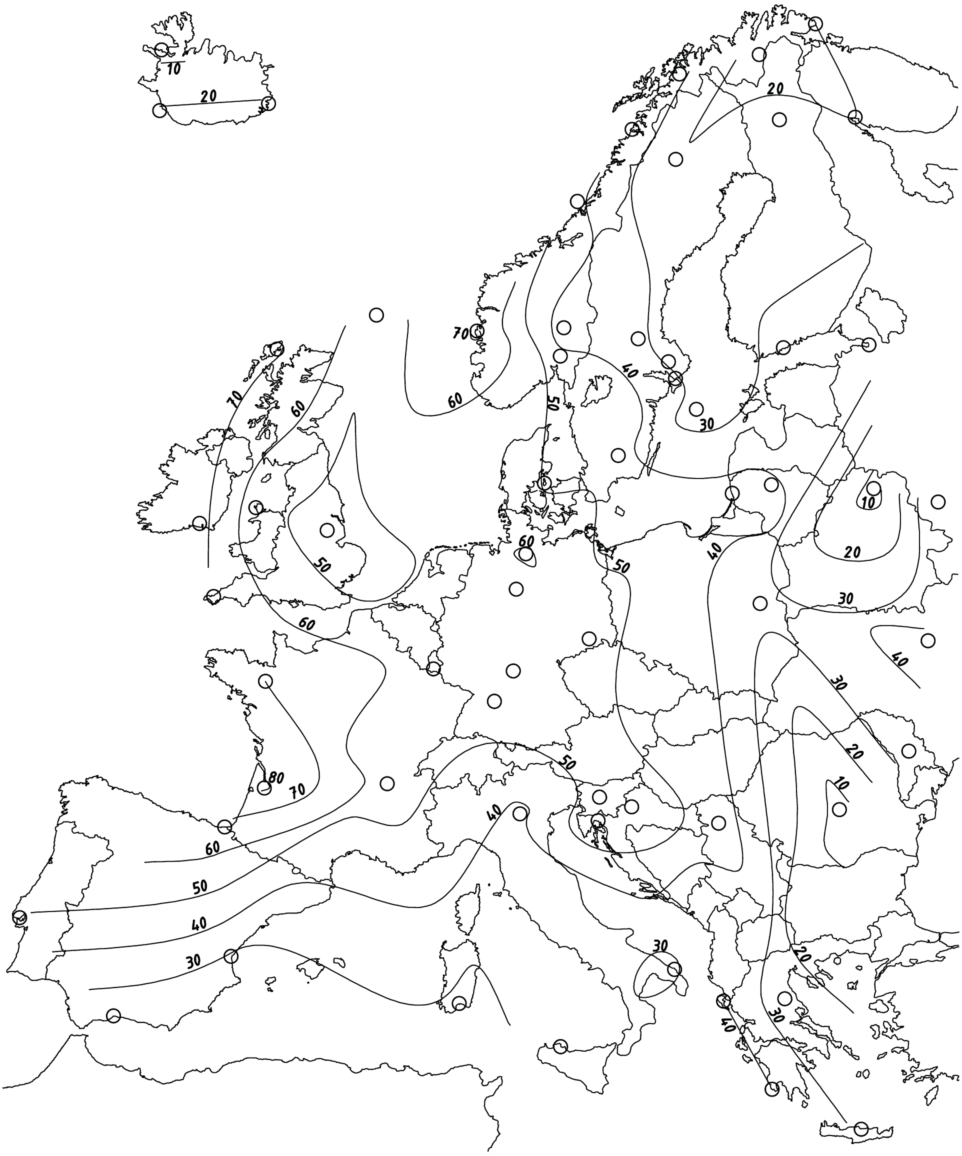


Figure 1.53 Relative decay potential for Europe defined in terms of Scheffer's Climate Index for various European sites (Brischke et al., 2011a).

create wind-driven rain maps. These studies are helping in developing better methods for assessing risks to timber components in use (e.g., cladding). A decay-risk model based on laboratory data was used to estimate wood decay across Europe (Viitanen et al., 2010). As a result, it was possible to determine the mass of Scots pine sapwood damaged as a result of brown rot decay related to the level of exposure to rain (Figure 1.54) and for similar wood samples protected from rain (Figure 1.55). Since the data in Figure 1.54 was based only on relative humidity and temperature data, no capillary uptake of moisture could be attributed via this model. The model appeared to deliver conservative results for sheltered wood. When wood was protected from rain—and provided there was no external moisture source—a lower loss of mass was expected from a biological viewpoint since the presence of liquid water inside wood was an essential requirement for degradation by fungi.

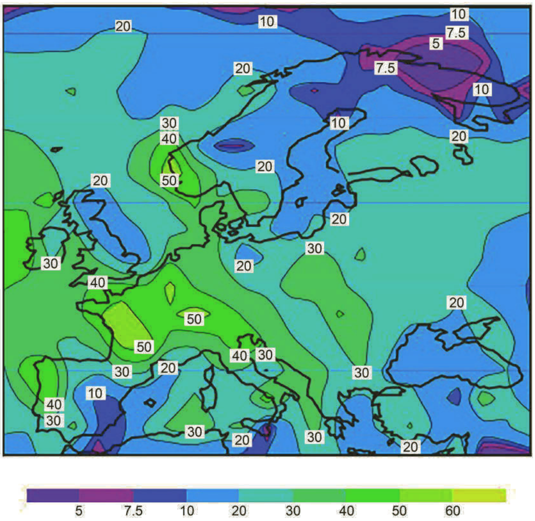


Figure 1.54 Modelled percentage mass loss of small pieces of Scots pine sapwood exposed to rain over a period of 10 years (1961–1979) in Europe (Viitanen et al., 2010).

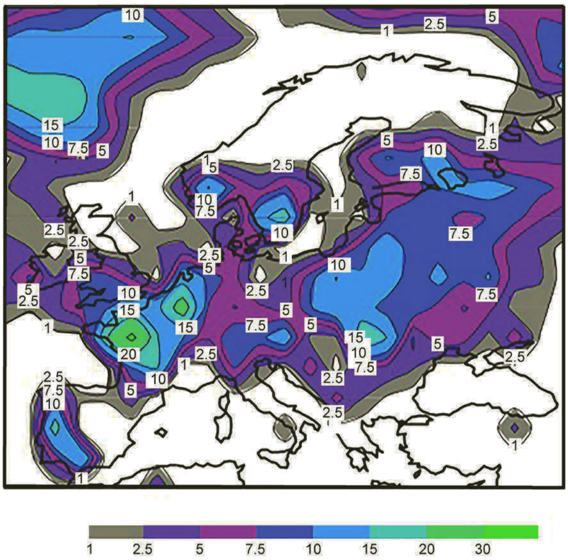


Figure 1.55 Modelled percentage mass loss of small pieces of Scots pine sapwood protected from rain over a period of 10 years in Europe (Viitanen et al., 2010).

Frühwald Hansson et al. (2012) developed a dose-based decay hazard map, further describing the relationship between wood moisture content and temperature and weather conditions, relative humidity, temperature and precipitation. The mapping was based on data collected from 206 sites across 38 European countries, standardised with respect to a fixed location, selected to be Uppsala in Sweden. Values below 1.0 were deemed to have a lower decay potential than Uppsala, and higher values a greater decay potential.

Comparison of data from different sites and different countries allowed for isoplethic mapping, as shown in Figure 1.56, where the dark red colour (e.g., western Ireland, north-west Spain) depicts

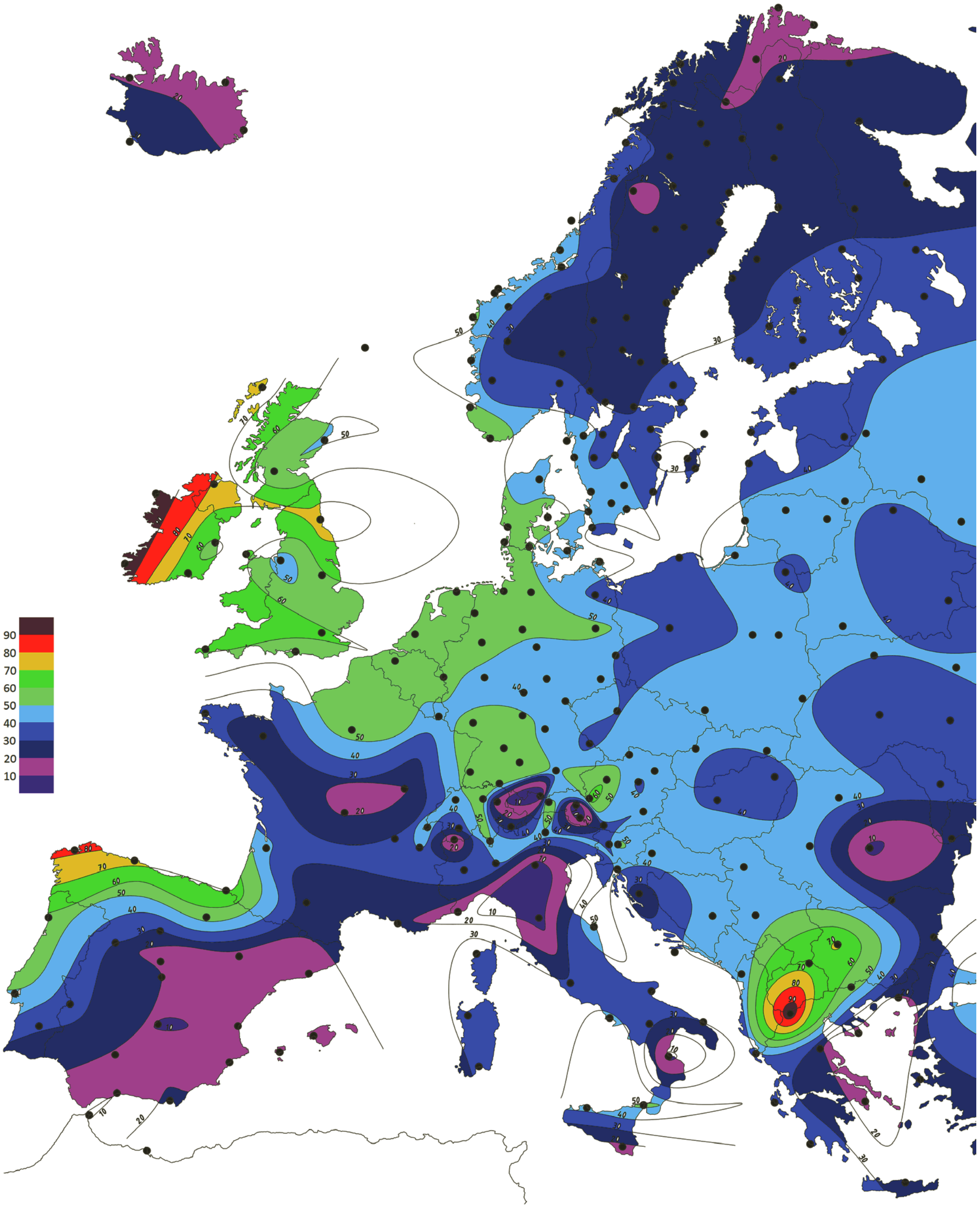


Figure 1.56 Relative decay potential in Europe relative to that in Uppsala, Sweden (Frühwald Hansson et al., 2012).

the areas of greatest decay risk and dark blue (e.g., most of Sweden and Finland) those of lowest decay risk.

Determining moisture in wood

It has already been pointed out that wood is a hygroscopic construction material. This means that the material constantly strives to remain in equilibrium with the local climate, i.e., the relative humidity (RH) and the temperature. The equilibrium moisture content (EMC) is defined as the moisture content of the wood when it is in equilibrium with the local climate conditions.

If the moisture content of the wood is higher than the equilibrium moisture content, the wood will dry out and if it is lower the wood will take up moisture. When the moisture content changes, below the fibre saturation point, the wood changes its volume, depending on whether moisture is being released or absorbed, the wood shrinks or swells. The dimension, strength and resistance to decay are key properties of wood that are affected by moisture.

The atmospheric content of water vapour, its vapour concentration, is usually stated in grams of water per cubic metre air and it varies throughout the year, depending on the climatic conditions in the particular region. For example in Sweden, the vapour concentration outdoors is at its highest in the summer (9–11 grams per cubic metre) and lowest in the winter (3–5 grams per cubic metre)—while the relative humidity and, thus, the wood's equilibrium moisture content in outdoor conditions is lowest in the summer (65–75% and 11–15% respectively) and highest in the winter (90–95% and 19–23% respectively).

Physically, the relative humidity is the ratio between the actual water vapour's partial pressure of the air and its saturation pressure at the temperature in question. The relative humidity of the air indoors in a heated room is highest in summer (45–60%) and lowest in winter (10–25%). The colder it is outdoors, the drier the air indoors. The moisture content in wood, both indoors and outdoors, adapts to the relative humidity and temperature of its surroundings. In heated Swedish homes in mid Sweden, the moisture content in wood averages out across the year at 7.5%, with the highest figures in summer (7–12%) and the lowest in winter (2–6%). On average, the indoor climate is drier in the north of Sweden than in the south during winter.

The most accurate yet simple method for measuring the moisture content in wood is through a gravimetric determination before and after drying. In the gravimetric method, which gives the mean value for the moisture content of the tested sample, the weight is first determined, after which the sample is then fully dried in an oven at 103°C, in accordance with EN 13183 (CEN, 2002a). Other more practical, but not as accurate methods exist. For exterior plywood, Van den Bulcke et al. (2009) mounted samples on a load cell and exposed them to climatic conditions, with variations due to rain and wind being taking in account through an inert material mounted onto an adjacent load cell.

Another indirect method of determining the moisture content is by measuring electrical resistance. Typically, wood is an electrical insulator, but the presence of moisture allows electrical current to be conducted. Readings are normally taken using stainless steel nails or screws attached to or inserted into the specimens to serve as electrodes. In some cases (Brischke et al., 2008), electrodes require a non-conductive covering whereby the tops need to be free to guarantee a top to top measurement, as shown in Figure 1.57. Resistive methods allow the moisture content to be determined at a certain position within a test specimen. The most common method used follows that outlined in EN 13183-2 (CEN, 2002b), where electrodes are hammered into the wood surface at a distance 300 mm from the end of a sample, to a depth of 0.3 times that of its thickness. The desire to undertake continual monitoring has, however, led to an increase in the use of the method reported by Brischke et al. (2008), and it has become a common method of analysis in many European research institutes and universities.

Instead of measuring the electrical resistance, it is also possible to determine the moisture content using a capacitance method, as outlined in EN 13813-3 (CEN, 2005). A capacitive electric field forms between the probes and the test material, and a higher moisture content leads to a high

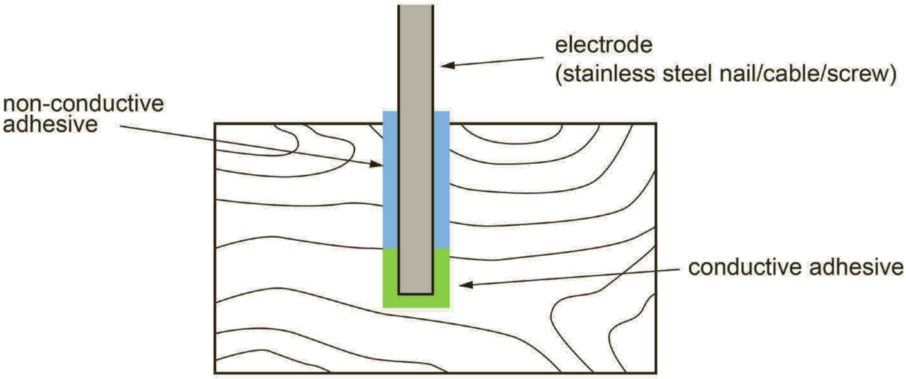


Figure 1.57 Method for connecting electrodes for continuous moisture monitoring of wood (Brischke et al., 2008).

dielectric constant, thus increasing the capacity. The capacitive method is a relative measurement, which means that the test result is given as the difference between the values for the dry and wet materials. Capacitive measurements suffer, however, from a strong relationship between permittivity and wood density, which is often unknown and may vary within a test object.

These methods are summarised in the work of Dietsch et al. (2015), together with other methods, some of which are gaining in popularity and are described below.

Sorption studies with dynamic vapour sorption (DVS) equipment

The rate at which wood adsorbs and desorbs water varies for each species. Never-dried wood, also known as green wood, has a desorption isotherm higher than the adsorption and desorption isotherms of oven-dried wood. The oven-dried desorption isotherm is always higher than the adsorption isotherm (Figure 1.58). The difference between desorption and adsorption curves is known as sorption hysteresis and this exists in many hygroscopic materials (Skaar, 1988). It is expressed as the ratio of the adsorption (A) to the desorption (D) moisture content at a given relative humidity. The A:D ratio ranges from 0.785 to 0.844 (mean value 0.812 ± 0.023) for conifer wood and 0.790 to 0.849 (mean 0.828 ± 0.018) for broad-leaved wood. Sorption hysteresis decreases with increasing temperature and disappears at a temperature of about 75°C (Skaar, 1988).

The theory behind the sorption process has been well documented (e.g., Engelund Thybring, 2013), and the thermodynamics of the process can be explained by the fact that any moisture change

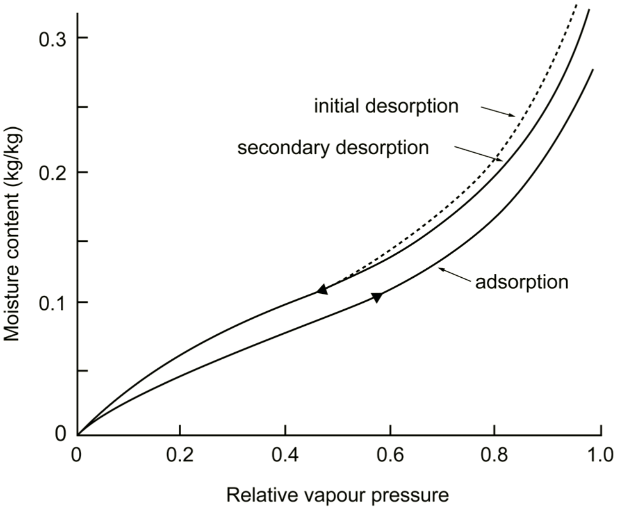


Figure 1.58 Typical adsorption and desorption isotherms for wood.

in wood is usually accompanied by an exchange of heat, heat being generated during adsorption via an exothermic process or consumed during desorption via an endothermic process (Zhao et al., 2013). The overall thermodynamic processes involved were described by Yang and Ma (2016) during their evaluations of the moisture sorption by a hybrid poplar.

Traditionally, one of the problems involved when undertaking sorption isotherm studies was the need to equilibrate samples to fixed relative humidities, using various salt solutions. This time-intensive method was made considerably easier in the 1990s through the development and commercialisation of *dynamic vapour sorption* (DVS) equipment. Since then, DVS has become an integral component of the equipment in most research facilities, whereby samples can easily be tested from one relative humidity to another as well as over a range of temperatures. Many studies of various timber species have been reported, e.g., Sitka spruce (Hill et al., 2010), radiata pine, black wattle and sesendok (Zaihan et al., 2009). Recent work by Uimonen et al. (2019) has demonstrated that it is possible to use DVS to ascertain the accessibility of hydroxyl groups using deuterium oxide as well as using alcohols as inclusion compounds.

X-ray computed tomography (CT)

X-ray computed tomography (CT) is a powerful method for the non-destructive evaluation of three-dimensional wood density and moisture content in wood and other bio-based materials. The density can be determined with an accuracy of about ±3 kg/m^3 for large specimens (sawn-timber dimension), and the accuracy in average moisture content below and above the fibre saturation point can be determined with an accuracy of about ±1 percent point. It is also possible to measure the density and moisture content in a randomly chosen volume element of say 1 × 1 × 1 mm within the wood. CT scanning can also be used for evaluating:

- the development of fungal attack in wood,
- the development of attack by marine borers in wood,
- the distribution and rate of penetration of preservatives or wood modification chemicals into wood, and
- to evaluate wood drying after impregnation.

X-ray CT was introduced in the medical field in the early 1970s to obtain a density profile through a body following the same principle as in other radiographic technologies. The technique was developed by Hounsfield (1973) and Cormack (1963) and they were awarded the Nobel Prize in physiology and medicine in 1979 for their work. This technique is nowadays a standard medical examination method for investigating the possible presence and size of tumours in the brain, for example. This X-ray computed tomography scanning technology has recently been developed as an industrial tool for outer geometry assessment and internal feature detection in logs for the optimisation of the sawing/cutting processes in the sawmill and veneer industries. In wood science, CT is used mostly for steady-state studies of internal anatomical features of the wood material, but it is also possible to study processes such as drying, thermal modification, water absorption, internal and external cracking, and material deformation in a temperature- and humidity-controlled environment.

CT is an imaging technique that measures the amount of X-radiation sufficient to pass through a body of a given material, a property that is defined by the attenuation coefficient of the material. The working principle of an X-ray CT scanner is that X-rays are sent through a material and the intensity of X-ray photons that reach the detector at the other side per unit time is quantified. The X-rays are generated in the X-ray tube, which is in a fixed position in relation to the detectors at the opposite side. In general, the X-ray tube and the detector rotate around the scanned object, but in other CT scanners, the scanned object rotates while the tube and detectors remain stationary, which simplifies the mechanics of the device. After the rotation is completed, a computer can calculate the X-ray attenuation in small volume elements (voxels) within the entire scanned volume (Figure 1.59).

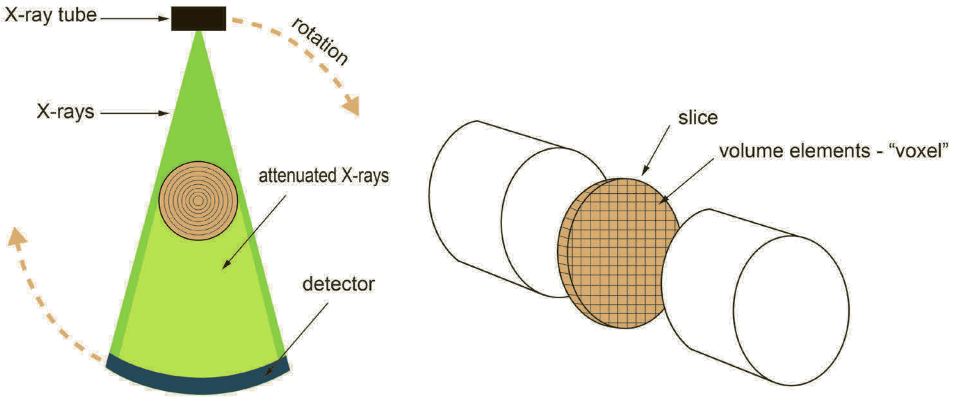


Figure 1.59 The working principle of a CT scanner (left), and voxels within a cross-section (right).

