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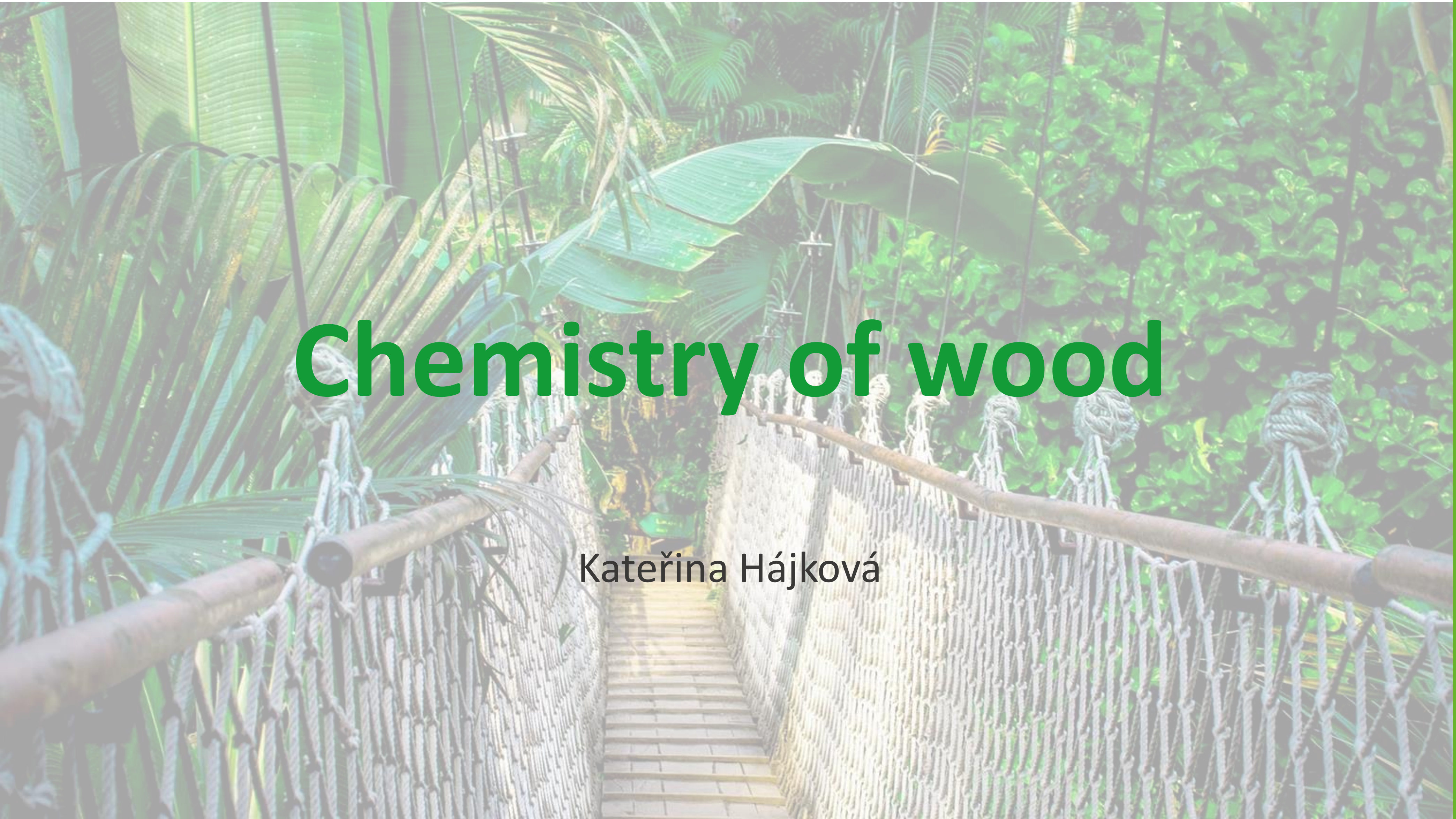

Chemistry of wood

Kateřina Hájková

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Chemical composition of wood

- C - 50%, O - 43%, N - 1%, H - 6%
- leaves - more protein, chlorophyll, H - 7%, N - 2%
- bark - more minerals, less C, O, more H
- bast - extractives, polyuronides, little lignin
- branches - more cellulose and pentosan
- kernel - more extractives

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Chemistry of wood

- macromolecular substances make up 95% of the wood mass of temperate trees
- adjuvants substances (extractives)
  - carbohydrates
  - fats, oils, waxes
  - essential oils (terpenes)
  - resin acids
  - tannins
  - proteins
  - sterols
  - aliphatic acids
  - minerals (ash)

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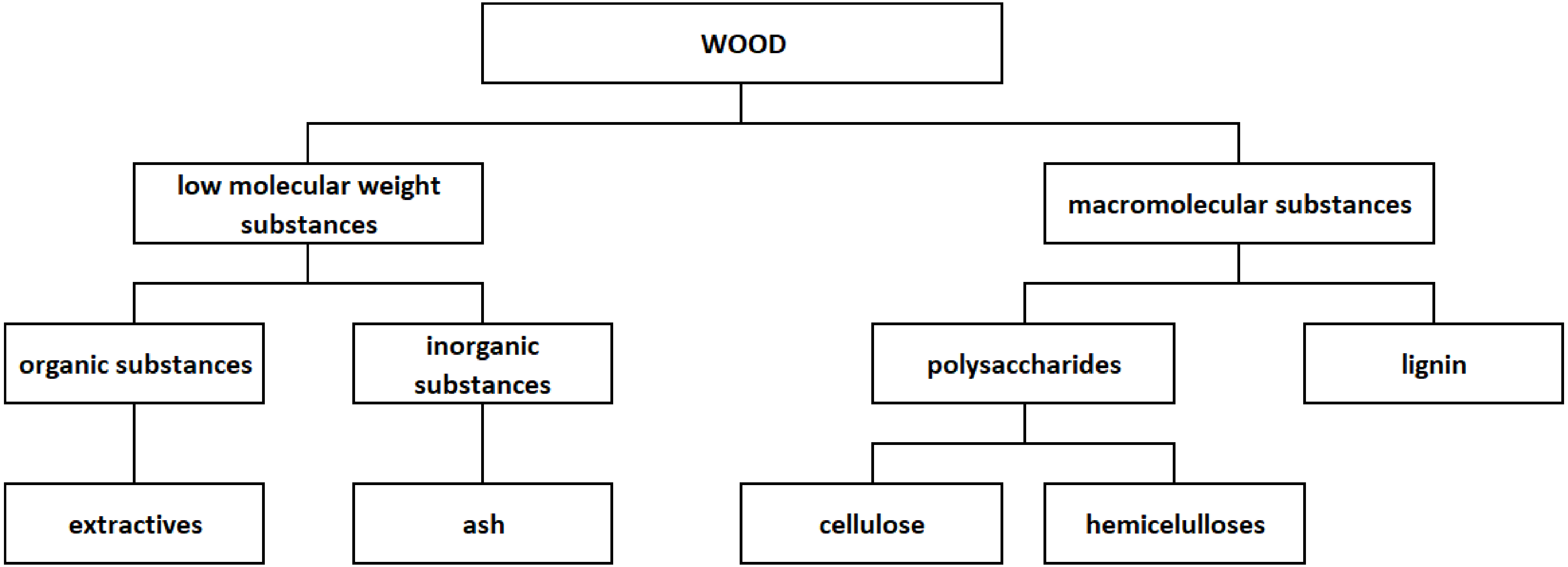
Chemistry of wood



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