The theoretical background of CT lies within Lambert-Beer’s law, which describe an exponential relationship between the intensity of the radiation and the attenuation coefficient:

$$I = I_0 e^{-\mu d} \tag{1.3}$$

where I is the intensity of the transmitted X-ray beam, I_0 is the intensity of the incident X-ray beam, µ is the linear attenuation coefficient of the material along the transmission path, d is the thickness of the body and e is Euler’s constant.

CT images are presented in a grey scale and, for most biological materials, the grey scale values are almost linearly related to density, being darker for lower density and brighter for higher density.

The linear attenuation coefficient is normalised with respect to the linear attenuation coefficient of water, leading to the CT number or Hounsfield number, according to:

$$CT\ number = \frac{1000 \cdot (\mu_x - \mu_{water})}{\mu_{water} - \mu_{air}} \tag{1.4}$$

where μ_x is the linear attenuation coefficient of the scanned material, μ_{water} is the linear attenuation coefficient of water, and μ_{air} is the linear attenuation coefficient of air. The formula applies for CT scanners with an average photon energy of 73 keV. In Equation 1.4, a CT number of –1,000 is the attenuation value for air, and a CT number of 0 is the value for water.

The image reconstruction results in a map of the inhomogeneity of the cross section in a position perpendicular to the rotation axis. This map can be represented as a two-dimensional raster (also known as bitmap) image of the scanned cross section, formed by pixels with values of the X-ray linear attenuation coefficient. In a CT, one pixel represents a three-dimensional entity (voxel) of the material scanned with the dimensions of the pixel and the thickness of the scanning beam (the beam depth). After the processing, a CT scanner provides a raster image in which the value of each pixel is the average CT number of the voxel which it represents according to Equation 1.4. There is great variability in voxel dimensions and pixel size due to the type of CT, the specific device and the chosen settings.

Although the attenuation coefficient of a material is dependent on the effective atomic number, there is an approximately linear relation between the CT number and the density of wood (Lindgren, 1992), so that the greyscale of a CT image of wood can be interpreted as a density scale. CT images can be calibrated so that white represents water (1,000 kg/m^3) and black represents air (≈ 0 kg/m^3). A water phantom is scanned with the specimen so that the density is defined by the greyscale, a model that is well suited for studying wood and wood-water relations. Figure 1.60 shows how sapwood in the green state is almost white because it is saturated with water, whereas dry wood has a darker grey hue.

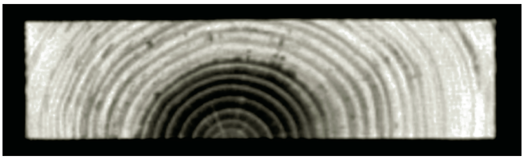


Figure 1.60 CT image of a cross section of Scots pine timber with heartwood (dark) and water filled sapwood.

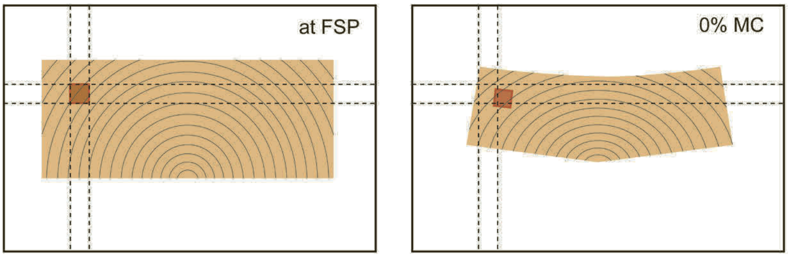


Figure 1.61 Deformation and displacement (exaggerated) relative to the pixel location of the voxel represented by one pixel when the wood piece is oven dried and re-scanned. FSP – fibre saturation point, MC – moisture content.

CT images are used to calculate moisture content by analogy with the gravimetric method. The estimation of the of the moisture content in wood using CT requires two images: one at the moisture content to be determined and another at a known reference moisture content, which here is, for practical reasons, always 0%. For pixel-wise distribution of the moisture content, image-processing algorithms are needed to compensate for the anisotropic distortion that wood undergoes as it dries. It is possible to determine the density of the material represented in any given pixel using the CT numbers of air and water as reference. The CT scanner settings establish the pixel size and scanning depth, and thus the voxel volume. It is then possible to calculate the mass of the material in the pixel and, if the mass is known, it is possible to calculate the moisture content.

Due to shrinkage anisotropy in the wood cross section, the region in a given pixel in the image at a certain moisture content is deformed when the wood is dried, and is then covered by more than one pixel because of the displacement (Figure 1.61). Since the voxels in the two images do not correspond to the same region in the specimen, the MC is determined as the average for the entire specimen. To quantitatively determine the moisture content in the local region, image processing must be performed to match the images (Couceiro, 2019). In the first applications of CT to wood drying, Lindgren (1992) attempted to take into account the swelling and shrinkage of cross-section images of timber by applying a linear transform combined with a bilinear and a non-linear transform to CT images of wood specimens at different moisture-content levels.

A considerable more accurate technique than X-ray to determine moisture in wood is to use neutrons for the detection. The neutron-based techniques are only used in research purposes, and are complicated to perform and require extremely expensive facilities, so they are not further discussed here.

Spectroscopy

Spectroscopy has become a valuable tool in the analytical arsenal of scientists, providing a means of identifying chemical bonds present as a result of their excitation at known frequencies. This technique has been further expanded through computer-aided deconvolution and processing, making identification much easier. Near-infrared (NIR) spectroscopy has applications in all facets of biological material assessments, given its non-destructive nature. NIR spectroscopic information on biological materials is particularly relevant given its ability to show water, which has specific absorption bands at 5,200 cm^{-1} (as a result of the combination of stretching and deformation vibrations for OH) and 6,900 cm^{-1} (due to the first overtone of the OH stretching vibration), and

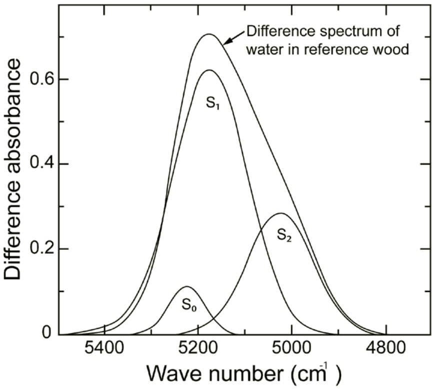


Figure 1.62 Difference and decomposed spectra of water in a hinoki wood sample (Inagaki et al., 2008).

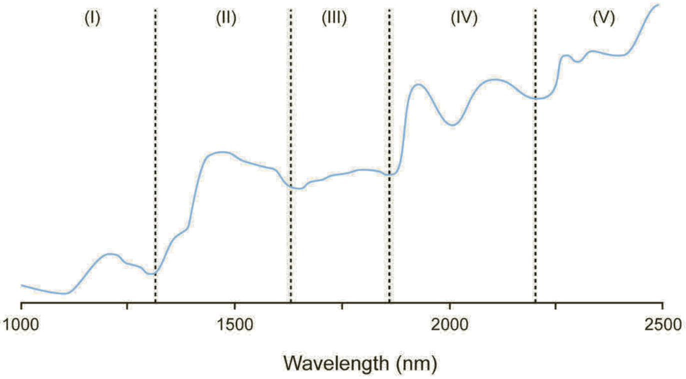


Figure 1.63 NIR spectrum of warty birch wood.

studies of these peaks can provide information on the amount of water present, as was demonstrated in work by Tsuchikawa and Tsutsumi (1998) for adsorptive and capillary-condensed water in wood, and further studies by Buijs and Choppin (1963) suggested that water molecules were classified into three components, free water molecules (S_0), molecules with one OH group engaged in hydrogen bonding (S_1), and molecules with two OH groups engaged in hydrogen bonding (S_2), as shown in Figure 1.62 for hinoki, whereby S_0, S_1 and S_2 components were separated (Inagaki et al., 2008). The effects of hydrogen bonding were found in subsequent work (Maeda et al., 1995) to be associated with two additional peaks (S_3 and S_4 respectively).

More detailed studies in the presence of moisture (Popescu et al., 2016) showed a range of spectral peaks (Figure 1.63) for untreated warty birch, which could be assigned the following chemical characterisations:

I) between 1,100–1,330 nm is mostly assigned to 1st and 2nd overtones of C-H stretching vibrations in methyl and methylene groups from carbohydrates and lignin;

II) between 1,330–1,640 nm is assigned to 1st overtone of the C-H combination bands, and 1st overtone of different O-H stretching vibrations;

III) between 1,640–1,850 nm is dominated by the 1st overtone of the aliphatic and aromatic C-H stretching vibrations and O-H combination bands in all wood components;

IV) between 1,850–2,210 nm is assigned mostly to C=O groups, O-H stretching and deformation vibrations and also to Car-H and C-H stretching vibrations, and

V) between 2,210–2,510 nm is assigned mostly to C-H stretching and deformation vibrations.

Wood modification and moisture

The ultimate aim of wood modification is to alter its performance and reduce the risks in service, particularly with regard to dimensional in stability and decay, both of which are strongly influenced by the presence of moisture. Wood in service in interior conditions is usually restricted to moisture contents below 10%, but design or exposure to high moisture conditions can significantly affect its performance. The same is true of wood in Use Classes 2 and 3, where the moisture content can often exceed 20% due to atmospheric conditions. The risk of decay is then increased, particularly if the exposure is over a prolonged period of time.

As was shown in Figure 1.51, the key components of wood contain large numbers of potentially accessible hydroxyl groups. These groups present a means by which modification can occur, either through direct chemical bonding or indirect bonding via hydrogen bonding of compounds that have been impregnated and subsequently polymerised, or as a result of a loss of functionality of the wood cell wall components. The mode of operation of the modifying agent can relate to one or more of these actions, as suggested in Figure 1.40. The key result of wood modification is, however, most often a decrease in the number of hydroxyl groups available for subsequent interaction with moisture in the environment. The higher the level of modification, the greater is the effect on the hydroxyl groups within the cell-wall components and hence on their potential interaction with moisture.

A comprehensive review by Engelund Thybring, 2013 has assessed the decay risk according to levels of moisture exclusion efficiency (MEE) and anti-swelling efficiency (ASE) and ASE* (an alternative measure of ASE, where the volume increase resulting from various wood modification methods has been deducted from the dry volume of the unreacted wood). Through the analysis of modification methods undertaken (Table 1.9), it was possible to estimate threshold levels for MEE, ASE and ASE* as well as the respective weight gain required for each treatment (a weight loss when considering thermal modification).

More detailed information on the effects on the moisture content of various modification methods is given in the sections on the respective modifications.

Table 1.9 Estimated threshold conditions for decay in various wood modifications (Engelund Thybring, 2013). WPG − weight percentage gain, MEE − moisture exclusion efficiency, and ASE − anti-swelling efficiency.

Modification	**Threshold (WPG)**	**MEE**	**ASE**	**ASE***
Acetylation	20%	42%	63%	60%
Furfurylation	35%	40%	74%	-
DMDHEU	25%	43%	45%	43%
Glutaraldehyde	10%	24%	50%	48%
Glyoxal	> 50%	-	-	-
Thermal modification	–15%	42%	46%	-

1.5 Wood biodegradation

Wood can be decomposed by a wide variety of biological systems, provided suitable environmental conditions are present. Wood is inherently biologically degradable and may be attacked by fungi, bacteria, and insects individually or in combination, based on a variety of metabolism mechanisms linked to the individual components present within the wood. Wood in its natural environment is known to undergo colonisation by these microorganisms and insects, particularly when the bark, the external protection of the tree has become damaged, allowing more accessible attack and accelerating decomposition. The same is true of felled round timber lying on the forest floor, further assisted by the timber being exposed to super-saturation conditions. The cleavage of the main macromolecules present within wood—cellulose, lignin and hemicelluloses—leads to smaller oligomer or single unit components, which are easier to degrade as a food source, with such digestion leading to the basic components of all flora—carbon dioxide and water—whilst providing energy to the organism that has digested the wood material residue. While this degradation process is an important feature of forest regeneration, it is a problem when wood is used in the built environment, since such degradation in buildings can lead to premature failure. This has led to the need to protect wood in construction and in other applications through the use of correct detailing, coatings, wood preservation and, more recently, wood modification.

Wood as a substrate for microorganisms

As expected, the structural features of wood depend on the species in question. However, all wood cells are composed of cellulose, hemicellulose, and lignin in various ratios. In general, broad-leaved wood have a lower lignin content than conifers, and the type of lignin monomer in the woods is different. Both syringyl and guaiacyl units (Figure 1.38) are found in angiosperm lignin, whilst only guaiacyl-type lignin is present in gymnosperms. There are contrasting differences in the hemicelluloses of the respective tree groups, with galactoglucomannans (Figure 1.37, middle) dominating in gymnosperms and glucuronoxylans (Figure 1.64) in angiosperms.

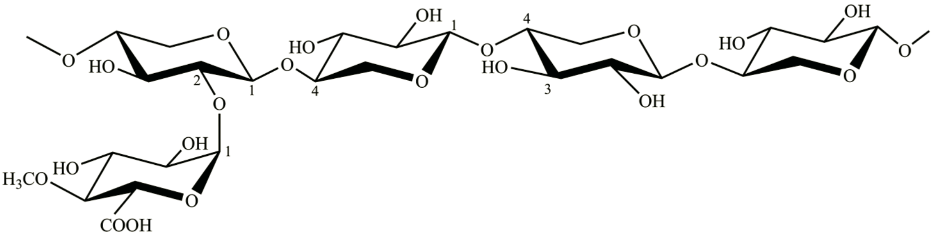
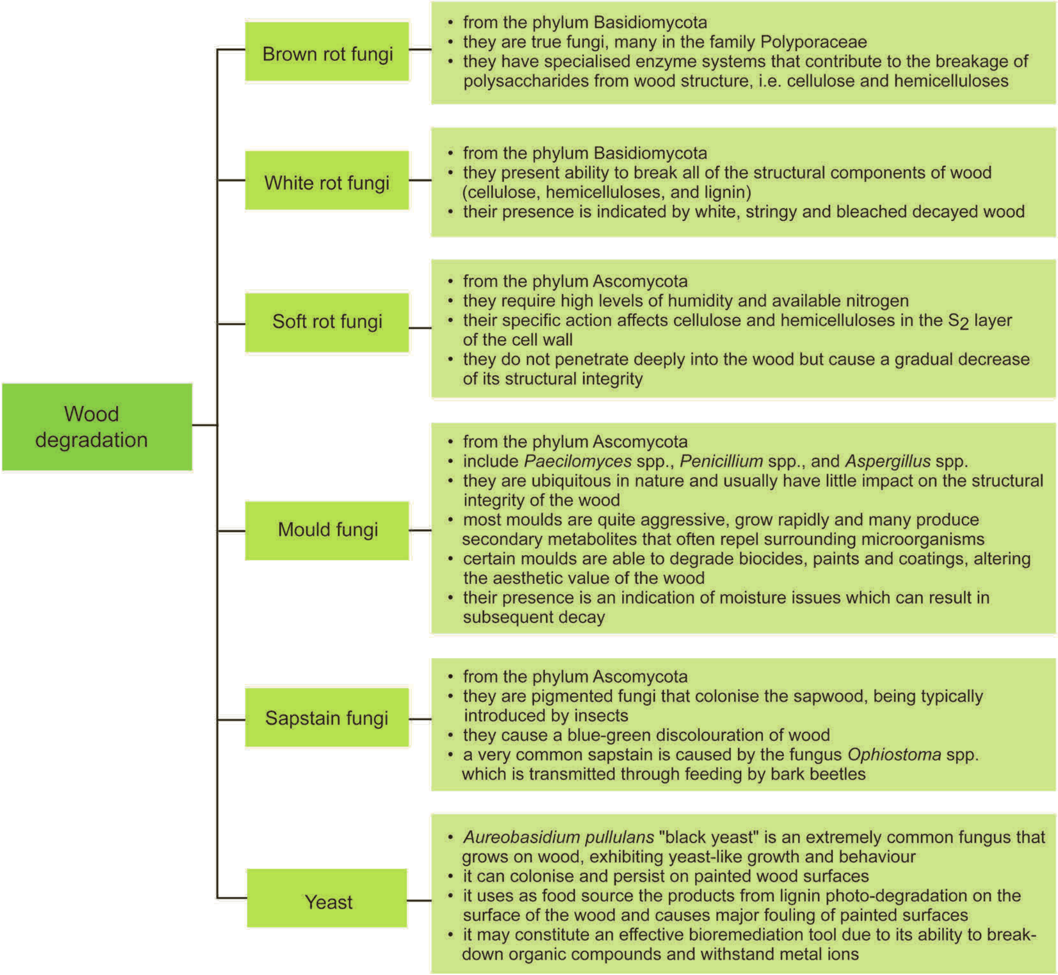


Figure 1.64 Structure of 4-O-methyl-D-glucurono-D-xylan in European beech (Strnad et al., 2013).

The greatest risk of germination and growth in fungal decay occurs when there is a suitable balance of five key factors: source of infection, suitable substrate (food), moisture, oxygen and temperature. The presence of the three main components of wood provide the necessary food for the attacking organism so that, if there is sufficient moisture and a suitable temperature, decay may be possible if colonisation occurs at sites of weakened resistance, such as damage to the bark of the tree. Many timber species produce a range of extractives as a natural protection against biological attack, but these are usually located within the heartwood of the tree, which can leave the outer sapwood more prone to decay. Preventing this has usually been undertaken with wood preservation, where a compound toxic to attacking organisms is introduced. However, wood modification is directed more to a reduction in the available moisture, so limiting one of the five key factors for decay.

Biological degradation of wood by fungi

The decomposition of wood by fungi is of two main types, often referred to as *brown rot* and *white rot*, together with the less common *soft rot* (Eriksson et al., 1990; Zabel and Morrell, 1992; Schmidt et al., 1996; Mohebby, 2003; Srivastava et al., 2013). There are also non-destructive fungi that result only in a discolouration of the timber, with no loss of mechanical strength. It has bed been suggested that these staining fungi help to provide channels of attack for wood-destroying fungal. In brown rot, the cellulose and its related pentosans are attacked while the lignin is more or less unchanged. This causes the attacked wood to darken in colour, undergoing shrinkage and cross-cracking into cubical or oblong pieces that can be readily broken and crumbled between the fingers into a brown powder. In white rot, all the components of the wood, including the lignin, may be decomposed and used by the growing fungus. White rot does not produce cross-cracking, but the wood becomes paler in colour, sometimes in pockets or streaks of various sizes and may eventually become a fibrous whitish mass. With some white rots, however, the cellulose may remain intact, while the lignin in the secondary wall and middle lamella is almost entirely removed. A graphical view of different rot-type fungi as well as moulds is shown in Figure 1.65 (Teacă et al., 2019).



Brown rot fungi

- from the phylum Basidiomycota
- they are true fungi, many in the family Polyporaceae
- they have specialised enzyme systems that contribute to the breakage of polysaccharides from wood structure, i.e. cellulose and hemicelluloses

White rot fungi

- from the phylum Basidiomycota
- they present ability to break all of the structural components of wood (cellulose, hemicelluloses, and lignin)
- their presence is indicated by white, stringy and bleached decayed wood

Soft rot fungi

- from the phylum Ascomycota
- they require high levels of humidity and available nitrogen
- their specific action affects cellulose and hemicelluloses in the S_2 layer of the cell wall
- they do not penetrate deeply into the wood but cause a gradual decrease of its structural integrity

Mould fungi

- from the phylum Ascomycota
- include *Paecilomyces* spp., *Penicillium* spp., and *Aspergillus* spp.
- they are ubiquitous in nature and usually have little impact on the structural integrity of the wood
- most moulds are quite aggressive, grow rapidly and many produce secondary metabolites that often repel surrounding microorganisms
- certain moulds are able to degrade biocides, paints and coatings, altering the aesthetic value of the wood
- their presence is an indication of moisture issues which can result in subsequent decay

Sapstain fungi

- from the phylum Ascomycota
- they are pigmented fungi that colonise the sapwood, being typically introduced by insects
- they cause a blue-green discolouration of wood
- a very common sapstain is caused by the fungus *Ophiostoma* spp. which is transmitted through feeding by bark beetles

Yeast

- *Aureobasidium pullulans* "black yeast" is an extremely common fungus that grows on wood, exhibiting yeast-like growth and behaviour
- it can colonise and persist on painted wood surfaces
- it uses as food source the products from lignin photo-degradation on the surface of the wood and causes major fouling of painted surfaces
- it may constitute an effective bioremediation tool due to its ability to break-down organic compounds and withstand metal ions

Figure 1.65 Overview of wood-destroying fungi and moulds (Teacă et al., 2019).

Since fungi do not contain chlorophyll, they are totally dependent upon extracting the necessary nourishment for growth and reproduction from their surroundings, i.e., from the timber under decay. When decay occurs in the core of a tree, it is in a zone where there is limited access to nitrogen and sulphur, essential components for growth (e.g., through enzymatic needs). This limitation to essential elements, in addition to the common increase in biologically restrictive chemicals as part of a species' natural durability, often limits the amount of decay which may occur the heartwood of a species.

As expected, decayed wood is less dense than sound wood and it usually exhibits a loss of strength, along with changes in firmness and the release of odours. Typically, the loss of weight resulting from brown rot is about 70%, since the lignin still remains, though its total destruction is possible in white rot. Even slight decay can reduce the toughness or shock resistance of wood and allow it to break easily under impact, although it may still appear hard and firm to the touch. Fungi that cause brown rot usually leads to a more rapid drop in most strength properties than those that cause white rot, although both types reduce the toughness of any wood that they attack. The fresh and resinous smell of sound wood is usually replaced by a distinctive mushroom odour as wood decays, and some wood-rotting fungi produce characteristic aromatic or sweet smells. Some examples of fungal species are given in Table 1.10.

Typically, the changes noted in wood as a result of fungal degradation can be summarised as in Table 1.11 (Blanchette, 1998).

Table 1.10 Examples of some discolouring and destroying fungi capable of attacking wood.

Destroying fungi		
Brown rot fungi	**White rot fungi**	**Soft rot fungi**
Basidiomycetes	**Basidiomycetes (ascomycetes)**	**Ascomycetes, deuteromycetes**
Coniophora puteana,[a,b] *Serpula lachrymans*, *Postia placenta*,[a,b] *Gloeophyllum trabeum*,[b] *Gloeophyllum sepiarium*, *Gloeophyllum abietinum*, *Antrodia Vaillantii*	*Trametes versicolor*,[a,b] *Donkioporia expansa*, *Schizophyllum commune*, *Phanerochaete chrysosporum*, *Pleurotus ostreatus*	*Chaetomium globosum*,[c] *Phialophora* spp., *Monodictys* spp., *Humicola grisea*,[c] *Petriella setifera*,[c] *Lechythophora mutabilis*,[c] *Trichurus spiralis*[c]
Staining fungi		**Surface moulds**
Blue stain fungi	**Other stain fungi**	
Ascomycetes, deuteromycetes	**Ascomycetes, deuteromycetes**	**Ascomycetes, deuteromycetes**
Aureobasidium pullulans, *Ceratocystis* spp., *Ophiostoma* spp., *Ceratocystiopsis* spp.	*Discula* spp., *Arthrographis cuboidea*, *Chlorociboria aeruginosa*	*Paecilomyces variotii*, *Aspergillus niger*, *Trichoderma* spp., *Bisporia* spp., *Penicillium* spp.

[a] Test fungus according to CEN/TS 15083-1 (CEN 2015)
[b] Test fungus according to EN 113 (CEN 1996)
[c] Test fungus according to ENV 807 (ENV 2001)

Table 1.11 Changes in wood due to degradation by fungi.

Decay	**Wood characteristics**	**Strength loss**	**Cell-wall components**	**Morphology**
Brown rot (dry rot)	Brown. Cracks and checks when dry, producing cubical fragments.	Large losses of strength in early stages of decay.	Cellulose depolymerisation and loss.	Porous and shrunken cell walls, skeleton of altered lignified wall material.
Soft rot	Brown. Often localised to wood surfaces. Cracks and checks when dry.	Loss in strength in late stages of decay.	Cellulose degraded.	Cavities present in secondary walls, or secondary walls eroded, leaving the middle lamellae.
White rot	Bleached appearance. Retains shape and composition until decay is advanced.	Major strength losses in intermediate to late stages of decay.	Lignin, cellulose and hemicelluloses degraded.	All secondary cell-wall layers and middle lamellae are eroded.
Fungal stain	Various discolouration in sapwood	No strength loss.	Free sugars, nutrients and wood extractives utilised, increase in melamin-like compounds and pigmented substances.	Preferential colonisation of ray parenchyma cells. No cell-wall degradation.
Surface moulds	Discolouration of wood surfaces only.	No strength losses.	Readily assimilated substances are removed.	Preferential colonisation of parenchyma cells. No cell-wall degradation.

Chemical and biochemical reasons for degradation

The general concepts of the chemical and biochemical reasons for degradation were covered in a comprehensive paper (Jeffries, 1987) considering wood as a whole as well as the constituent components.

White rot fungi use all the chemical components of wood cell walls. Bari et al. (2015) considered two of the more common fungi having differing modes of attack. *Pleurotus ostreatus* gains its nourishment from the breakdown and absorption of nourishment from dead and fallen trees (saphrophytic means) as well as acting as a wounding parasite in living trees. On the other hand, *Trametes versicolor* causes extensive saprophytic degradation in dead and decaying wood. In a study by Riley et al. (2014), the range of secondary enzymes resulting from basidiomycetes attack were analysed, with particular emphasis on fatty acid synthases, non-ribosomal peptide synthases, polyketide synthases and terpene synthases. It was shown that the enzymes acting during white rot were more varied than those depicted for brown rot, though glucose–methanol–choline (GMC) oxidoreductase, a group comprising enzymes such as aryl-alcohol oxidoreductases, alcohol oxidases, cellobiose dehydrogenases, glucose oxidases, glucose dehydrogenases, pyranose dehydrogenases and pyranose oxidases (Sützl et al., 2019) was responsible for the highest levels of lignin degradation for both white and brown rot fungi. It is the activity of enzymes such as class II peroxidase, copper radical oxidase and laccase that provide the increased levels of degradation in lignin compared to the brown rot fungi.

The fungal biodegradation of wood may be utilised for industrial purposes and it has a great potential in the cellulose-producing and wood-processing industries. Fermentation with a selective white rot fungi can provide a route for the production of biofuel or cellulose-enriched forage for ruminants, and can serve as a delignifying pre-treatment to expose the polysaccharides to a subsequent hydrolytic digestion and increase the efficiency of biogas or bioethanol fermentation with bacteria or yeasts (Itoh et al., 2003; Amirta et al., 2005). Besides the fermentation of woody substrates using fungi which degrade or modify the different wood components, isolated oxidative enzymes from the same origin may be valuable tools for more specific and targeted chemical reactions in fibre bleaching and fibre modification and they have been tested for possible applications in the pulp and paper industries (Grönqvist et al., 2003; Maijala et al., 2008). It has long been noted (Leatham et al., 1990) that the use of enzymatic digestion can help for save energy in the pulping sector.

Brown rot fungi, on the other hand, have a unique ability to attack the cellulose fraction of wood while avoiding the surrounding lignin. It has been suggested that the fungi accomplish this by using a two-step process, first secreting chemicals and enzymes that open up the lignin framework and then releasing a second set of enzymes that break down the cellulose chains into sugars. The sugars are absorbed by the fungi to use as biofuel.

To accomplish this task, brown rot fungi generate highly reactive oxygen species that alter the chemical structure of the wood, working with enzymes that break down the cellulosic chains. However, reactive oxygen species might damage the fungal enzymes as easily as the wood structure, and it has long been hypothesised that the fungi spatially segregate the oxidant generation process from the secreted enzymes using chemical barriers. This is achieved through the enzymes incorporating the Fenton reaction, and generating highly reactive hydroxyl radicals:

$$H_2O_2 + Fe^{2+} \rightarrow OH^- + Fe^{3+} + \bullet OH \qquad (1.5)$$

Since the specificity of cellulosic attack negates the decay of lignin, brown rot decay typically occurs more rapid than white rot decay.

Decay mechanisms

Depending on the type of fungal species and the group to which it belongs, different mechanisms for the decay of wood are involved, although most of them involve enzymatic attack. Mahajan et al. (2012) reported that certain white rot fungi, such as *Trametes versicolor* and *Phanerochaete chrysosporium*, are capable of simultaneously degrading lignin, cellulose, and hemicelluloses. On the other hand, other white rot fungi, such as *Ceriporiopsis subvermispora*, appear to selectively degrade lignin and then only slowly to hydrolyse cellulose. The ability of fungi to selectively degrade lignin has been used to pre-treat and defibrillate wood in the production of mechanical pulp up to commercial production levels (Scott et al., 2002; Fackler et al., 2006; Mendonça et al., 2008).

The enzymes responsible for lignin decay have been reviewed by Abdel-Hamid et al. (2013), including the so-called class II secreted fungal peroxidases, all of which are extracellular heme peroxidases: lignin peroxidase (LiP), manganese-dependent peroxidase (MnP) and versatile peroxidase (VP). The mechanism by which they are understood to work is shown in Figure 1.66.

In order to describe the modes of action of these class II secreted fungal peroxidases, Abdel-Hamid et al. (2013) also described prokaryotic peroxidases (class I) and classical secreted plant peroxidases (class III), dye de-colourising peroxidase (DyP), copper-containing phenol oxidases (Laccase), and several oxidoreductases, which include glyoxal oxidase, aryl alcohol oxidase (veratryl alcohol oxidase), pyranose 2-oxidase (glucose 1-oxidase), cellobiose/quinone oxidoreductase and cellobiose dehydrogenase.

The catalytic cycle of MnP (shown diagrammatically in Figure 1.67) is similar to those of other heme-containing peroxidases. Activated through the initiation with hydrogen peroxide of a conventional ferric enzyme, it is unique in utilising Mn^{2+} as the electron donor to form Mn^{3+}. Abdel-Hamid et al. (2013) also suggest that MnP is capable of cleaving non-phenolic lignin substrates.

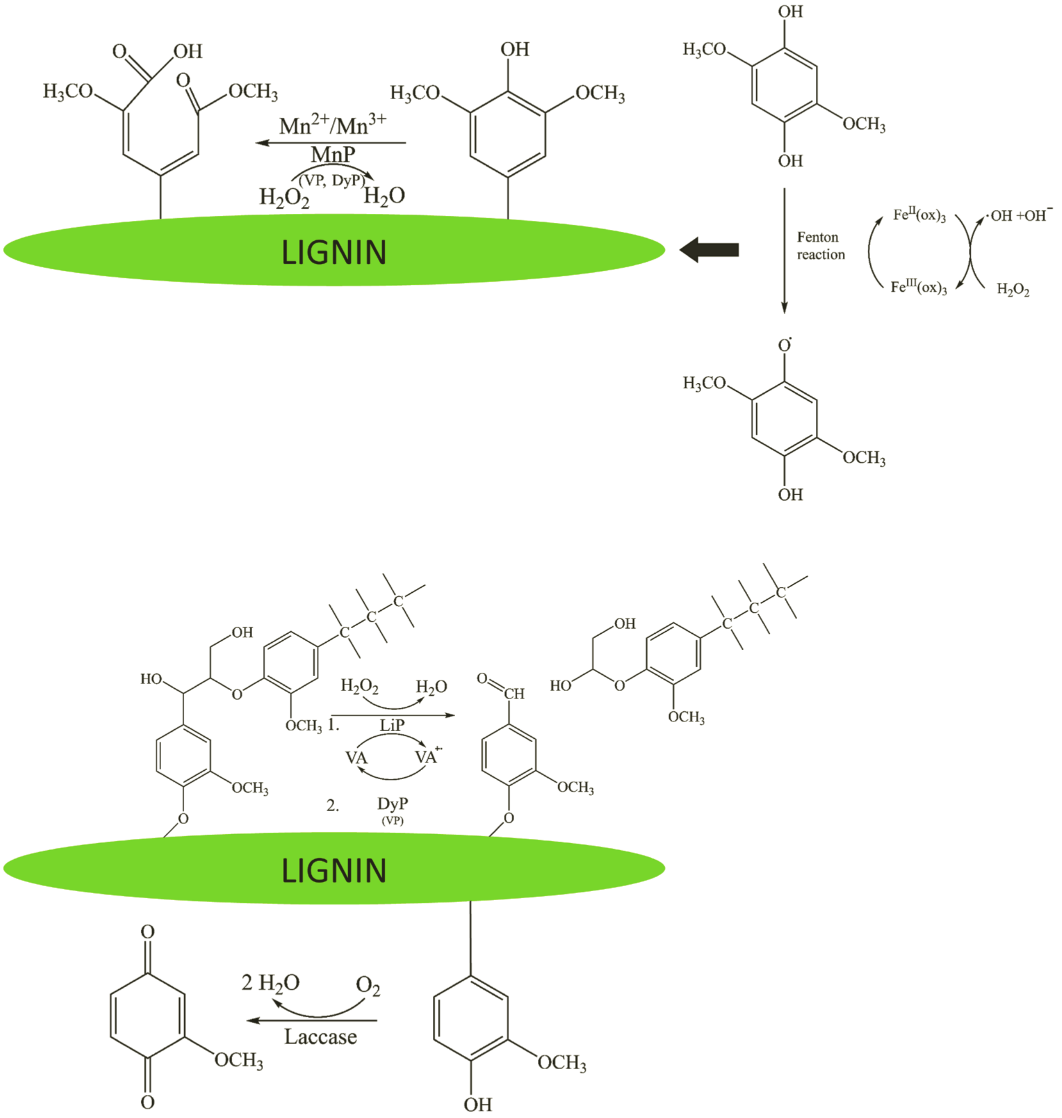


Figure 1.66 The reaction catalysed by lignin-degrading enzymes. LiP: lignin peroxidase, MnP: manganese peroxidase, VP: versatile peroxidase, AAO: aryl alcohol oxidase, GLOX: glyoxal oxidase (modified after Janusz et al., 2017).

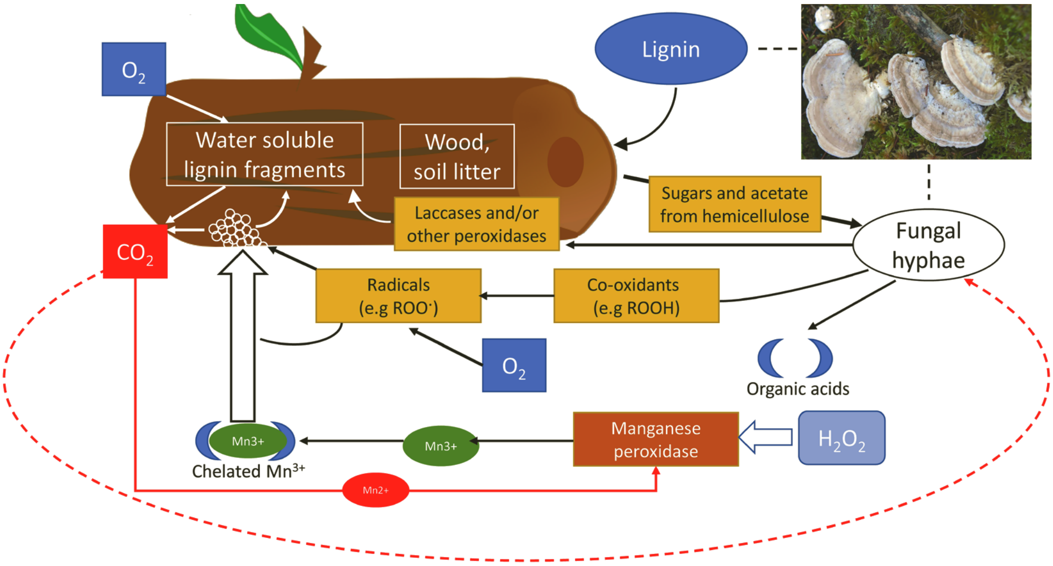


Figure 1.67 Diagram of lignin degradation by white rot fungi (after Zhou et al., 2013).

The white rot fungi also contain laccase, which is widely distributed in wood-degrading fungi, such as *Trametes versicolor*, *Trametes hirsuta*, *Trametes ochracea*, *Trametes villosa*, *Trametes gallica*, *Cerrena maxima*, *Phlebia radiata*, *Ceriporiopsis subvermispora*, and *Pleurotus eryngii* (Baldrian, 2006), although more than one iso-enzyme has been identified in many white rot fungal species. Typically, the laccases are monomeric proteins, although their structures often consist of two identical subunits. Baldrian (2006) presented a comprehensive list of substrates and inhibitors for fungal laccases.

It is known that brown rot fungi specifically attack the cellulose, leaving the lignin relatively intact, but it is not fully understood, since this would seem to restrict access to the polysaccharide food sources. This is thought to have developed during the evolution of brown rot fungi from an ancestral form of white rot fungi, a range of cellulose- and lignin-modifying enzymes being eliminated during its evolution.

Brown rot attack is initiated when germinated spores result in hyphae growth through the cell lumens to colonise ray cells and axial parenchyma. These are readily accessible sources of carbohydrate, providing the necessary nourishment for the hyphae to continue growing through pit membranes and accessing tracheid lumens. Growth is further aided by a glucan coating secreted during the growth process, which allows the hyphae to bind to the wood cell wall, and specifically to the S_3 layer (Illman and Highley, 1989). By linking onto the S_3 layer, the fungus can directly attack and severely degrade the S_2 layer, which has a lower lignin level than both the S_1 and S_3 layers respectively. This fairly rapid degradation of the cellulosic components can lead to a catastrophic strength loss, as much as 70% of the modulus of elasticity (MOE) and modulus of rupture (MOR) (Wilcox et al., 1974).

The mechanism for brown rot decay is understood to be based on the Fenton reaction, and the basic principles have been well reviewed by Arantes and Goodell (2014). Modified wood is still recognised as a possible nutrient source for fungal degradation, since fungi induce genes that are involved in cellulose degradation to even higher levels than in untreated wood (Alfredsen and Fossdal, 2010; Pilgård et al., 2012; Ringman et al., 2014). In some wood modifications, micro-pores are partly blocked, but it has been calculated that the low molecular weight molecules needed for oxidative degradation should be able to penetrate the modified wood (Hill et al., 2005). The next step is the diffusion of these low molecular weight molecules into the wood cell wall, though it has yet to be determined whether this occurs in modified wood. It is possible that the moisture levels inside the wood cell wall are too low to allow diffusion.

Recent work (Ringman et al., 2019) has emphasised the importance of moisture in the brown rot attacks on modified wood, with an emphasis on the *Fenton reaction* allowing hydrogen peroxide produced by the fungus to initiate the formation of hydroxyl radicals capable of degrading the hemicelluloses, although the mechanism of attack has still to be identified. The modes of action suggested to date (Zelinka et al., 2016) include (i) inhibition of diffusion through an increase in the glass-transition temperature of hemicelluloses (assumed to be the medium of transport); (ii) inhibition of diffusion by nano-pore blocking; (iii) no inhibition of diffusion but instead a lower rate of diffusion and/or inhibition of chemical reactions leading up to the Fenton reaction through alteration of the pH level, for example.

Moisture has been recognised as a key parameter in the infestation and decay of wood by wood destroying fungi. In addition to the supply of oxygen, a favourable temperature, and accessible nutrients, it is an essential factor in the fungal decay of wooden commodities and structures. For many decades, it was therefore essential to define the critical moisture content thresholds allowing the transport and activity of fungal enzymes in the wood cell walls leading to the degradation and severe rot of wooden elements. Nowadays, the wood moisture content is the most important input variable in many service life and performance prediction models, both in engineering and natural sciences (Brischke and Thelandersson, 2014).

Degradation models and service life

The group of wood-degrading organisms includes termites, wood-boring beetles, marine borers and various wood-destroying fungi and bacteria, all of which need to be considered when defining the natural durability of timber. In principal, this natural durability can be determined either in the field or in the laboratory by various standardised and non-standardised methods (Råberg et al., 2005; Brischke et al., 2011b; Curling, 2017). While laboratory tests require clearly defined conditions which give a high level of reproducibility, it is usually impossible to fully mimic real life conditions. On the one hand, there is a risk of creating a test scenario that is too severe in terms of moisture and temperature, which are ideal for the degrading organism and have been criticised as 'torture testing' (e.g., Brischke et al., 2011b). On the other hand, some parameters having an important impact on the degradation process cannot be considered adequately, for instance, the detoxification through so-called 'non-target organisms' or the limited number of test organisms considered in European test standards, which are not necessarily responsible for decay under real life situations.

It is generally accepted that field tests provide more realistic test conditions, but they often suffer from unacceptably long test durations. In-ground tests with buried stakes need at least five years to give an indication of the effectiveness of a wood preservative (Larsson-Brelid et al., 2011; Hansson et al., 2013), but the onset of decay in above-ground trials takes place significantly later, and service lives cannot be calculated before decades have passed (Wang et al., 2008; Brischke et al., 2012). For these reasons, the results of laboratory decay tests as well as field test data from in-ground graveyard tests are often presented, and natural durability studies with respect to above-ground exposure are rare, although they play a more important role in timber engineering. When considering moisture risk, the effects of mould and surface disfiguration must also be taken into account.

Three recent European projects—*PerformWOOD*, *Wood Build* and *Timber Bridges*—have attempted to determine issues related to moisture risk and the service of timber products. These projects have looked at a wide variety of timbers (both untreated and treated), but only in recent years has modified wood undergone a critical evaluation.

PerformWOOD: The objective of the project was to kick-start the development of new standards to make possible the service life specification of wood and wood-based materials for construction. This is critical to ensure the future sustainable use of European forests, to ensure that customers of wood products receive satisfactory and reliable products and to provide supplementary evidence of life cycle evaluations of construction products (e.g., Environmental Product Declarations).

Figure 1.68 Sneek bridge in The Netherlands, constructed using Accoya^{TM}.

Wood Build: The overall objective of the project was to raise awareness and to disseminate knowledge and expertise on damp proofing and, from the viewpoint of resistance, durable wood materials for the construction industry, in order to strengthen the competitiveness of timber as a building material. This will be achieved through the production of new knowledge that enhances our understanding of the link between climate exposure and the resistance of wood to biological attack.

Timber Bridges: The project considered the increased use of wood for bridge construction, emphasised by the building of some 650 timber bridges in Sweden alone in the past 15 years. Naturally, a strong emphasis must be placed on the durability and performance of the materials used, adopting best practice for the prevention of moisture uptake and the risk of decay. The construction of the Sneek bridge in The Netherlands using Accoya^{TM} acetylated wood (Figure 1.68) has demonstrated how modified wood may help in overcoming some of the issues linked to premature failure.

Fungal testing of modified wood

The complexity of testing the durability of wood results from the different areas and exposure situations in which wood is used. Wood is used for constructions in the building sector ranging from simple constructions like range-land fences to more complex balconies and recently also roller coasters and multi-storey buildings. These different constructions and their specific components are accompanied by a wide range of different loads, where the decisive loads responsible for the risk of damage can be reduced to moisture, temperature and the presence of wood-destroying organisms. The first step in classifying a wooden component with respect to an expected load is to distinguish between in-ground and above-ground exposure. Numerous tests have been conducted all over the world and have been described in the literature referring to both of these exposure conditions (e.g., Fougerousse, 1976; De Groot, 1992; Fredriksson, 2010; Brischke et al., 2012). The ones referring to above-ground exposure have been less frequently used to determine durability and only a few have been standardised. The reason for this can be found in the long exposure periods for above-ground tests compared to in-ground tests. Testing wood durability exclusively in ground is however in contrast to the fact that most timber products in outdoor use are exposed above ground, e.g., façades, terrace decking, windows, balconies and carports (Blom and Bergström, 2005; Friese et al., 2009). A range of different standardised test methods have been drawn up and applied over the years (Table 1.12) but they have dealt exclusively with naturally occurring wood or material that has been subjected to a traditional wood preservation procedure.

To overcome this drawback a comparative study on the moisture performance and resulting decay response has been conducted (Meyer et al., 2013). Five different wood species were used in 27 different test set-ups representing a wide range of different exposure situations. The test set-up included established and standardised test methods (e.g., L-Joint test, decking test, ground proximity test) as well as some new test methods. Figure 1.69 shows schematic drawings of various tests conducted to date. A more comprehensive overview including the dimensions and details of the exposure conditions and specimen compositions of all the test methods is given by Meyer et al. (2013). This work expanded the methodologies and data available for exposed wood, which

Table 1.12 Standardised tests involving fungal decay of wood (modified from Brischke et al., 2013).

Standard	Title of standard
CEN/TS 15083-1	Durability of wood and wood-based products. Determination of the natural durability of solid wood against wood-destroying fungi, test methods. Basidiomycetes
CENT/TS 15083-2	Durability of wood and wood-based products. Determination of the natural durability of solid wood against wood-destroying fungi, test methods. Soft rotting micro-fungi
EN 113	Wood preservatives - Method of test for determining the protective effectiveness against wood destroying basidiomycetes - Determination of the toxic values
ENV 807	Wood preservatives - Determination of the effectiveness against soft rotting micro-fungi and other soil-inhabiting micro-organisms
TS 12404	Durability of wood and wood-based products - Assessment of the effectiveness of a masonry fungicide to prevent growth into wood of dry rot *Serpula lacrymans* (Schumacher ex Fries) F.S. Gray
EN 15457	Paints and varnishes - Laboratory method for testing the efficacy of film preservatives in a coating against fungi
EN 152	Wood preservatives - Determination of the protective effectiveness of a preservative treatment against blue stain in wood in service
CEN/TS 839	Wood preservatives - Determination of the protective effectiveness against wood destroying basidiomycetes - Application by surface treatment
AWPA E 10	Standard method of testing wood preservatives by laboratory soil-block cultures
ENV 12038	Durability of wood and wood-based products - Wood-based panels - Method of test for determining the resistance against wood-destroying basidiomycetes
JIS K 1571	Wood preservatives - Performance requirements and test methods for determining their effectiveness

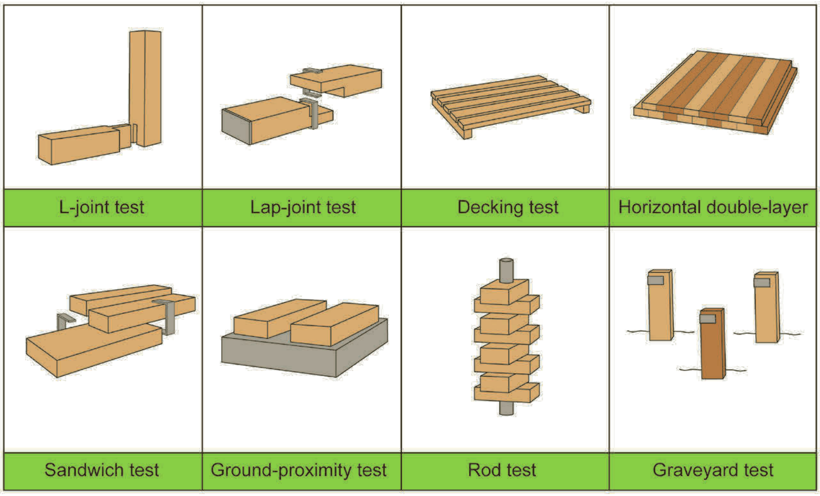


Figure 1.69 Examples of outdoor exposure test rigs (Meyer et al., 2013).

are being further applied to modified wood are developing the understanding of decay in both laboratory and field trials, as previously reported by Alfredsen and Westin (2009).

Minimum moisture thresholds and other physiological requirements of decay fungi were sought in field tests (Scheffer, 1971; Van den Bulcke et al., 2009; Meyer-Veltrup et al., 2017) as well as in experiments under laboratory conditions (Viitanen, 1997; Viitanen et al., 2010). The general consensus is that water availability in the cell walls is critical, but not necessarily in the cell lumens (Schmidt, 2006; Stienen et al., 2014).

Understanding the decay mechanisms due to fungal attack is essential in helping to predict building performance and the durability of wood components. The use of modified wood has the potential to provide additional durability. While the use of existing methods and standards provides a comparative performance factor against reference material, the modes of attack used in a particular standard may differ for modified wood compared to that suitable for untreated or preservative-treated materials. It has been suggested by several research groups at previous European Conferences on Wood Modification that modified wood should be considered as a new wood species. Since there may be issues to the means by which material-moisture interactions occur, and the fact that degradative pathways are blocked due to the inability of a decay organism to recognise the matrix means there is the need to undertake a systematic programme of research at laboratory and field study levels to determine how to correctly access the service life of modified wood products.

Biological degradation of wood by invertebrate organisms

Lignocellulosic materials, such as wood, are often used by insects for food, shelter and breeding, the most common lignocellulose-destroying insects belonging to Coleoptera (beetles) and Blattodea: Isoptera (termites) orders. They are referred to as wood-eating (xylophagous) insects, which means that they cannot live without wood or other lignocellulosic materials. Attack is not limited to above or in- or above-ground cases. Marine decay also occurs through the attack of worms or gribble.

Wood-boring insects

All insects have a similar structure. They have three pair of legs (gr. *Hexapoda*) and segmented bodies supported by exoskeletons, the hard outer covering being mostly chitin. The segments of the body are organised into three distinctive but interconnected units or tagmata: a head (the caput), a chest (the thorax) and the posterior (the abdomen). The insects pass through a metamorphic transformation during their life span. Beetles undergo four developmental stages (holometabolism), egg, larvae, pupa and adult (imago). Eggs are laid within the wooden structure, and they hatch and the subsequent larval and pupal stages are spent within the wood structure, from which nourishment is obtained. Most beetles are defined by having part of the hardened exterior forming the front wings (elytra), defining them within the family Coleoptera

The powderpost beetles are members of the *Lyctinae* subfamily within the *Bostrichidae* family, of which over 20 species have been identified, including *Lyctus brunneus* (brown powderpost beetle). Whilst it now has a global distribution, it was probably originally native to Central and South America. The common furniture beetle (woodworm, *Anobium punctatum*) spread from Europe to areas with similar climatic conditions, mainly as a result of colonisation over the past 300 years. In the larval stage, it bores into seasoned sapwood between 12−16% moisture content and feeds upon it. The adults are between 3.0 and 4.5 mm in length and have brown ellipsoidal bodies with a prothorax resembling a monk’s cowl. Because of the risk to sapwood, some building regulations state that timber with more than 25% sapwood may not be used, in order to limit the risk of loss of structural integrity through wood-borer infection. The death watch beetle, *Xestobium rufovillosum*, is a species of wood-boring beetle that sometimes infests the structural timbers of old buildings, although its natural occurrence is in broad-leaved trees that have been dead for around 60 years. It has been suggested that this is due to fungal decay with an associated softening of the wood constituents and increased digestibility. Infestation in European oak has been associated with its colonisation by the fungus *Donkioporia expansa*. The death watch beetle originated in regions in Europe, but is now also present in USA. The house longhorn beetle (*Hylotrupes bajulus*) originated in Europe, and has been spread in timber and wood products as colonisation and emigration have progressed, so that it now has a practically world-wide distribution, including southern Africa, Asia, the Americas, Australia, and much of Europe and the Mediterranean. *Hylotrupes bajulus* preferentially attacks freshly produced sapwood of timber, though there have been cases of attack of the sapwood of certain broad-leaved species such as oak. In conifers such as Norway spruce, it also attacks the heartwood. An excellent summary of the attack by various wood-boring insects

Table 1.13 Some common wood-boring insects (modified from Blanchette, 1998).

Insect	Wood	Distinguishing characteristics
Common furniture beetle, *Anobium*	Sapwood of conifers and broad-leaved woods. May attack heartwood if fungal decay is present	Meandering tunnels 1−2 mm in diameter, often in direction of grain, filled with frass consisting of oval pellets and wood powder.
Lyctus, powderpost beetle	Sapwood of broad-leaved woods with large vessels, such as oak and elm	Damage in sapwood with high starch content. Circular tunnels 1–2 mm in diameter, usually parallel to the grain, filled with fine powder.
Bostrychid powderpost beetle	Sapwood of tropical timbers	Convoluted tunnels 3–6 mm in diameter, packed with fine powder.
Wood-boring weevil	Decayed conifer and broad-leaved woods	Tunnels 1 mm in diameter, orientated in the direction of the grain, with fine, granular powder.
Ptilinus beetle	Sapwood of broad-leaved woods	Meandering tunnels 1–2 mm in diameter, packed with fine bore dust.
Death watch beetle	Sapwood and heartwood of decayed broad-leaved woods	Tunnels variable in diameter from 0.5–3 mm, randomly orientated but common in the direction of the grain. Bore dust consists of fine, disc-shaped pellets.
House longhorn beetle, cerambycid beetle	Sapwood of conifers	Tunnels 6–10 mm in diameter with similar-sized oval emergence holes. Bore dust contains cylindrical pellets with fragments of wood. Most of the sapwood may be consumed, with just a veneer of surface wood left.

Figure 1.70 Common adult wood-boring beetles that can damage wood, from the left: True powder-post beetle (*Lyctus brunneus*), Woodworm or common furniture beetle (*Anobium punctatum*), Death watch beetle (*Xestobium rufovillosum*), and House longhorn beetle (*Hylotrupes bajulus*) (Wikipedia Commons, and BioLib.cz.).

was provided by Blanchette (1998), and it is shown in Table 1.13, with some examples of beetles in Figure 1.70.

Termites

Termites (Blattodea; formerly Isoptera) are consumers of cellulose and lignocellulose found in dead wood, grass, microepiphytes, leaf litter, and sometimes cultivated fungi. Some 3,000 species of termites have been described, most having a tropical and temperate distribution across the USA, central America, most of South America, southern Europe, Africa, Middles East, Southern Asia, Japan and Oceania. Figure 1.71 shows the global distribution of the three species most responsible for structural damage, namely *Coptotermes formosanus*, *Coptotermes gestroi* and *Cryptotermes brevis* (Rust and Su, 2012). Of these 3,000 species, only 83 are considered to present a risk to wooden structures and furniture (Rust and Su, 2012).

As can be seen in Figure 1.71, Europe lies on the border of traditional termite presence, but there are concerns as to how global warming may affect their distribution and spread then into more northern areas. A more detailed European distribution is shown in Figure 1.72 (Kutnik et al., 2020),

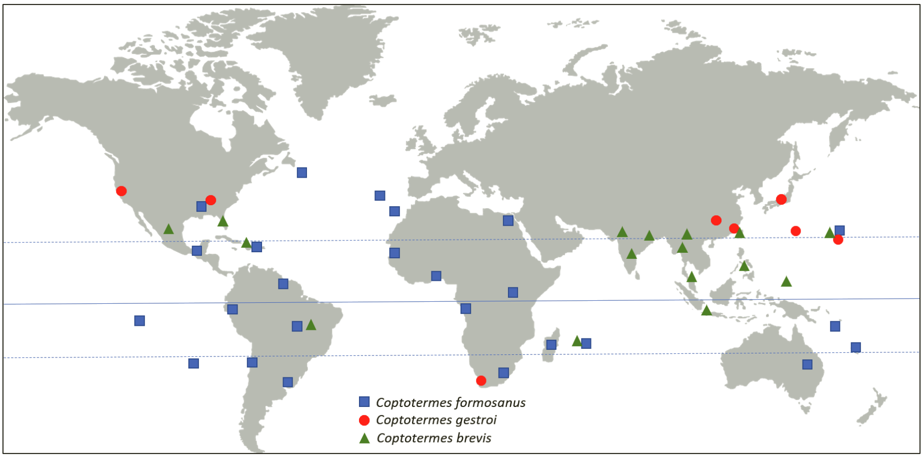


Figure 1.71 Distributions of the three most economically important and widely distributed termite pest species: *Coptotermes formosanus*, *Coptotermes gestroi*, and *Cryptotermes brevis* (Rust and Su, 2012) (Global map downloaded from www.presentationgo.com).

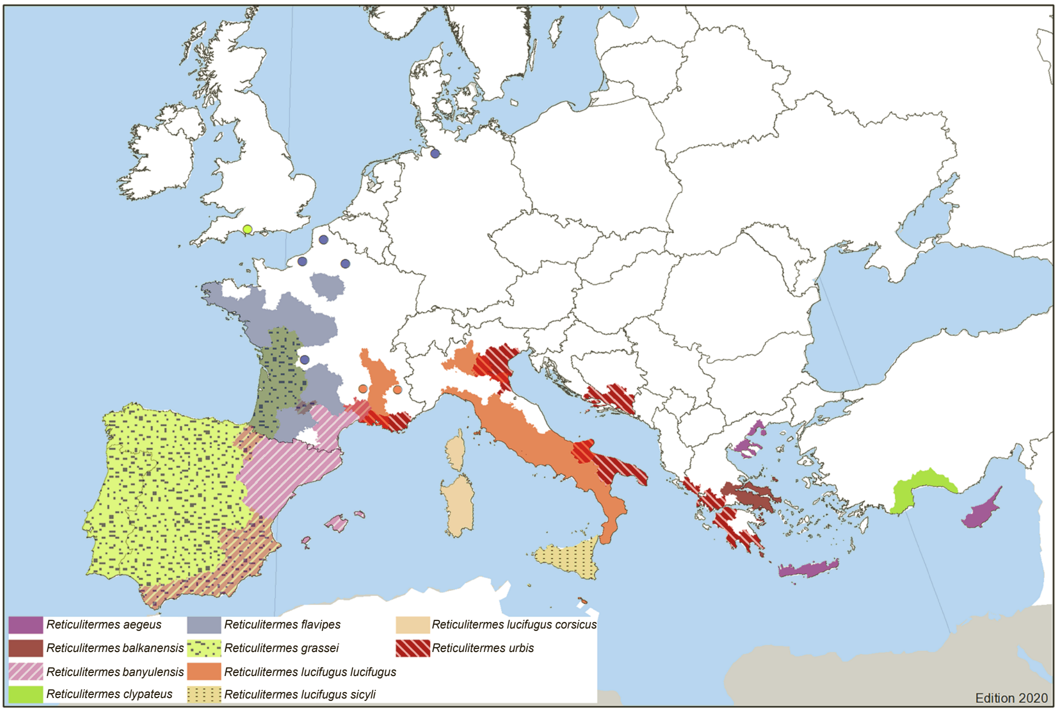


Figure 1.72 European distribution of termite species (courtesy of Magdalena Kutnik).

which shows the traditional range of a variety of *Reticulitermes* species. Additional sites outside these ranges have however occurred as a result of human activity and the importation of infected timber. This was the case with the infestation of *Reticulitermes flavipes* in Hamburg, Germany, a common termite in the eastern part of the USA, which established a colony in the city in the 1930s, taking advantage of the warm, moist conditions in the district heating system. Another species, *Reticulitermes grassei*, normally found in south-western France, Spain and Portugal, was identified in infested timber in Devon, England in the 1960s, but it has been suggested that this was a case of natural colonisation from France.

Traditionally, termites are classified into lower and higher termites, where the higher termites have undergone more evolutionary changes and live in more advanced social structures and colonies. Lower termites include eight families (*Mastotermitidae, Archotermopsidae, Hodotermitidae, Stolotermitidae, Kalotermitidae, Stylotermitidae, Rhinotermitidae,* and *Serritermitidae*), whereas higher termites belong to a single family, *Termitidae* (Engel, 2011). A detailed description of the types of termites and their economic and environmental impacts is available (e.g., Govorushko, 2019).

Termites are highly effective at processing biomass and are estimated to process up to 35% of dead wood in the tropics (Verma et al., 2009), and another study of tropical and subtropical areas has suggested that termites account for 10% of animal biomass and 95% of soil insect biomass (Donovan et al., 2007). Their activities create favourable conditions for primary producers, including maintaining soil pH, increasing water retention, mediating decomposition and nutrient cycling, and creating surface areas suitable for microbial colonisation. However, the desire to source suitable feedstocks has led to problems where timber has been used in construction and our built environment. Some species of termites feed on plant material and timber used by humans, necessitating expensive repairs, prevention and control efforts. Termites may also damage materials adjoining or close to timber that is being attacked for food, and this can create problems with items such as electrical and telephone wiring, cables, dams and farming equipment. One study (Jones et al., 2015) has suggested that a colony of 200,000 termites can consume up to 5.9 kg of cellulose per year. It is estimated that worldwide the costs for termite control and repair are round $30 billion (Rust and Su, 2012), most of these costs (80%) being associated with subterranean termites.

Marine wood borers

Marine wood borers have long been recognised for the damage the caus to wooden boats and infrastructures in maritime conditions. The marine wood borers are known to include *Bivalvia* (*Teredinidae* and *Xylophagaidae*), *Isopoda* (*Limnoriidae* and *Sphaeromatidae*), and *Amphipoda* (*Cheluridae*) Treu et al. (2019). Some examples of these are shown in Figure 1.73. In Europe most wood boring bivalves belong to the *Teredinidae* class, but species of the *Xylophagaidae*, such as *Xylophaga dorsalis*, have also been reported near the sea-bed in Europe (Santhakumaran and Sneli, 1978). Attack by *teredinids* is difficult to detect with the naked eye, but a magnifying glass can easily reveal entrance holes of larvae. The degree of attack is usually analysed by X-ray computer tomography, when a non-destructive evaluation is required (Charles et al., 2018), but it can also be investigated by density measurements or strength determination as well as by wood sample preparation and borer species identification (Turner, 1966).

The European wood boring *Crustacea* belong to the *Limnoriidae* and *Cheluridae* families. Their attack pattern is shaped by their tunnelling activities on the wood surface and it is usually easier to detect with the naked eye than shipworm attack. In combination with wave action in the tidal zone, wooden piles develop an hour-glass shape. More recent reviews of the potential attack by wood borers in a marine environment have been given for *Limnoriidae* by Cookson (1990) and Cragg (2003) and for *Teredinidae* by Distel (2003) and Voight (2015), where the authors provide the current state of knowledge of the biogeography, competition and predation among wood borers and the role of bacterial endosymbionts.

Figure 1.73 Examples of marine wood borers, from the left: *Limnoria quadripunctata* (courtesy of Graham Malyon), *Neoteredo reynei* (courtesy of Reuben Shipway), and *Sphaeroma terebans* (All images printed with permission of University of Portsmouth, U.K.).

Mechanism of decay due to insects

Many wood-destroying insects use wood not only as a food source but also as a home, in some cases for the adult insects but more commonly for juvenile (larval) growth. Very few of these insects can digest wood with their own enzymes and even then they are not very effective in their action. For this reason, it seems that most wood-boring insects have evolved a complex symbiosis with micro-organisms specialised in the degradation of wood components (Battisti, 2001).

In the case of *Hylotrupes bajulus*, early studies suggested that the secretion of cellulase allowed the enzymatic digestion of about 20% of the cellulose and hemicelluloses of the wood that was attacked the remaining indigestible 80%, including all the lignin components, being expelled with the faeces (Falck, 1930). Further studies at that time suggested that there were no micro-organisms in the gut and, therefore, that the cellulose is endogenous (Mansour and Mansour-Bek, 1934; Müller, 1934). With more sensitive analytical methods, however, the presence of glucosidase and carboxy methylcellulases (CMC-ase) activity throughout the whole *Hylotrupes* gut was demonstrated (but more pronounced in the foregut), with only a small number of bacteria being present in the midgut, suggesting the endogenous nature of the cellulolytic enzymes (Cazemier et al., 1997). The presence of beta-glucans in the faecal matter was interpreted as proof that starches did not form part of the nutritional needs of the insects (Höll et al., 2002). Since the lignin components remain intact during the colonisation and growth of *Hylotrupes baljulus* larvae, it has been suggested that the composition of the lignin and the presence of terpenoid extractives as a result of tree maturity increase the resistance to insect attack (Venäläinen et al., 2003). In order to gain access to the genetics and physiology of uncultured micro-organisms, the use of metagenomics, which enables the isolation of bacterial genomic DNA from an environment followed by its direct analysis, has emerged as a powerful identification technique (Handelsman, 2004).

In the case of *Anobium punctatum*, it was found (Baker, 1969) that 26–29% of the wood consumed by the larvae was digested. This is slightly higher than for *Hylotrupes bajulus*, but the more significant fact was that the levels of nitrogen was more than double the level available in the digested timber. The mechanism for this is still not fully understood, but it has been suggested that the mechanism involves atmospheric nitrogen, although more recently the presence of gut symbionts has been suggested. The level of decay of the timber also plays a significant role in this mechanism, particularly for *Anobium punctatum*, where the nitrogen within the fungal decay enzymes provides the required level. The role of microbial symbionts within insects has been evaluated in terms of biological nitrogen fixation within terrestrial ecosystems (Nardi et al., 2002). A review of a range of symbiotic mechanisms for different woodboring insects has been published (Chiappini and Aldini, 2011), covering ectosymbiosis, extracellular endosymbiosis and endocytobiosis.

As has been suggested for *Anobium punctatum*, fungal decay can contribute significantly towards the ease of digestibility for insects. Historically, this could pose a problem for timber used in high moisture conditions, as demonstrated in Table 1.14, where temperature and moisture thresholds have been identified (Brischke and Unger, 2017). However, the increase in central heating and more controlled moisture levels in buildings have led to a reduction in the risk of insects such as *Anobium punctatum*.

Many termites feed exclusively on wood, despite its low nutritional level and poor nitrogen content. This can be overcome through a symbiotic relationship with gut protists or flagellates, providing the bacterial capability to break down the wood components, and thus helping the overall

Table 1.14 Typical survival conditions for some wood-boring insects (Brischke and Unger, 2017).

Species	**Moisture content (%)**			**Temperature (°C)**			
	Min.	**Optimum**	**Max.**	**Min.**	**Optimum**	**Max.**	**Lethal**
Anobium punctatum	10–12	28–30	50–57	12	21–24	29	50–57
Hylotrupes bajulus	9–10	30–40	65–80	16–19	28–30	35	55–57
Lyctus brunneus	7–8	14–16	23	18	26–27	30	49–65

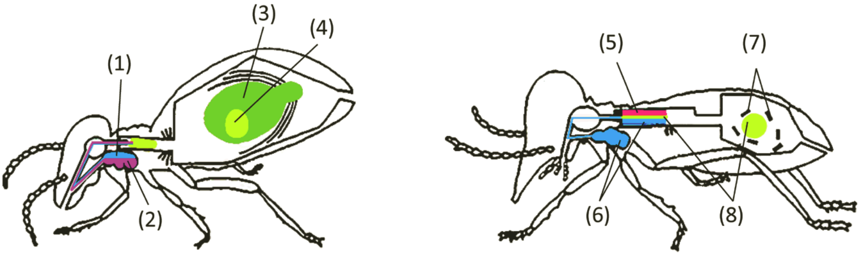


Figure 1.74 Comparison of degradative pathways of lower termites (left) and higher termites (right). Modified after Tokuda (2019). Lower termites (flagellate-harbouring): (1) Endo-β-1,4-glucanases (EGs) that primarily hydrolyse amorphous cellulose. (2) β-glucosidases (BGs) that hydrolyse cellobiose into glucose. (3) Protistan cellulases, hemicellulasesand cellobiohydrolases that actively hydrolyse crystalline cellulose, xylanases, and mannanases that participate in hemicellulose degradation. (4) Cellulose and hemicellulose digestion (in the midgut lumen and protistan cells). Higher termites (flagellate-free): (5) Endo-β-1,4-glucanases (EGs) that primarily hydrolyse amorphous cellulose are secreted in the midgut. (6) β-glucosidases (BGs) that hydrolyse cellobiose into glucose are secreted both in the salivary glands and the midgut. (7) Hindgut bacteria producing cellulases and hemicellulases; xylanases and cellulases are predominantly expressed in the fibre-associated bacterial community. (8) Cellulose and hemicellulose digestion (in the midgut and hindgut lumen).

digestion process, since termites often have their own endemic enzymes, such as endoglucanases and cellobiases. Lower termites harbour flagellated cellulolytic protists in the hindgut, whereas the higher termites have lost the protists but have a greater range of gut microbes. The digestive mechanisms of lignocellulose are, thus, differentiated between lower and higher termites in a way that higher termites can also feed on soil and a wider range of plant materials. The differences in indigestion processes between lower and higher termites are shown in Figure 1.74 (Tokuda, 2019).

These are not however sufficient alone and many termite species depend on the flagellate relationship to survive. If these are present within the gut systems, they must be transferred to the larvae, which is done through the larvae eating faecal matter containing these flagellates. The symbiotic relationship has been reviewed elsewhere, e.g., by Brugerolle and Radek (2006) who indicated that two lineages of protozoa exist in termites: the Oxymonadida and the Parabasalia, with more than 400 species identified in total. A more detailed breakdown of the major flagellate protist groups has been given by Duarte et al. (2017).

The flagellates work by breaking down the cellulose and hemicellulose via enzymatic pathways in order to release glucose and other sugar oligomers. These can then undergo secondary conversions (e.g., to pyruvate) or be used directly as energy and food sources. Since each flagellate has a distinct biochemical process, a thorough understanding of these and how certain enzymatic digestion can be limited or even inhibited can in principle offer routes to new biochemical processing for biorefining or even for developing new termiticides. Tartar et al. (2009) have also suggested that for some termites (e.g., *Reticulitermes flavipes*) there is laccase activity present in the foregut and salivary glands and that this aids the digestion of lignocelluose, as has been demonstrated through phenoloxidase activity.

In the case of marine borers, the levels of attack depend on the species and location. In a study of European marine borer activity (Borges, 2014), it was noted that in northern Europe, *Teredo navalis* was the species that posed the highest borer hazard, whereas *Lyrodus pedicellatus* was the most destructive species in the Atlantic coast of southern Europe, with the exception of two sites in Portugal. In those sites, *Limnoria tripunctata* was more destructive than *L. pedicellatus*. In the Mediterranean, both *T. navalis* and *L. pedicellatus* posed very high borer hazards to wooden structures. It has been well documented that the *Limnoria* genus is one of the few animals that can feed on wood without having any gut enzymes present to allow its digestion. Instead it has been suggested that the digestion process is assisted by the presence of hemocyanin, a copper-containing protein thought to be derived from phenoloxidases, recreating the activity of phenoloxidases via a loosening of the tertiary structure, enabling access to the copper-complexed active site. In addition,

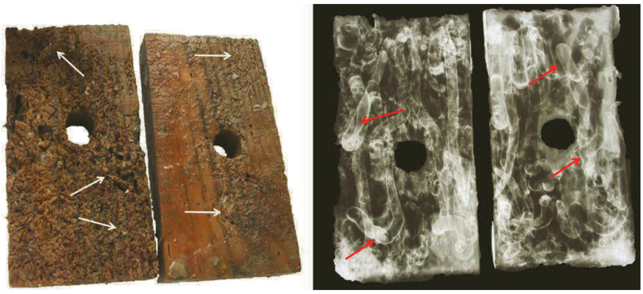


Figure 1.75 Evidence of teredinid attack on wood: the white arrows (left) show the extent of Limnoriid attack, and the red arrows (right) show the presence of shipworms within the wood structure (Borges, 2014) (courtesy of Luisa Borges).

it has been shown (Besser et al., 2018) that glycosyl hydrolases (GHs) are present, and in particular GH7 and GH9, which account for over 50% of the soluble GHs in the gut fluids.

Shipworms, such as *Teredo navalis*, have historically caused major problems for wood immersed in sea-water, as can be seen in Figure 1.75. As with *Limnoriids*, they have been found to be devoid of symbiotic microbres assisting in the enzymatic digestion of wood, but have a variety of carbohydrate active enzymes produced by endosymbiotic bacteria housed in specialised cells (bacteriocytes) in the animal's gills. The range of these glycosyl hydrolases is much wider than those found in *Limnoriids*. Earlier work by Hashimoto and Onoma (1949) showed the presence of cellulase, alginase and xylanase activity in the "liver" of Teredo worms, and Mawatari (1950) also reported the presence of amylase, cellulase, cellobiase, saccharase and maltase in the midgut.

Test methods for the risk of insect attack and the use of modified wood

Until recently, the main method for preventing insect damage to timber in constructions and in the built environment has been through the use of wood preservatives. This is clearly demonstrated by the test methods currently employed, as shown in Table 1.15, a thorough description of these methods being given by Curling (2017).

Table 1.15 Typical test methods used in the analysis of insect attack on wood.

Standard	Title of standard
EN 117	Wood preservatives: Determination of toxic values against *Reticulitermes* species (European termites)
EN 118	Wood preservatives: Determination of preventive action against *Reticulitermes* species (European termites)
ASTM D3345	Standard test method for laboratory evaluation of wood and other cellulosic materials for resistance to termites
AWPA E1	Standard field test for evaluating the termite resistance of wood-based materials: Choice and no-choice tests.
JIS K 1571	Test methods for determining the effectiveness of wood preservatives and their performance requirement
EN 46	Wood preservatives: Determination of the preventive action against recently hatched larvae of *Hylotrupes bajulus* (L.)
EN 47	Wood preservatives: Determination of the toxic values against larvae of *Hylotrupes bajulus* (L.)
EN 370	Wood preservatives: Determination of eradicant efficacy in preventing emergence of *Anobium punctatum* (De Geer)
EN 49	Wood preservatives: Determination of the protective effectiveness against *Anobium punctatum* (De Geer) by egg-laying and larval survival
EN 20	Wood preservatives: Determination of the effectiveness against *Lyctus brunneus* (Stephens)
EN 275	Wood preservatives: Determination of the protective effectiveness of preservatives against marine borers

Although there is a standard test for marine borers (EN 275), the testing of marine borers is generally based on design specifications put forward by whoever is conducting the test. EN 275 is a field trial method limited by the need to have active marine borers present, and this limits the number of sites that can be used for this test. The limnoriids *L. quadripunctata* or *L. tripunctata* and the teredinid *L. pedicellatus* are suitable for laboratory tests focused on wood for use in temperate to warm-temperate marine climate. Meaningful results can be gained within a month of experimentation. Tests using individual *Limnoriids* allow sufficient biological replication for determining optima in treatment variables. It has, however, been found that tests using feeder blocks with a colony of *Limnoriids* do not have a well-defined or easily replicated level of borer activity, and, although it has been suggested that laboratory tests can detect differences between experimental treatments relative to the level of attack on control blocks, such tests cannot be used to predict service life. Hence, for marine borers, it is necessary to undertake marine trials, under the caveat that only treatments causing the greatest mortality or reduction in feeding are valid.

The use of modified wood in limiting insect decay

As the production of modified wood has increased, its range of possible uses has also expanded, and it has become necessary to undertake a range of evaluations to see whether the material is fit for use and can provide the necessary service life. Given that wood modification has the ability to alter the chemical constituents of the wood as well as to reduce the equilibrium moisture content, it is logical to expect that wood modification techniques are capable of reducing insect decay by rendering the material inedible and/or by reducing the level of moisture needed for enzymatic processing. Evaluations are still ongoing, but known results relating to each modification method are given in their respective chapters. Attack by termites is still a matter of study, given that tests are often carried out on only small colonies and these may give incorrect results. It has been suggested (Kutnik et al., 2009) that such methodologies are not always relevant, especially when the tests are performed under laboratory conditions, since the reported mortality rates in isolated groups of termites do not reflect the behaviour of a termite colonies in natural field conditions. Mortality rates in field conditions are always worse than under laboratory conditions. Above ground resistance tests or laboratory tests performed on larger colonies could provide more reliable results. Another concern is that laboratory results based only on insects' mortality cannot provide information about the service life expectations, such as the impact of aesthetic damage, maintenance and expected durability of wooden construction components.

Insect attack, whether on land (above or below soil level) or in water (marine conditions), poses major problems to the use of wood in construction. The test methods currently advocated by testing bodies show that the prevention of attack has to date mainly been limited to the use of preservatives, although naturally durable species have been used in the past. These naturally durable species often come from tropical regions, and this makes their continued use a contentious issue. In addition, the leachability of preservatives into the surroundings has raised concern over the use of many historically effective treatments.

The use of modified wood continues to gain market acceptance in many cases, and there is a growing amount of data supporting its performance against insects. Further improvements in testing methodology may help increase these opportunities, or at least ensure that the correct treatment and usage has been ascribed to a particular wood product.

1.6 Weathering of wood

As a bio-based material, wood will eventually break down into its constituent components. In its natural environment, wood is protected from exterior forces by bark, but when it is exposed outdoors in typical construction uses above ground, it undergoes a series of bio-degradative processes linked to its exposure to chemical, mechanical, and light energy factors, resulting in what is known as weathering. Weathering should not however be confused with decay, which results from decay organisms, such as fungi which are able to attack and ultimately destroy wood in the presence of excess moisture and air over an extended period of time. A thorough overview of weathering of wood has already been published (Williams, 2005).

Factors contributing to weathering

Light – Photochemical degradation due to sunlight occurs fairly rapidly on exposed wood surfaces, with the effect increasing with the intensity and duration of exposure. It has been suggested by Tolvaj et al. (2001) that fluctuations in the ozone layer have increased the effects of UV-B irradiation, and this in turn has influenced the use of mercury lamps for artificial weathering tests. The initial effect of UV exposure is a colour change where wood become more yellow as a result of radical-initiated photodegradation of lignin components (Evans et al., 1992; Müller et al., 2003; Turkoglu et al., 2015). These compounds can be further degraded, resulting in compounds with a carbonyl bond conjugated to double bonds, resulting in an FT-IR spectral peak at 1,615 cm^{-1} (Cogulet et al., 2016). These compounds may be leached with rain/moisture to eventually leave the grey appearance due to the birefringence of cellulose, the optical characteristics of the cellulose having a refractive index which relies on the polarisation and propagation direction of light. Cogulet et al. (2016) used Raman spectroscopy to show that the α and β carbons in lignin are photosensitive, and tend to undergo radical degradation, although there is a noted photoresistance of 5–5′ linkages between diaryl moieties within lignin. These studies also showed that holocellulose undergoes various degrees of photodegradation, although the results were all based on artificial ageing.

The colour variations resulting from photodegradation are caused by surface effects and reactions, and are species dependent. Oberhofnerová et al. (2017) undertook the simultaneous testing of the photodegradaticy of multiple species in an outdoor weathering experiment in Prague, Czech Republic. The results (Table 1.16) show that the lowest degree of discolouration was observed for English oak and the highest for Norway spruce. The depth of the colour changes was relatively similar among the conifers, but greater variations were noted with the broad-leaved woods. The values given in Table 1.16 are calculated as:

$$\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2} \tag{1.6}$$

where ΔL, Δa and Δb are, respectively, the differences in the L*, a* and b* coordinates of the CIELAB colour system.

When wood has a high content of extractives, the chemical processes leading to the colour changes tend to take place fairly quickly, wheres those with a low extractives content tend to be slower but more consistent in overall speed. It has been suggested that the initial increase in b* value indicates the degradation of lignin (Evans et al., 1992; Müller et al., 2003; Turkoglu et al., 2015).

Table 1.16 Colour variations from photodegradation. Mean values and standard deviation (in parentheses) for 24 measurements.

Wood species	1 month		3 months		6 months		12 months	
	ΔE*	gloss	ΔE*	gloss	ΔE*	gloss	ΔE*	gloss
Black alder	6.4 (2.5)	5.9 (1.2)	7.0 (2.5)	5.8 (1.9)	20.9 (2.5)	7.6 (2.2)	27.0 (2.8)	5.9 (2.1)
Black locus	8.8 (0.9)	6.9 (1.1)	12.5 (1.6)	7.0 (1.3)	16.0 (1.5)	9.0 (1.3)	29.6 (2.0)	6.3 (1.2)
Douglas fir	10.5 (0.8)	6.4 (0.8)	10.9 (1.4)	6.3 (1.0)	16.0 (1.3)	8.5 (1.3)	28.1 (1.6)	7.3 (1.2)
English oak	3.2 (0.7)	6.9 (0.7)	4.0 (1.5)	8.0 (1.4)	12.7 (1.5)	11.4 (2.0)	23.0 (1.2)	5.6 (0.8)
European larch	11.3 (2.9)	5.7 (1.2)	12.7 (3.8)	5.5 (1.5)	17.9 (2.7)	8.9 (2.4)	30.5 (2.6)	7.0 (1.6)
Norway spruce	11.4 (0.7)	12.9 (2.1)	13.9 (0.9)	12.7 (2.7)	18.3 (0.8)	16.3 (1.5)	34.1 (0.9)	9.3 (1.7)
Poplar	7.5 (1.3)	10.5 (0.8)	7.8 (2.8)	10.7 (1.1)	18.9 (5.9)	12.1 (2.1)	33.6 (2.5)	7.3 (1.5)
Scots pine	8.3 (2.1)	8.5 (0.9)	10.8 (1.8)	9.4 (1.7)	15.8 (2.4)	14.3 (2.3)	28.9 (2.3)	9.5 (1.9)
Sycamore maple	5.2 (0.7)	10.4 (2.1)	2.9 (0.6)	11.9 (1.2)	11.7 (2.1)	15.8 (2.1)	28.5 (1.6)	9.3 (1.5)

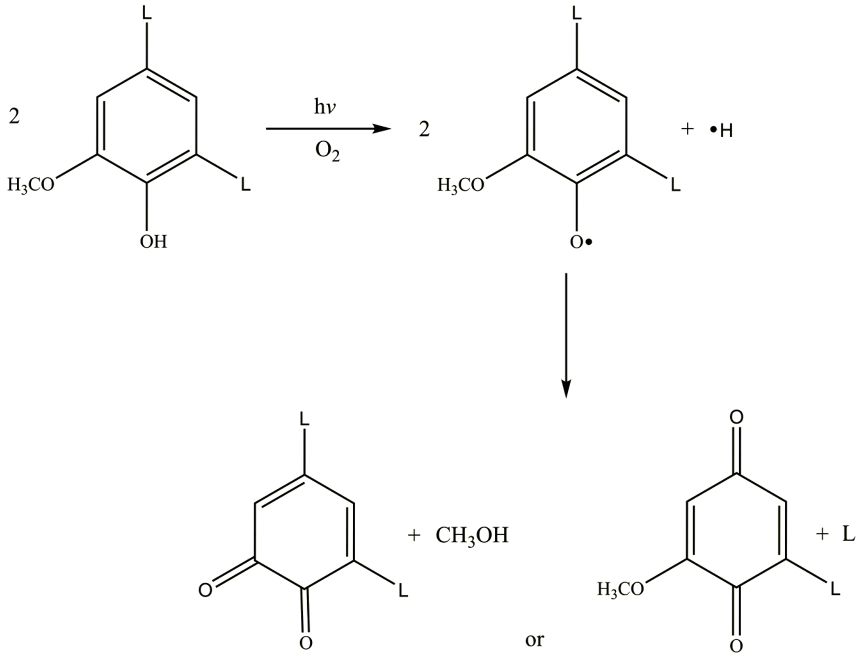


Figure 1.76 Proposed lignin photo-degradation mechanism by Anderson et al. (1991).

The subsequent decrease in yellowness may be attributed to the leaching by water of decomposed lignin and extractives (Turkoglu et al., 2015). The changes in a* values are determined mainly by the changes in the chromophore groups in the extractives. The results confirmed that in the early stages of weathering, dark woods tend to become light and light woods to become dark or turn into the silver grey colour (Dawson et al., 2008; Saei et al., 2015).

These photodegradative changes were initially thought (Hon and Ifju, 1978) to be limited to the outer 75−200 μm of the wood, but subsequent work (Kataoka and Kiguchi, 2001) has shown evidence of photo-induced degradation with extended exposure time at depths of around 500 μm. Figure 1.76 shows one example of a lignin photo-degradation mechanism given by Anderson et al. (1991), which suggested that lignin fragments can be activated via quinone methide intermediates which then undergo attack by light-induced radicals. It has also been proposed that, as a result of further photochemical cleavage of the C-C bonds adjacent to carbonyl groups, small, water-soluble, and leachable chemical compounds can be generated and removed during wetting cycles.

Moisture – One of the principal causes of weathering is the frequent exposure of the wood surface to rapid changes in moisture content. Indoor climates tend to be fairly constant in terms of their moisture content, except for areas with high moisture or liquid water (e.g., bathrooms, saunas, kitchens) or as the result of water leakage. In exterior use, wood is more prone to exposure to great variations in moisture, particularly if exposed in Use Class 3 situations, when direct rain or dew falling upon an unprotected surface is quickly absorbed by capillary action into the surface layer of the wood, followed by adsorption within the wood cell walls. The penetrative effect of the rain and moisture depends on the severity and time during which the wood undergoes the wetting process, and the dimensions of the wood element also contribute to the overall effect of wetting through the cross section of the material. The adsorptive uptake and release of water by the wood results in swelling and shrinkage, accentuated by changes in the climatic conditions, particularly when a wetting episode is immediately followed by exposure to direct sunshine. This results in stresses between the surface and the (often) drier interior, which can result in distortion or checking. The grain can rise as a result of poor wood processing, and differential swelling and shrinkage of the earlywood and latewood subsequently occur when the wood is wetted. The wetting process can also result in lifting of the grain, which also increases the level of leaching from the surface as a result of the UV-degradative process due to exposure to light. As the surface becomes more stressed, cracking leads to moisture ingress below the surface layers where photodegradation has occurred, opening up new sites for photodegradation as well as creating sites for possible colonisation by staining and decay fungi.

Building location – The position of a building and the exposure of its wooden elements can have a significant role on the weathering (micro-climate), degradation and performance of the material. The major factor is whether materials are in direct sunshine. This is further aggravated by the direction of the prevailing wind (and thus wind-driven rain). Further complications can be created by the channelling effects of surrounding mountains, ridges, tall buildings, etc. Typically, materials such as uncoated timber cladding will become grey over a relatively short period of time, with surfaces having unfavourable orientations undergoing a more rapid and variable weathering. Those having a less severe orientation (e.g., northern facing in the northern hemisphere), weather at a slower rate and more uniformly.

The orientation of a building and its components with respect to prevailing weather conditions significantly influence the periods of wetting experienced by products such as cladding. The ISO 15927-3 (ISO, 2009) standard describes how to determine the hygrothermal performance of buildings and more specifically their vertical surfaces and takes into account factors such as terrain, topography, wind sheltering and the presence of obstructions within close proximity. Many of these factors were also considered within the European-funded WoodBuild project, which considered the performance of a wooden material in terms of decay risks (Isaksson et al., 2015) and factors relating to weathering can be similarly applied.

Building design – In addition the location of a building, its design can also affect the weathering of wood components. The traditional design in a specific region often considers performance and modern design methods may pose a great stress on the performance of the materials. The use of eaves can help protect timbers from weathering from sunshine and excessive wetting (and ultimately from decay) but at the risk of creating different degrees of photodegradation and staining on a surface. This can be seen as unsightly by some, although over time all materials tend to achieve a uniform greying. The design should not only take into account how the material can be protected from the effects of exposure, but also how the material recovers after a weathering episode. When exposed to wind-driven rain, this means how long it will take for the material to dry back to normal conditions (i.e., to below the moisture threshold associated with fungal degradation). With timber cladding, ventilation has been shown to provide a better reduction than non-ventilated boards of the risk associated with wetting as shown in Figure 1.77 (Isaksson et al., 2015).

In a similar way, exposure at ground level tends to pose additional problems, not only from increased exposure to sunshine but also increased wetting as a result of splashing. Therefore, it is important to design the construction so that wood products are maintained at a height above ground level. There may also be regions which, due to the design of the building, are subject to splashing—and this can lead to the wood weathering faster. For wooden products, such as cladding, keeping the material at least 250 mm above the ground level is considered to be a practical means of limiting the risk of wetting.

Temperature – Although it is not as critical as UV irradiation or water, the temperature increases as a result of solar exposure and this affects both photochemical and oxidative reactions. Exposing wood to a high temperature after a wetting episode can lead to the rapid drying of the surface creating stresses that increase the risk of splitting and checking, which in turn can accelerate weathering and decay. Similarly, the expansion and contraction of water as a result of freezing and thawing may lead to similar degrees of wood checking.

Other climatic conditions – The effect of wind or wind-driven particles (such as sand) can accelerate the weathering process, and weakened surfaces may suffer fibre losses. This is often noticed as

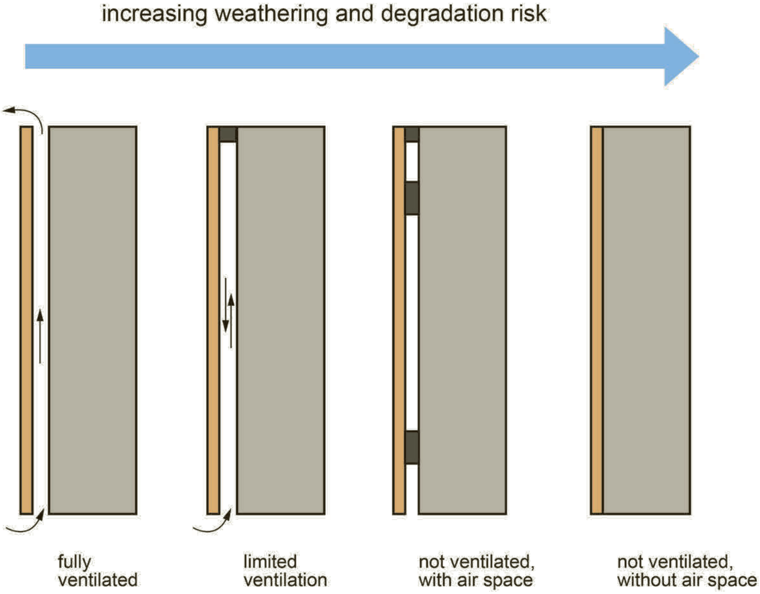


Figure 1.77 The way in which ventilation can alter the performance of timber cladding (modified from Isaksson et al., 2015).

raised grain on a wooden surface, as well as if the timber is coated with a deeply dark colour, which can cause extreme heating of the wood surface when exposed to intensive sunshine.

Human wear and tear – As with climatic abrasion, human use, such as walking on decking, can loosen fibres on surfaces that have undergone a period of weathering.

Effects of weathering

If left untreated, wood will suffer irreversible surface damage due to weathering. This damage can be divided into structural effects and aesthetic effects, both of which often necessitate physical replacement with significant financial implications (Figure 1.78).

Structural effects: the effects of weathering from the microscopic to the macroscopic level can be summarised thus:

Molecular level – UV-initiated degradation of selected bonds (e.g., $–CH_2$, –CH or –OH) within the lignin and the generation of carbonyl-containing extractives (Hon and Chang, 1984). In addition, there is evidence (Pandey, 2005) that cellulose can undergo a reduction in the degree of polymerisation, even though the crystalline zones are the most stable.

Cellular level – As the molecular degradation proceeds, there is an increasing effect on the surface of the wood, the degradation being followed by leaching and colonisation of staining fungi (Ghosh et al., 2009), with surface cracking increasing the available sites for such colonisation.

Tissue level – The degree of weathering has been shown to be more rapid in the less dense earlywood than in the latewood, as this leads to an uneven and rougher surface (Williams et al., 2001). In addition, surface cracks can occur on the radial section at the growth-ring border as a result of density variations in the transition zone (Sandberg, 1999).

All these factors contribute towards the overall ageing of the wood leading to a grey appearance when the material exposed over a period of time.

Although the aesthetic aspects of a material are subjected to individual opinions, it is generally accepted that wood has a "feel-good" factor and has links to health and well-being (Burnard and Kutnar, 2015). The most obvious way of evaluating the aesthetic appearance of wood is by visual

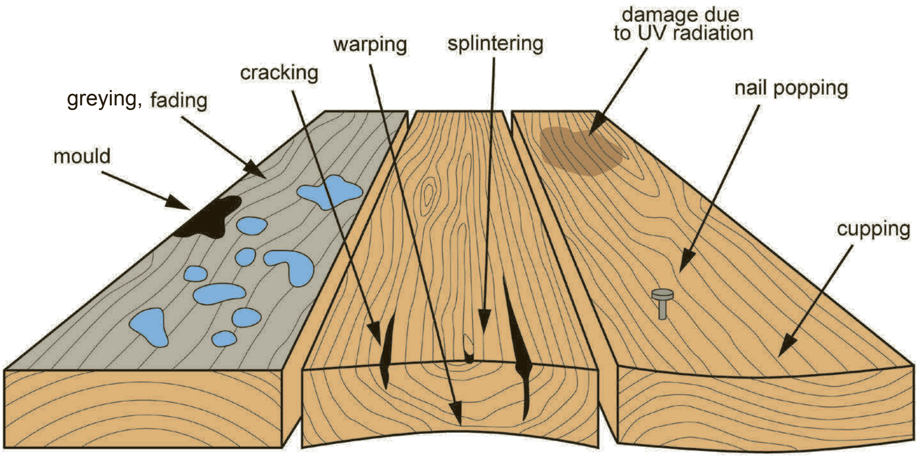


Figure 1.78 Examples of structural and aesthetic damage due to weathering.

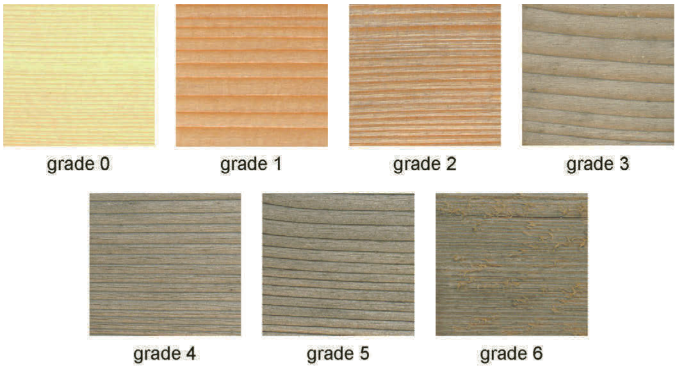


Figure 1.79 A proposed grading system for the degree of weathering of a wood surface (Sandak and Sandak, 2017).

assessment, but this can have a high level of variability, and the conditions under which an evaluation is undertaken can have an impact on the assessment, e.g., the amount of sunshine and dryness of the surface. An attempt to define conditions and provide some degree of quantification has been devised (Osgood et al., 1957), which for a material such as wood depends on the observed colour (or its greyness), surface cracking, checking, erosion, etc. For consistency, it is prudent to undertake evaluations by persons with some degree of training, and preferably to repeat evaluations carried out by the same person. In a recent overview of aesthetics, a grading system for evaluating the degree of weathering on wood was recommended, as shown in Figure 1.79 (Sandak and Sandak, 2017).

One of the key methods for limiting the weathering of wood has been by the application of a coating, such as paint. Here, it suffices to recognise the importance of this topic, and the reader is directed to several key reviews on this subject (Evans et al., 2015; Petrič, 2017; Teaca et al., 2019).

Monitoring weathering

When studying weathering, it is important to consider whether studies have been made under artificial conditions (e.g., by using a climate chamber) or on natural weathering (outdoor exposure). With artificial weathering, specimens are exposed to fixed periods of exposure to UV and driven water to correspond to conditions associated with natural outdoor weathering. However, these methods do not account for wind effects, or for erosion by particulates. Different regions have different climatic conditions, as indicated by the Scheffer Climate Index (Scheffer, 1971). This has been demonstrated with the establishment of several accredited test sites worldwide for the outdoor weathering of various materials, including wood. Table 1.17 shows the different climatic conditions,

Table 1.17 Overview of some accredited weathering test sites, showing the variation in exposure to radiation (McGreer, 2001).

Location	Latitude	Longitude	Elevation (m)	Average ambient temperature (°C)	Average ambient RH (%)	Annual mean rainfall (mm)	Annual mean total radiation exposure (MJ/m^2)
Lochem (The Netherlands)	52°30’ N	6°30’ E	35	9	83	715	3,700
Hoek van Holland (The Netherlands)	51°57’ N	4°10’ E	6	10	87	800	3,800
Sanary (France)	43°08’ N	5°49’ E	110	13	64	1,200	5,500
Chenai Airport (Singapore)	1°22’ N	103°59’ E	15	27	84	2,300	6,030
Melbourne (Australia)	37°49’ S	144°58’ E	35	16	62	650	5,385
Townsville (Australia)	19°15’ S	149°46’ E	15	25	70	937	7,236
Ottawa (Canada)	45°20’ N	75°41’ W	103	6	73	1,910	4,050
Sochi (Russia)	43°27’ N	39°57’ E	30	14	77	1,390	4,980
Dharan (Saudi Arabia)	26°32’ N	50°13’ E	92	26	60	80	6,946

but more importantly the annual mean radiation exposure for timber at these sites (McGreer, 2001). Naturally, these conditions can vary on a year-by-year basis, depending on local weather during the timeframe of the exposure trial.

In addition to the test sites listed in Table 1.17, there are a number of other test sites capable of undertaking evaluations according to national and international standards.

The variation in annual mean total radiation exposure shown in Table 1.17 also demonstrates the difficulty in establishing a direct correlation between artificial weathering data and outdoor weathering. According to the European standard EN 927 Part 6 (CEN, 2018), a standard artificial weathering cycle takes a total of 12 cycles each of 1 week. Given these variations, there is no direct correlation between artificial and natural weathering, and the standard EN 927 Part 3 (CEN, 2000) states that the results of this test do not necessarily correlate with those from natural weathering. Both these standards relate to coated wood, but similar concepts apply to uncoated wood. Attempts were made within the WoodExter project (Jermer, 2011) to determine the exposure risks leading to overall decay of wood, taking in account sunshine, temperature, rainfall and prevailing wind conditions, all based on climatic data that could be processed through the computer software Meteonorm. This showed (Figure 1.80) a range of exposure risks across Europe, ranging from 0.6 in northern Scandinavia to 2.1 on the Atlantic coast in southern Europe. It is prudent to acknowledge that the effect of sunshine bleaching the wood surface will be much greater in areas such as southern Europe than in northern Europe, for example, but the overall weathering effect maybe reduced by the limited rainfall and subsequent less leaching of photodegraded components.

Although the monitoring of prevailing conditions can provide an indication of the weathering of a material, accurate results can be obtained only through the actual observation of timber in service. A number of methods have been used to determine the effects of weathering, on a variety of sample sizes. Microtensile testing of microtomed samples has been investigated (Derbyshire et al., 1995; Turkulin et al., 2004) and this initially demonstrated that the method gave a reliable means for determining the photostability of wood, and later how moisture increased the strength loss of the thin strips, although this was sometimes after an initial increase in strength, which was attributed to changes in the cellulose. Further studies of the microtensile strength (Klüppel and Mai, 2018) demonstrated a greater loss of finite-span tensile strength on exposure to sea water, but not of zero-span tensile strength. Furthermore, FTIR of these samples showed higher levels of lignin than in conventionally weathered samples, presumably as a result of the deposition and crystallisation of salt within the cell-wall nanopores, which in turn inhibited lignin photodegradation. Diffuse

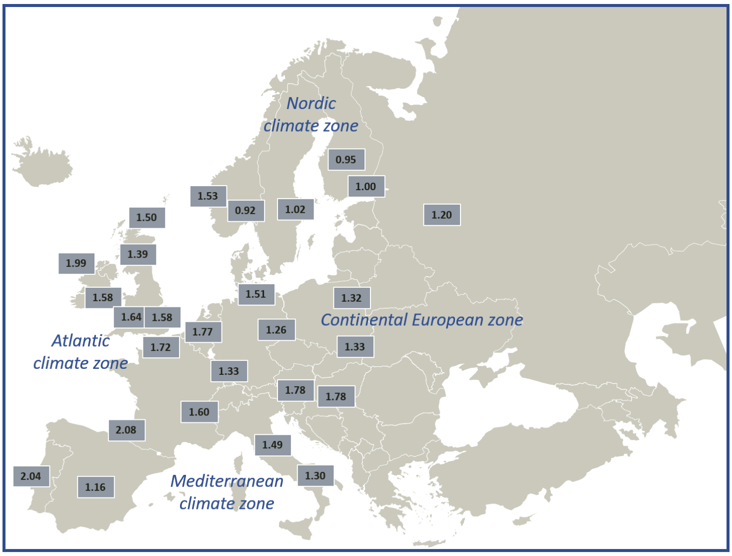


Figure 1.80 Exposure risk for wood product according to Meteonorm data (Jermer, 2011).

Reflectance Infrared Fourier Transform (DRIFT) Spectroscopy (Faix and Nemeth, 1988) used reflectance spectra instead of the conventional transmissive spectra in FTIR. Spectral equipment has now become portable, and handheld systems have made it possible to make field measurements to be taken.

More recently, the use of Near Infrared (NIR) Spectroscopy has gained in popularity and has been used (Sandak et al., 2016) to determine the kinetics involved in the weathering process based on lignin measurements, and thus to suggest weathering index rates for different wood samples. The use of NIR was part of the toolbox of evaluation methods, together with colour measurement, gloss measurement, and the spectral and statistical analysis of artificially weathered samples (Petrillo et al., 2019), showing the potential of a multi-sensor approach.

Although most spectroscopic methods have focussed on an analysis of the photodegradation of lignin, X-ray diffraction in association with FTIR has been applied to assess the cellulose crystallinity in weathered wood (Lionetto et al., 2012). Turkulin (2004) reported the use of scanning electron microscopy (SEM) for evaluating the natural and artificial weathering of wood. For unweathered wood, the mechanical properties of latewood dominated the tensile behaviour of the strips, with tension failure beginning in the latewood via a brittle-like mode (Figure 1.81, left), and spreading to the earlywood zones in an interlocked, ductile mode. Weathered wood were however more prone to brittle failure (Figure 1.81 right).

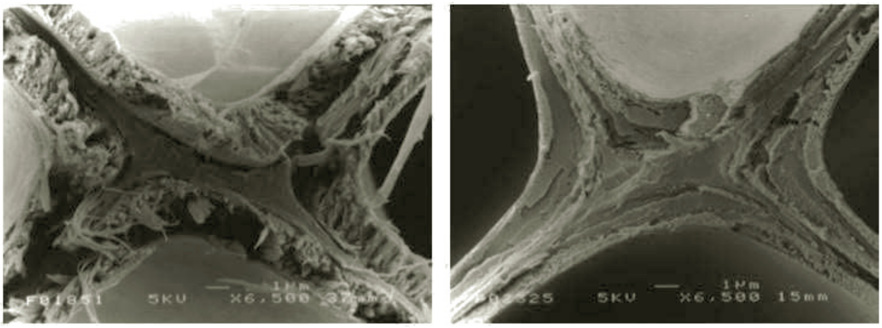


Figure 1.81 SEM analysis of conifer earlywood tracheids at their corner joint. Unweathered samples (left), and samples naturally weathered for 14 days (right).

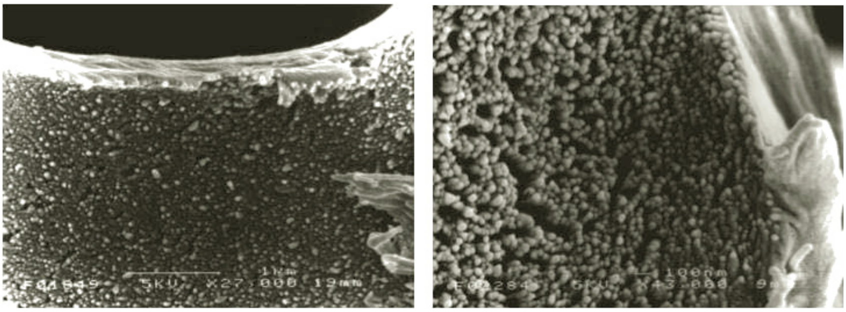


Figure 1.82 High resolution SEM microscopy of conifers. Unweathered wood showing S_2 and S_3 layers (left), and S_2/S_3 layers of a weathered earlywood tracheid with micro-voids between the fibrils due to delignification (right).

This work also reported that the breakdown of the middle lamella during weathering often causes the detachment of surface cells, while the thinning of the cell walls (Figure 1.81, right) was attributed to the breakdown of the lignin in the S_2 layer of the cell wall. Higher resolution microscopic images (Figure 1.82) further emphasise the photodegradative delignification, with the exposed fibrils being loosely packed and lacking radial agglomeration (Figure 1.82, right).

Wood modification and weathering

The aim of wood modification is to provide a more stable substrate for use in products exposed to various Use Classes. Many of the modification processes occur with chemical bonding other than that directly affected by the UV-degradation process, so that the weathering may occur at a rate different from that of the unmodified material. Some overviews of early studies into the effects of chemically modified wood have been published (Plackett et al., 1992; Evans et al., 2000; Williams, 2005), together with some studies into thermal modification (Nuopponen et al., 2005; Yildiz et al., 2011; Srinivas and Pandey, 2012). Recent studies into the effects of the various wood-modification techniques are reported in the relevant sections of this book.

1.7 Stress-strain response of wood: Considerations in wood modification

A structure is often defined as “any assemblage of materials which is intended to sustain loads”, such as an airplane, a bridge, or a building. Only a minority of structures are made by human beings; the rest are products of biological design. The majority of living tissues have to carry mechanical loads of one kind or another. Plants, such as trees, are structures, designed to grow tall and stand up to strong winds. Biology places a great premium on strength and metabolic efficiency. In fact, trees are very efficient structures indeed. Nowadays, with modern knowledge, we tend to shy away from making too sharp a division between structure and material, at least in sophisticated structures. In biology, the distinction is often not possible.

The mechanical or strength properties of wood determine its fitness and ability to resist an applied or external forces, an external force being any force outside a given piece of material that tends to alter its size or shape or to deform it in any manner. Deformation may also be brought about by forces acting entirely within a piece, such as those which arise in wood due to changes in its moisture content, but these forces are concerned chiefly with the physical properties of wood other than those strictly pertaining to strength (Wangaard, 1950).

The English natural philosopher, architect and polymath Robert Hooke (1635−1703) studied the effects of forces on different materials and structures. He took a considerable variety of wires, springs, and wooden beams and loaded them progressively by adding weights to scale-pans, and measured the resulting deflections. When he plotted the variation in load against the deflection he found in each case that the graph was a straight line, and when the load was progressively removed the recovery was also linear, and the specimens returned to their original dimensions when they were unloaded. Thus, Hookean behaviour of a material or a structure was discovered, and Hooke concluded:

"It is very evident that the rule or law of Nature in every springing body is that the force or power thereof to restore itself to its natural position is always proportionate to the distance or space it is removed therefrom, whether it be rarefaction, or separation of the part from one another, or by condensation, or crowding of these parts together. Nor is it observable in those bodies only, but in all other springy bodies whatsoever, whether metal, wood, stone, baked earth, hair, silk, bones, sinews, glass and the like."

Hooke was saying that a solid can resist an external force only by changing its shape: by stretching if it is subjected to a tensile force, or by contraction if it is compressed. There is, according to Hooke, normally no such thing as an absolutely rigid material or structure. His discovery was a logical consequence of Newton's third law. A perfect elastic material follows Hooke's law of 1678 which requires small deformations and states that the stress (σ) is directly proportional to the strain (ε) and independent of the strain rate:

$$\sigma = E\varepsilon \tag{1.7}$$

The modulus of elasticity (E) is a material dependent constant, corresponding to the inclination of the straight line in the stress-strain plot. Nowadays, it is known that materials in general do not show soley Hookean behaviour under load, and that some materials behave in a fully elastic manner but not linearly elastic as Hooke suggested for all materials. The basic nature of this complex behaviour of materials under load is exemplified in Figure 1.83:

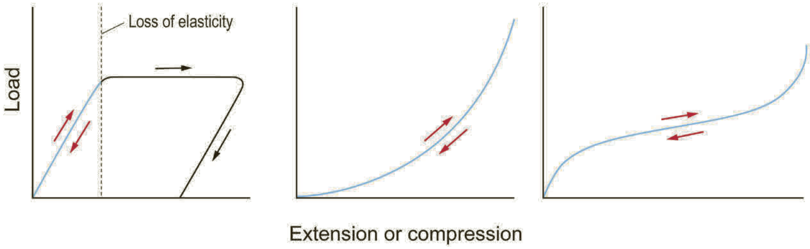


Figure 1.83 Examples of general stress-strain responses of different types of materials and structures.

Left: The elastic or plastic behaviour shown by a ductile metal. Under small loads the extension or compression increases linearly with increasing load (Hookean), but when a certain load is reached, the metal yields, undergoing a large additional extension or compression, that is mostly irreversible when the load is removed.

Middle: Fully elastic J-curve behaviour shown by many animal tissues. The greater the load, the smaller the additional extension or compression, and when the load is removed the material return to its original dimension.

Right: Fully elastic S-curve behaviour shown by many synthetic rubbery solids. Most extension or compression occurs over a relatively narrow medium load range. Such solids may be brittle under higher loads, i.e., when reaching the region of steep increase in load to the right of the curve.

Wood is a material with a complex behaviour under load, that depends not only on the external load such as force, moisture or temperature, but also on its ultra-, micro-, and macrostructure, as well its chemical constituents. Wood is an "elasto-viscoelastic" material because its response to loads may be Hookean (linear elastic) but also shows a time-dependent deformation behaviour depending on the loading history, the temperature, and the moisture content, as well as on moisture variations (Navi and Sandberg, 2012). These parameters may interact together producing coupling effects. It is essential to understand the elasto-visco-plastic behaviour of wood during wood-modification processes such as steam-bending (the Thonét method, see Figure 1.92) and densification, where an understanding of the stress-strain response of the wood material is essential.

Elastic behaviour of wood

Wood has an anisotropic mechanical behaviour, the strength and rigidity of wood in its longitudinal (L) direction being much greater than in the transverse, radial (R) and tangential (T), directions, and strength and rigidity in the radial direction being different from that in the tangential direction. In a tree trunk, which is more or less cylindrical, the circular shape of the growth rings and the organisation of the longitudinal cells give the wood axis-symmetric mechanical properties. Apart from defects and natural growth imperfections, the similarity of the two local symmetry planes, i.e., longitudinal-radial (L-R) and radial-tangential (R-T) passing through any given point means that wood may be considered to be a cylindrical orthotropic material. Figure 1.84 shows an idealised cylindrical trunk and a close-up element with the principal directions R, T, and L corresponding to a given arbitrary point in the trunk.

Under a longitudinal tensile force, wood is typically elastic with a quasi-linear elongation up to a breaking point, as shown in Figure 1.85 (left). Breaking occurs by a brittle fracture, under controlled force conditions when the ultimate strain is 1−3%. Nevertheless, when the displacement is controlled, the response of a wood specimen to a simple tensile force, as shown in Figure 1.85 (right), is different. The force displacement curve shows a strain-softening behaviour after the peak, as a consequence of strain localisation (fracture with a damaged zone) in the wood.

When wood is subjected to a tensile force in the transverse direction, the force-displacement curve have features similar to those in the longitudinal direction, but the breaking and peak stresses are much lower.

Under a compression force, the deformation of wood depends on the direction of loading. Typical stress-displacement curves of poplar under compression in the three principal directions radial, tangential and longitudinal under controlled deformation are shown in Figure 1.86.

Under axial compression, the specimen shows an almost three segmented stress-strain curve: a quasi-linear segment followed by a non-linear second segment curve with a negative slope due to localised longitudinal buckling of the cell walls and/or local fracture. The third segment shows an increase in the modulus of the specimen with increasing compressive force.

Transverse compressive stress-strain curves (corresponding to the radial or tangential direction) also consist of three segments. The first segment shows a quasi-linear behaviour and is followed by a non-linear curve showing a decreasing modulus corresponding to a transverse flexural buckling of the cell walls. The third segment is a quasi-linear curve indicating a progressive increase in the modulus, which can reach a value higher than that of the first segment. During segments 2 and 3,

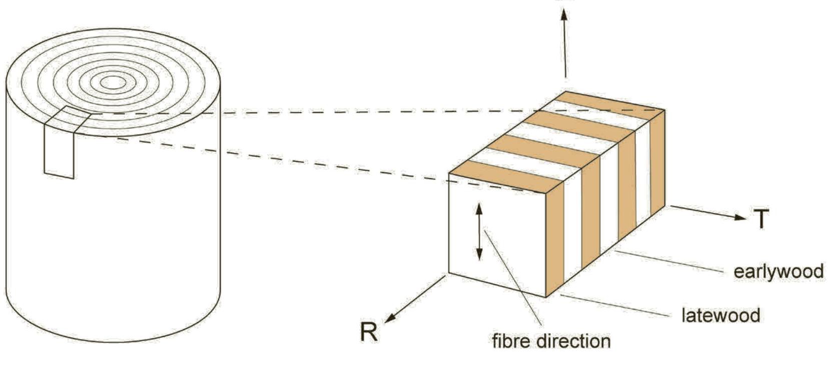


Figure 1.84 An idealised cylindrical trunk represented by a circular cylinder and circular growth rings (left), and a close-up view of a small element cut from the trunk where L, R and T are the local principal axes in the longitudinal, radial and tangential directions, respectively (right).

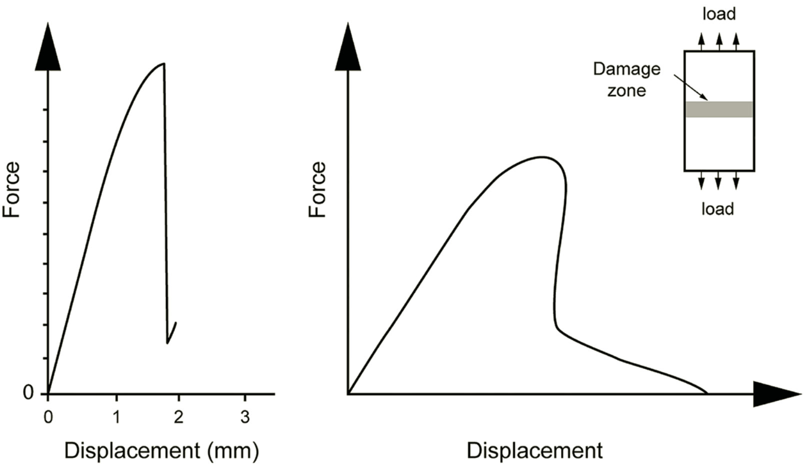


Figure 1.85 Typical force-displacement curve of a wood specimen subjected to a controlled longitudinal tensile force (left), and a controlled longitudinal tensile displacement (right) where the curve shows not only the linear region but also strain softening of the specimen after the peak force.

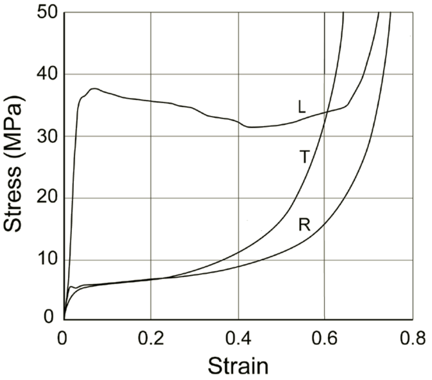


Figure 1.86 Stress-strain curves of specimens of poplar (density 350 kg/m^3) of dimensions 25 × 25 × 5 mm subjected to compression in radial (R), tangential (T) and longitudinal (L) directions under controlled displacement at a rate of 1 mm/min (Roussel, 1997).

the strain can be greater than 50%, indicating a large transversal deformation, which leads to a densification of the wood cells.

Viscoelastic behaviour of wood

Wood behaves in a viscoelastic manner, where its anisotropic mechanical properties depend on time, temperature and moisture. In relative terms, at short times, low temperatures and low moisture content, wood exhibits glassy behaviour and can be characterised as stiff and brittle. At long times, high temperatures and high moisture contents, wood exhibits a rubbery behaviour and can be characterised as compliant. The temperature associated with the phase transition between these two distinct regions is called the glass-transition temperature T_g (Wolcott et al., 1994). The glass-transition temperature is also known as the softening temperature, since it characterises the softening behaviour of amorphous polymers. When wood is subjected to thermo-hydrous conditions, its amorphous components soften and become easy to deform and this opens the way to many industrial processes such as moulding, densification, large bending, shaping, surface densification, etc. Many properties of the amorphous constituents in wood change dramatically when the material passes the glass-transition temperature (Figure 1.87).

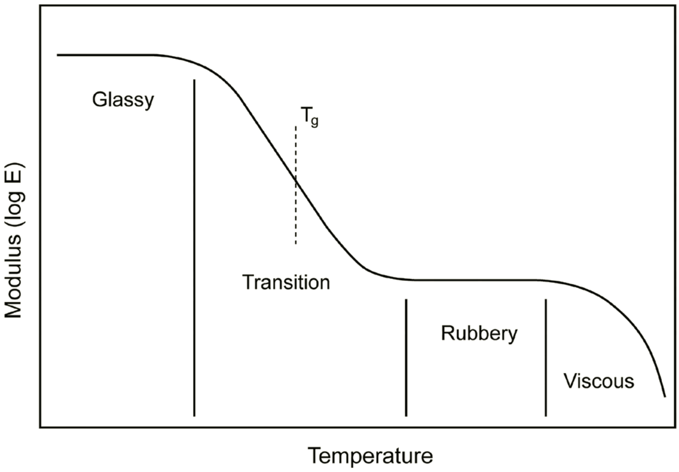


Figure 1.87 General behaviour of the relaxation modulus with temperature of an amorphous polymer (Lenth, 1999). T_g – glass-transition temperature.

The physical and mechanical properties of an amorphous polymer such as wood lignin, can be characterised as follows:

- In the glassy state, the physical properties are very similar to those of a solid phase whose values are not strongly sensitive to the chemical nature of the material like the topology of the polymeric network. The molecular movements which occur in this state are of low amplitude (movements of side groups or co-operative movements of a few monomeric units).
- In the rubbery state, the modulus of elasticity is 3,000 to 4,000 times lower than that of the glassy state, and the elongation at rupture is about 100 times greater. This high extensibility is due to the fact that thermal action decreases both the inter- and intra-molecular cohesion (Van der Waals forces, hydrogen bonds). Molecular movements of large amplitude (macromolecular movements) and the complete extension of the segments of the macromolecular chain are then possible, thanks to rotation around the covalent carbon-carbon (-C-C-) and carbon-oxygen (-C-O-) bonds.

Hillis and Rosza (1978) studied the influence of wood components on the softening of wood and suggested that moisture lowered the softening points of hemicelluloses and lignin, which are above 160°C when these two components are isolated. Hemicelluloses in the cell wall softened first (at 54–56°C), and this decreased the wood stiffness. This enabled wood fibres to adapt their cross-sectional shape to the applied forces. The softening of lignin (at 72–128°C) in the cell wall and middle lamellae permitted further cross-sectional movement within and between the fibres. Hillis and Rozsa (1985) investigated the softening of wood in different growth rings taken from young radiata pine trees. They reported softening points of about 80°C due to hemicelluloses and 100°C due to lignin, and based on the results of Hillis (1984), they attributed differences in the softening curves to the differences in the chemistry of the hemicelluloses in sapwood and heartwood. Baldwin and Goring (1968) have shown that T_g of the isolated components of wood differ from those in native wood, but the dependence of the T_g of wood components on the relative humidity is now fairly well known. Navi and Sandberg (2012) have presented thorough review of the glass-transition temperature of amorphous and semi-crystalline polymers.

The glass-transition temperature T_g of the amorphous components of wood decreases with increasing moisture content and vice versa (Figure 1.88). Östberg et al. (1990) showed that an increase in moisture content of Norway spruce and silver birch leads to a decrease in the softening temperature. Water molecules plasticise wood polymers, forming secondary bonds with the polar groups in the polymer molecules, and spreading them apart, thus reducing the secondary bonding

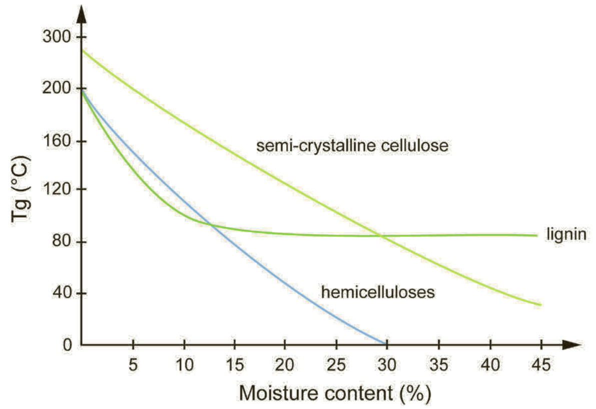


Figure 1.88 Glass-transition temperatures T_g of the isolated components in wood as a function of moisture content (Salmén, 1982).

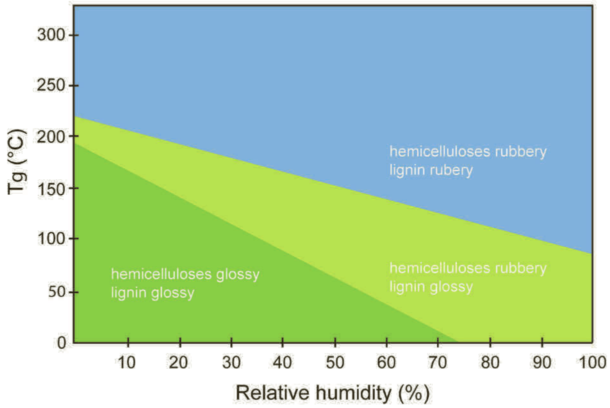


Figure 1.89 Glass-transition temperature for a matrix of native hemicelluloses-lignin as a function of the ambient relative humidity (Salmén et al., 1986).

between the polymer chains and providing more room for the polymer molecules to move around. Moisture thus increases the free volume of the system, the wood becomes more easily deformed and the glass-transition temperature is lowered.

Figure 1.89 shows the glass-transition temperature of the native matrix of hemicelluloses and lignin as a function of the relative humidity. It is, thus, essential to consider wood as a cellulose–hemicelluloses–lignin material whose three principal components are chemically linked together. Figure 1.89 shows that, at relatively high moisture content, the lignin has the highest glass-transition temperature, apart from crystalline cellulose. Therefore, the glass-transition temperature of lignin determines the limiting lowest temperature of the thermo-hydro-mechanical (THM) process for wood. The forming temperature must be selected according to two criteria. Firstly, the minimum temperature under which the wood can be formed, which is usually considered to be at least 25°C higher than glass-transition temperature of the lignin, i.e., approximately 110°C under moisture-saturated conditions and approximately 140°C at 80% relative humidity, and secondly, the maximum temperature, usually considered to be 200°C when air is saturated, to avoid thermal degradation of the wood components. The thermo-hydrous window for the forming of wood is thus limited to temperatures and relative humidity from respectively 110 to 140°C and 80 to 100%. Under these conditions, the lignin, hemicelluloses and the semi-crystalline cellulose are relatively mobile and can be deformed easily thanks to two molecular phenomena:

1) The inter- and intra-molecular interactions of the Van der Waals type in an amorphous or semi-crystalline polymer decrease strongly when their temperature is higher than their T_g.
2) The inter- and intra-molecular interactions of the hydrogen bond in an amorphous polymer which has functional hydroxyl (-OH) groups decrease strongly when its moisture content increases. The adsorbed water molecules are placed between the molecules, so they are by the hydroxyl groups. Consequently the average inter-molecular distance and the mobility of the molecules increase, and this lowers the T_g.

The water molecules act as a plasticiser by decreasing the interactions between the hemicelluloses and lignin macromolecules and the amorphous regions of the cellulose.

The semi-crystalline cellulose and hemicelluloses have many hydroxyl groups and are highly hygroscopic so that, when they are saturated with water, their T_g drops to a temperature lower than room temperature (Figure 1.88).

On the other hand, lignin contains fewer hydroxyl groups and water does not, therefore, have a great impact on its T_g which is approximately 85°C from about 15% moisture content to the saturation condition. The large number of hydrogen bonds in crystalline cellulose means that it has a compact and stable structure and that water is unable to penetrate into the crystal lattice. Only the hydroxyl groups on the surface of crystallites can adsorb water. Apparently the percentage of semi-crystalline cellulose is not really important. Consequently, the fibrils remain crystalline and very rigid at high temperatures and high moisture contents.

Various transitions in polymers are commonly studied by Dynamic Mechanical Analysis – DMA (Menard, 2007). DMA is a method based on sinusoidal tests that enable the characterisation of bulk properties directly affecting material performance. The DMA is a common method to decouple thermal activation from the time effect, characterising of materials that exhibit significant changes in their viscoelastic behaviour with changing conditions of temperature and the frequency of a dynamic force. It is often used to study the behaviour of polymers, e.g., to determine their glass-transition temperature.

DMA can be simply described as the application of an oscillating force to a material and the material's response to that force. DMA enables various properties of the material to be determined like the tendency to flow (viscosity) from the phase lag and the stiffness (modulus) from the recovery of the material. These properties are often described as the ability to lose energy as heat (damping) and the ability to recover from deformation (elasticity). The DMA also data relating to the relaxation of the polymer chains and the changes in the free volume of the polymer that occur. The storage modulus E', which is a measure of the material stiffness, the loss modulus E", which reflects the amount of energy that has been dissipated by the sample, and the ratio $E''/E' = \tan\delta$ (δ is the phase angle), an index of material viscoelasticity, are calculated from the material response to the sine wave. These different moduli give a better characterisation of the material, since they show the ability of the material to return or store energy, its ability to lose energy, and the ratio of these effects, which is called damping. In a DMA test, where a specimen is heated stepwise and the three parameters are plotted as a function of temperature, a strong change in $\tan\delta$ indicates a change in the material´s viscoelastic properties, e.g., the transition from a glassy to a rubbery state. Since cellulose, hemicellulose, and lignin are polymeric, DMA can be used for the characterisation of wood. Depending on the environment (humidity, air, inert-gas), thermal degradation and/or reconfiguration of the constitutive polymers of wood can occur during a DMA test revealing its viscoelastic properties (Assor et al., 2009). Internal friction ($\tan\delta$) is particularly sensitive to the structure of the wood cell-wall "matrix" (lignin and hemicelluloses). Therefore, DMA data as a function of temperature simultaneously provide mechanical data and indications of chemical modifications (Laborie, 2006). Due to the hygroscopicity of wood, DMA tests on wood as a function of temperature are usually conducted either in a completely dry or in a saturated state. However, the versatility of DMA has seldom been applied to monitor the *in situ* changes occurring during thermal treatment processes involving several steps under different conditions.

Mechano-sorption

The variation in the viscoelastic behaviour of wood under varying climatic conditions is referred to as the mechano-sorptive effect, a phenomenon that is still not understood in detail. The term *mechano-sorption* is often used to express the effect under carrying conditions of the coupling between mechanical stress and moisture content in wood. It has been shown that a variation in the moisture content of a wood specimen subjected to a mechanical load can lead to important deformations. Under high load and extreme variations in moisture content, high deformation can lead to structural damage of the wood. Figure 1.90 illustrates the effect of cyclic variation in the relative humidity combined with a different stress level on the delayed deformation in bending of small clear specimens.

Phenomena observed under varying moisture conditions have been described by Grossman (1976). These phenomena are presented in Figure 1.91 which shows a typical mechano-sorptive creep curve after deduction of the free swelling or addition of the free shrinkage observed under zero loading.

The main features are:

- The deformation increases during drying, as has been shown by Armstrong and Kingston (1962), Hearmon and Paton (1964), Pittet (1996) and others.
- The first re-humidification lead to an increase in the deformation. Subsequent re-humidification leads to a minor reduction in deformation when the applied compressive load is low and an increase in deformation under high compressive loads (Armstrong and Kingston, 1962; Hearmon and Paton, 1964; Navi et al., 2002).
- The mechano-sorptive deformation is independent of time. It is determined by the degree of variation in the moisture content while it is below the fibre saturation point (Armstrong and Kingston, 1962; Leicester, 1971).
- When the stress level is less than 15–20% of the short-term ultimate stress, the deformation due to mechano-sorption seems to be linear.
- A constant flow of moisture through the wood without any local change in the moisture content does not lead to any mechano-sorptive effect (Armstrong, 1972).

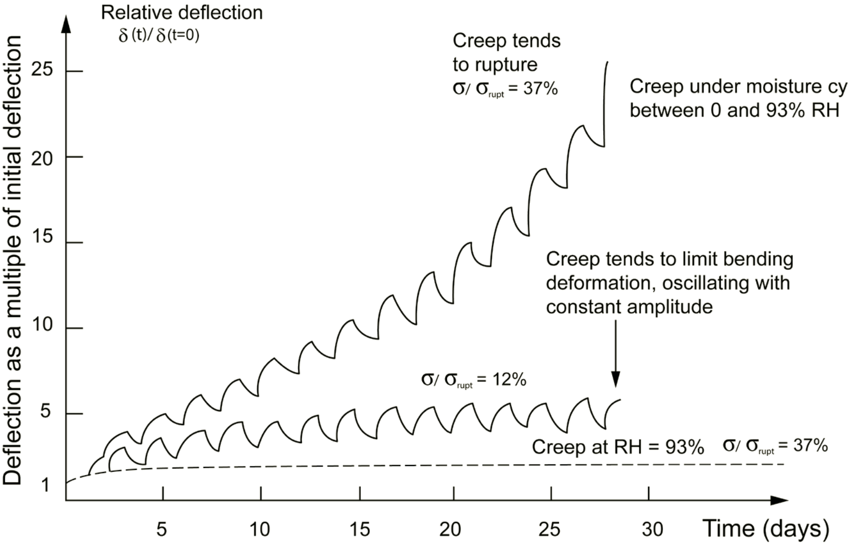


Figure 1.90 Creep in bending of small specimens of European beech with dimensions (2 × 2 × 60 mm) under cyclic variations in relative humidity (Hearmon and Paton, 1964).

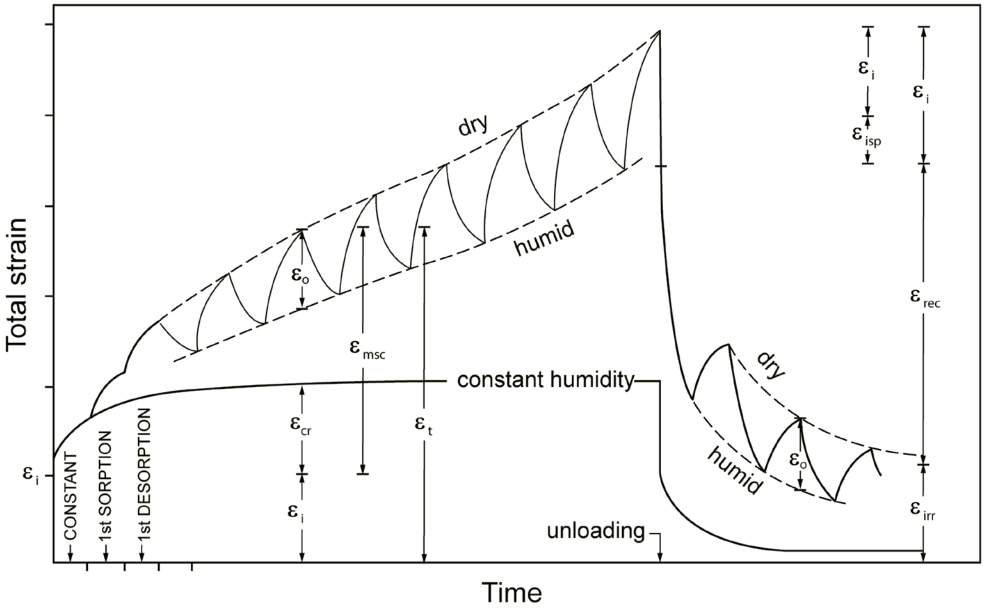


Figure 1.91 Typical curve showing the mechano-sorptive creep of wood (Grossman, 1976).

- After unloading and recovery of the instantaneous elasticity, most of the total deformation is irrecoverable. New humidification-drying cycles lead to a decrease the residual deformation. The recovery is greater during drying than during humidification (Armstrong and Kingston, 1962; Pittet, 1996).

It is important to note that the dimensions of the specimen have a considerable influence on the kinetics of the mechano-sorptive effect. This is partly related to the time to reach moisture equilibrium in the specimen. For example, in the case of two specimens, one 1 × 1 × 60 mm and the other 20 × 20 × 900 mm in size, the mechano-sorptive effect is similar, but the time necessary to reach moisture equilibrium in the thin specimen is only 2–3 hours, whereas about 50 hours are necessary for the thick specimen.

Stress-strain relationship under longitudinal deformation

The stress-strain relationship in the longitudinal direction of wood is of interest in bending after plasticisation. The reason for the difficulty in bending solid wood is the low extension of wood in tensile failure, see, e.g., Prodehl (1931a). Wood in its natural state exhibits elastic properties over only a limited stress range (Figure 1.92). When the stress is removed within this limited elastic range, the wood returns to its original shape. If the deformation in tension exceeds the limit stress in the longitudinal direction of wood, the wood remains bent. If the deformation strain exceeds the strength of the wood, it breaks.

However, when wood is plasticised it becomes *plastic shapable*. Its compressibility in longitudinal direction is then greatly increased, to as much as 30−40%, although its ability to elongate under tension is not appreciably affected (Figure 1.92). After plasticisation, a combination of bending and compression in the longitudinal direction of the wood can be used to limit the extension of the wood in tension and it is then possible to bend wood through a relatively sharp curvature. In practice, this means that the manufacturer has to control the length of the pieces during bending; to use some type of end stops (strap-and-stop) on the tensile side of the pieces being bent will prevent it from being streched by more than 1−2%.

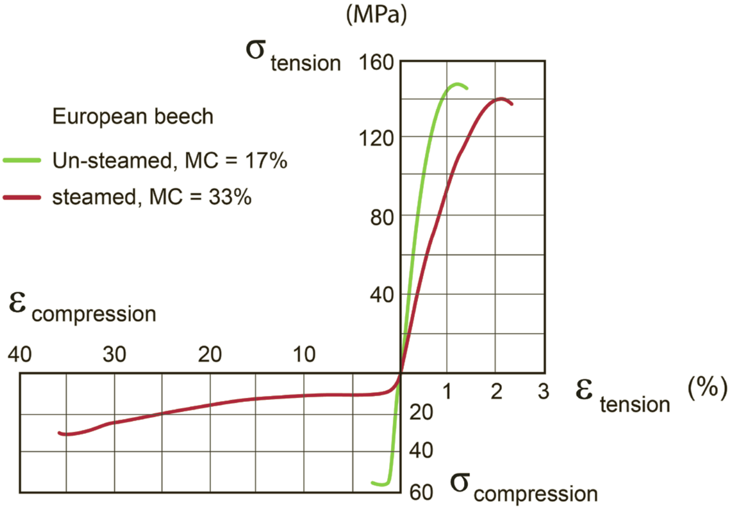


Figure 1.92 Stress-strain (σ-ε) diagrams for air-dry un-steamed beech and for steamed European beech. Note that the stress and strain axes have different scales to the right and to the left of the origin (after Prodehl, 1931a,b). MC – moisture content.

The theory of solid wood bending is relatively complicated, for the following reasons:

1) Wood is an elasto-visco-plastic material with no distinct yield point.
2) The yield points in tension and in compression are numerically very different, as shown in Figure 1.92, and the difference increases with increasing moisture content as well as with increasing temperature.
3) The strain to failure is much greater in compression than in tension.

Many researchers have studied the mechanisms of deformation of conifer and broad-leaved wood under compression at the cellular level (e.g., Kučera and Bariska, 1982; Gibson and Ashby, 1988; Hoffmeyer, 1990; Boström, 1992; François and Morlier, 1993; Gril and Norimoto, 1993; Roussel, 1997; Navi and Heger, 2005). In this section, only the large deformation of conifer wood is presented. In conifers at the cellular level, the microstructure is more simple and uniform than that of broad-leaved wood. In conifers, the longitudinal tracheids occupy approximately 90% of the volume and the remaining cells, the ray cells, are directed in the radial direction in the R−L plane (see Section 1.2 for details of the micro-structure of wood).

Physical mechanisms of deformations at the cell level under longitudinal tension

A tensile force tends to pull apart the material. This external force is communicated to the interior, so that each portion of the material exerts a tensile force on the remainder, due to the property of cohesion in the material. The result is an elongation or stretching of the material in the direction of the applied force. Wood exhibits its greatest strength in tension parallel to the fibres, i.e., in the longitudinal direction. A typical stress-strain response for a conifer specimen is shown in Figure 1.93. When a direct tensile load is applied, the strain is proportional to the stress up to the point of failure and there is no well-defined proportional limit. Wood is capable of only yielding a slight amount prior to ultimate failure in tension.

The strength of wood parallel to the fibre direction depends on the strength of its fibres and this is affected not only by the nature and dimensions of the wood elements but also by their arrangement. It is greatest in straight-grained wood with thick-walled fibres (high density), its strength is greatly reduced if the force is applied in a direction deviating from parallel to the fibres.

Failure of wood in tension parallel to the fibres practically always occurs when dry wood is subjected to bending. In tensile failure, the fibre walls are torn across obliquely and usually in a

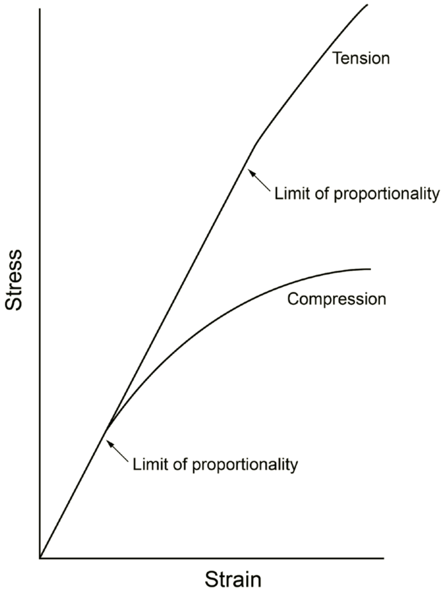


Figure 1.93 Stress-strain curves of wood subjected to tension and compression in the longitudinal direction under controlled displacement.

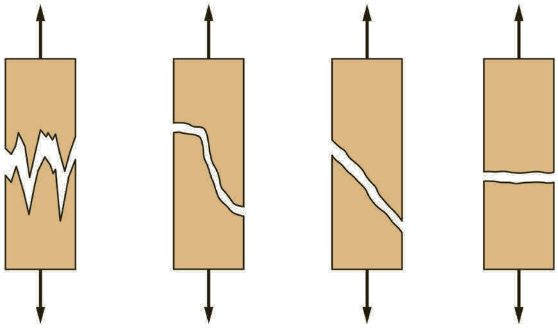


Figure 1.94 Failure types in clear wood stressed in tension parallel to the fibre direction in the wood. From the left: splintering tension, combined tension and shear, shear, and brittle tension failure.

spiral direction following the sprial-grain of the tree. There is practically no pulling apart of the fibres from each other, i.e., no separation of the fibres along their walls, regardless of their thickness. The nature of tensile failure is apparently not affected by the moisture content of the wood, at least not as much as the other strength values.

Figure 1.94 shows commonly occurring types of failure of wood loaded in longitudinal tension, such as splintering tension, combined tension and shear, shear, and brittle tension failure.

Earlywood and latewood zones of a conifer specimen loaded to failure in tension parallel to the longitudinal direction exhibit completely differing patterns as shown in Figure 1.95. The earlywood zone typically shows a brittle failure, with separation occurring across the tracheid walls. In latewood, the failure is typically a combination of shear and tension failure.

Physical mechanisms of large deformation under longitudinal compression at the cell level

In compression, the force acts on a body in the direction opposite to that in tension. As shown in Figure 1.96, the strain response of a low-density wood specimen to a uniaxial longitudinal compressive force can be divided into three distinct segments. The specimen behaves almost linearly up to a certain limit (B). The slope of the first segment of the curves (A–B) gives the

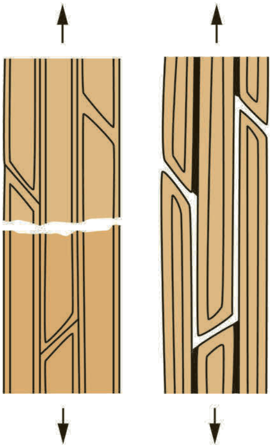


Figure 1.95 Most common failure of tracheids in tension parallel to the fibres: earlywood (left) and latewood (right).

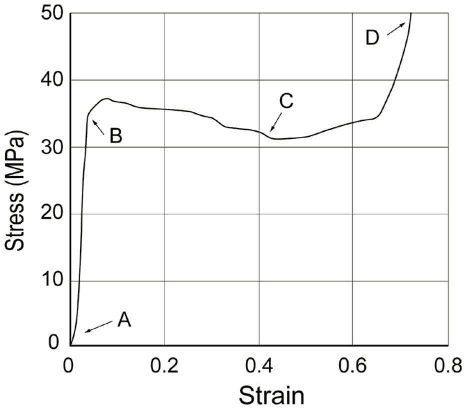


Figure 1.96 Stress-strain curve of a specimen of low-density wood subjected to compression in the longitudinal direction under controlled displacement.

effective Young´s modulus of the specimens in compression in the longitudinal direction. Above the linear limit, the specimen shows a smoothing or strain softening behaviour (B−C), followed by a rigidification or densification (C−D).

The softening behaviour can be explained by various mechanisms of deformation at the cellular level. Many researchers have observed this behaviour in various kinds of wood. For wood of low density (< 300 kg/m^3), Easterling et al. (1982) showed that the cause of the wood softening was the collapse of fibres by rupture at the ends of the cells. This mechanism is shown schematically in Figure 1.97 (middle). In wood of higher density, Kučera and Bariska (1982) have shown that the softening of the wood can be due to a local Euler-type buckling in the walls of the cells, illustrated in Figure 1.97 (right), which generally leads to the formation of a shear band as shown in Figure 1.98 on the wood macro-level.

Stress-strain relationship under transverse compression

The stress-strain behaviour of wood loaded in the transverse direction is of interest mainly under compression, where the cells are greatly deformed and the cell-lumen volume decreases, resulting in an overall decrease in the volume of the compressed specimen. Since the amount of material in the specimen is unchanged, the density increases with increasing compression, and the process is called *wood densification*.

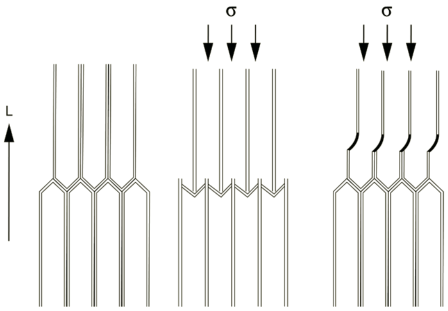


Figure 1.97 Diagrammatic representation of the mechanisms of local large deformations of wood cells under a longitudinal compression force. Wood before the application of the force (left), collapse of fibre by rupture (middle), and collapse by the buckling of cell walls (right). L is the longitudinal direction of wood.

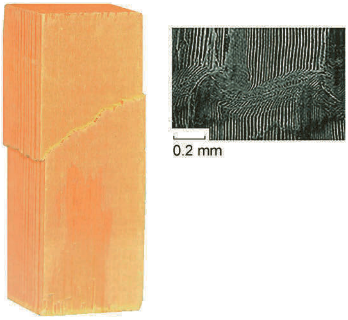


Figure 1.98 Collapse of the specimen by the buckling of fibre walls, which creates a shear band: observed at the macro-level (left), and at the cell-wall level (right).

Wood densification can be achieved in one or more directions, but wood is mostly densified along one of its orthotropic axes, diffuse broad-leaved wood being preferred to conifer wood for its anatomical structure. Primarily, wood and in particular conifer wood, is densified along the radial axis, as its latewood is much more dense than its earlywood. If it is tangentially densified, the latewood spreads into the earlywood forming waves or zigzags (Küch, 1951).

Densification in the transverse direction of wood flattens the cells without any noticeable damage on a macroscopic level, so that the strength increases with increasing density (Sandberg, 1998; Haller and Wehsener, 2004). Because of its viscoelastic nature, wood also exhibits rheological properties such as creep and relaxation. The strain-time curve of wood under a compressive load can be divided into four parts: initial elastic deformation, viscoelastic deformation, final elastic spring-back and time-dependent spring-back or creep recovery (Tang and Simpson, 1990). Wood densification can have both permanent and recoverable components, which together have a significant influence on the physical and mechanical properties of the material (Lenth and Kamke, 2001). The viscoelastic behaviour results in densification due to a permanent transverse compression of the cells.

During transverse compressive loading, a typical stress-strain curve of wood has three distinct regions (Bodig, 1963; Bodig, 1965; Kennedy, 1968; Wolcott et al., 1994; Uhmeier et al., 1998; Reiterer and Stanzl-Tschegg, 2001; Nairn, 2006), as shown in Figure 1.99. The initial part of the stress-strain curve for wood is a linear elastic region, in which the stress is directly proportional to strain. The second part is a “plastic” or collapse region, in which the stress is relatively constant even though the strain increases and the wood is deformed. After the plastic region, the stress increases strongly with little further strain. This region is termed the densification region (Tabarsa and Chui, 2000).

A yield point is found at the beginning of cellular collapse. When most of the cells have collapsed, densification begins (Wolcott et al., 1994). During densification, the stress rapidly increases as a result of the elimination of air voids and compression of the solid wood structure—consolidation

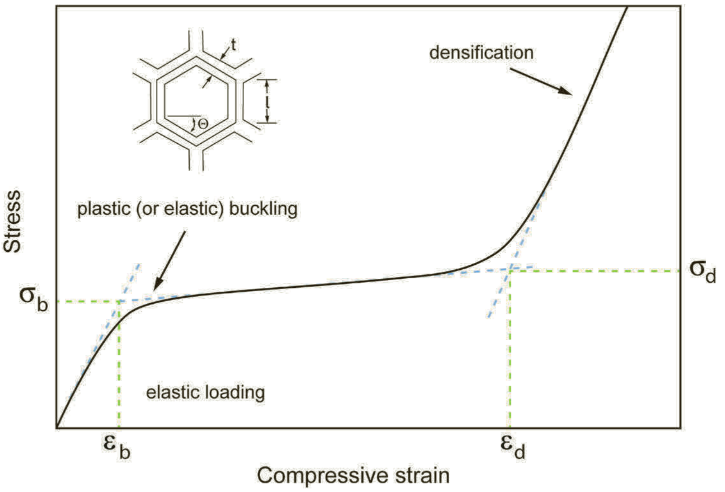


Figure 1.99 Schematic view of a transverse compressive stress-strain curve for wood (after Nairn, 2006).

of the collapsed cell walls. Cellular collapse occurs by elastic buckling, plastic yielding, or brittle crushing, depending on the test conditions and on the nature of the cell wall material (Wolcott et al., 1989).

The key details of the compression properties are dependent on various anatomical features of the wood specimen such as density, percentage of latewood, ray volume and loading direction (Nairn, 2006). Kunesh (1968) noticed that in the radial compression of solid wood, failure starts with the buckling of rays in an earlywood layer and results in progressive failure by buckling of the rays throughout the specimens. The first failure in earlywood was also found by Bodig (1965). Tabarsa and Chui (2000) found that earlywood primarily controlled the elastic and plastic parts of the stress-strain response for white spruce under radial compression. The first collapse of the cellular structure, which signified the onset of the plastic region, occurred in the cell layer with the lowest gross density in the earlywood. The initial part of the densification region was largely an elastic response of the latewood to the compressive stress, and collapse of latewood cells may not have occurred due to their large wall thickness. In broad-leaved woods, the first failure was initiated in the largest vessels surrounded by thin-walled paratracheal parenchyma cells (Tabarsa and Chui, 2001).

Several researchers have reported that wood responds differently to radial and tangential compression due to its anisotropic nature (Kennedy, 1968; Kunesh, 1968; Bodig and Jayne, 1982; Dinwoodie, 2000; Tabarsa and Chui, 2001; Wang and Cooper, 2005). In radial compression, the final consolidation stage is dominated by the elastic deformation of latewood, and in the tangential direction the final stage begins after readjustment of the latewood layer by buckling (Tabarsa and Chui, 2001). Reiterer and Stanzl-Tschegg (2001) studied the compressive behaviour of spruce wood under uniaxial loading at different orientations to the longitudinal and radial directions. Their results showed that the deformation pattern is highly dependent on the orientation. In the case of loading in the longitudinal direction, buckling deformation and cracks occurred, but no densification was observed, whereas loading in the radial direction resulted in plastic yielding and (gradual) collapse of the wood cells starting in the earlywood region of a whole growth ring and followed by densification at higher strains. Schrepfer and Schweingruber (1998) studied the anatomic structures of reshaped press-dried wood and found that earlywood cells were deformed more easily than latewood cells, which resulted in zones of compressed cells next to zones of uncompressed cells, in wave-like patterns. Kultikova (1999) also showed wave-like patterns of compressed and uncompressed cell zones in densified wood. The differences in compressibility of the wood tissue affects the distribution of void areas, and thus also the vertical density distributions and mechanical properties of compressed wood (Lenth and Kamke, 1996).

The type and amount of cell collapse have a great effect on the physical and mechanical properties of densified wood (Wolcott, 1989). The strength usually increases less than the density in relative terms, since uniaxial compression of solid wood results in a general collapse of the structure and possibly also in crushing and checking (Blomberg et al., 2005). This relation was found by Perkitny and Jablonski (1984) for bending strength and axial (parallel to grain) compressive strength. Blomberg et al. (2005) used a strength potential index to quantify how much the strength of densified wood increased relative to what could be expected for non-densified wood of similar density.

Hydrothermal treatment has a strong influence on the mechanical behaviour of wood during compression/densification. Softening and degradation occur depending on factors such as temperature, moisture, steam, and time (Morsing, 2000). The degree of improvement in the properties of densified wood due to hydrothermal treatment is affected not only by softening but also by the amount of thermal degradation induced by the compression process (Reynolds, 2004). Thermal degradation of amorphous wood components causes a weight loss of wood, and this can influence the mechanical strength properties (Jennings, 1993). For wet Norway spruce in radial compression, a thermal degradation process was observed between 150 and 200°C (Uhmeier et al., 1998).

Physical mechanisms of large deformations at the cell level under transverse compression

As shown in Figure 1.100, the stress-strain response of a specimen of poplar wood to uni-axial compressive force in the transverse direction has three distinct segments. The specimen behave almost linearly up to a certain limit which depends on the direction of the applied force. The slopes of the first segments of the curves (A−B) give the effective Young's modulus of the specimens in the radial and tangential directions, respectively. In the radial and tangential directions, unlike in the longitudinal direction, wood exhibits a typical plastic behaviour with a positive work hardening (B−C) followed by a rigidification or densification (C–D).

In the radial and tangential directions, the first zone represents a linear elastic behaviour, while the second zone (B−C in Figure 1.100) represents a plastic behaviour of the cell walls. In the third zone (C−D), a densification of the cells occurs.

Different researchers have explained these phenomena. Under the application of a compressive force in the radial direction, deformation occurs as a flexural buckling (or crushing) of the fibre walls starting in the weakest layer of the material. The cells of earlywood are the first to buckle because their walls are thinner (~ 2 μm) than the walls of latewood cells (~ 10 μm). This buckling leads to the densification of the fibres, the establishment of points of contact between the cell walls and increases the local rigidity so that this layer can support force sufficient to buckle the next layer which is the weakest one (Figure 1.101).

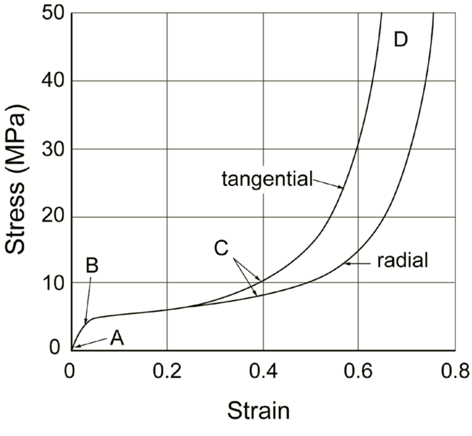


Figure 1.100 Stress-strain curves of specimens of low-density wood subjected to compression in the radial and tangential directions under controlled displacement.

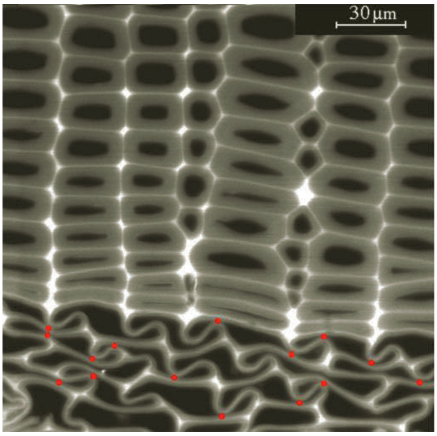


Figure 1.101 Micrograph of Norway spruce densified in the radial direction obtained with a confocal microscope. The contact points between cellular walls in the earlywood zone are shown by red dots.

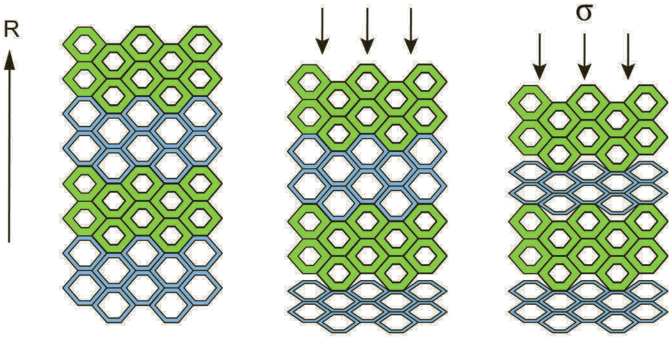


Figure 1.102 Diagrammatic representation of the compressive deformation of wood of the cellular level in the radial direction: wood before compression (left), localisation of the deformation and crushing of fibres in the weakest layer, partial densification (middle) and, crushing of fibres in the weakest layer followed by densification (right). R is the radial direction of wood.

These deformation mechanisms consisting of localisation, buckling and densification have been observed by, e.g., Gibson and Ashby (1988) and Navi and Heger (2005). A diagrammatic representation of this phenomenon is given in Figure 1.102. After the yielding of the first cellular row, the phenomenon is propagated progressively to the other rows of earlywood cells and later to the latewood cells.

In conifers compressed in the radial direction, the earlywood rows yield first, followed by the latewood rows, whereas in the tangential direction, thanks to the micro-structure of wood, buckling of the cell walls occurs at the same time in both latewood and earlywood cells.

The slope in the second segment of the curves increases with increasing compressive force, due to a closing of the lumens and multiplication of the points of contact between the walls of the cells of the initial wood. As the compression force increases, the thickness of these bands of buckled cells increases and others start to yield. When all the lumens are closed, the stress necessary to continue the deformation increases exponentially in the third segment. Wood densification starts in segment two and continues in segment three. As a result of this linear and non-linear behaviour, as shown in Figure 1.100, the wood can be deformed by more than 50%.

To model the behaviour of wood under compression deformation, constitutive equations of wood undergoing linear and non-linear deformation therefore become important.

Kutnar and Kamke (2013) studied the transverse compressive behaviour of Douglas fir wood and hybrid poplar at high temperature (170°C) and saturated steam conditions. They applied modified Hooke's law and modelled the compressive stress as a function of elastic modulus of cell wall substance and a non-linear strain function. They found that Douglas fir and hybrid poplar behave differently when subjected to transverse compression due to the different cellular structures of conifer woods versus broad-leaved woods. However, when the comparison was made on the basis of the relative density change, the transverse compressive responses of Douglas fir and hybrid poplar were remarkably similar. The relative density of a cellular material, defined as the ratio of the apparent density of the material to the real density of the solid of which it is made (Ribeiro and Costa, 2007), is a characteristic feature that significantly affect compressive behaviour of cellular materials.

The effect of the steam environment on the stress-strain response, non-linear strain function $\psi(\varepsilon)$, and relative density change can be studied by a modified Hooke's law based on the load-compression behaviour of flexible foams (Gibson and Ashby, 1988; Dai and Steiner, 1993; Wolcott et al., 1994; Lang and Wolcott, 1996; Dai, 2001; Zhou et al., 2009; Kamke and Kutnar, 2010) in the following form:

$$\sigma = \mathrm{E}\cdot\varepsilon\cdot\psi(\varepsilon) \tag{1.8}$$

where σ is compressive stress, E is transverse compression Young's modulus of the cellular material, ε is compressive strain, and $\psi(\varepsilon)$ is a dimensionless non-linear strain function. The value of $\psi(\varepsilon)$ can determined (Wolcott, 1989; Lang and Wolcott, 1996) as:

$$\psi(\varepsilon) = \frac{\in_y / C_2}{\varepsilon}\left[\frac{1-\rho_r^{1/3}}{1-\rho_r(\in)^{1/3}}\right]^3 \tag{1.9}$$

where ε_y is yield strain, C_2 is a linear elastic constant, ε is the compressive strain, ρ_r is the relative density of the wood (a ratio of the bulk-wood density to the cell-wall density, assumed to be 1,500 kg/m^3), and $\rho_r(\varepsilon)$ is the change in relative density under a compressive strain. A thorough development of (Equation 1.9) is provided by Gibson and Ashby (1988).

The change in relative density under a compressive strain $\rho_r(\varepsilon)$ at a given strain ε can be determined (Wolcott, 1989; Lang and Wolcott, 1996):

$$\rho_r(\varepsilon) = \frac{1}{\rho_r\left[1-\varepsilon_p+\frac{2}{3}\mu\varepsilon_p-\mu\in_p^2\right]} \tag{1.10}$$

where ε_p is plastic strain ($\varepsilon_p = \varepsilon - \varepsilon_y$) and μ is the expansion ratio defined as the ratio of lateral strain to compressive strain in the nonlinear stress-strain region.

The Young's modulus of the cell wall (E_{cw}) can be determined by an expression given by Wolcott (1989), where the Young's modulus of any wood species can be calculated as a function of the cell-wall modulus and the relative density:

$$E = C\, E_{cw}\, \rho_r^3 \tag{1.11}$$

where C is a constant, E_{cw} is the cell-wall modulus, and ρ_r is the relative density, i.e., the density of the wood divided by the cell-wall density.

1.8 Conclusions on wood and wood modification

The aim of this chapter has been to lay the basis of how wood modification has been developed and where it has occurred, been commercially manufactured. To do this, it is necessary not only to understand the wood modification processes, but also the substrate being modified, i.e., wood. As can be seen from subchapters herein, it is necessary to understand the basics behind:

- the different wood species and their structures,
- the chemical composition of wood and how these chemicals can react,

- the reaction between wood and its components and water,
- the biological nature of wood and its susceptibility to degradation,
- the effects of exposing wood to outdoor conditions, and
- the effects of loads on the mechanical performance of wood.

The introduction to these areas will provide the reader with a sufficient entry knowledge to more detailed understandings of the wood modification methods described in the following chapters. Based on the information in these chapters, combined with the introductions provided in this chapter, it is hoped that the reader will increase their knowledge of wood modification processes to help in new research and applications of this expanding technology.

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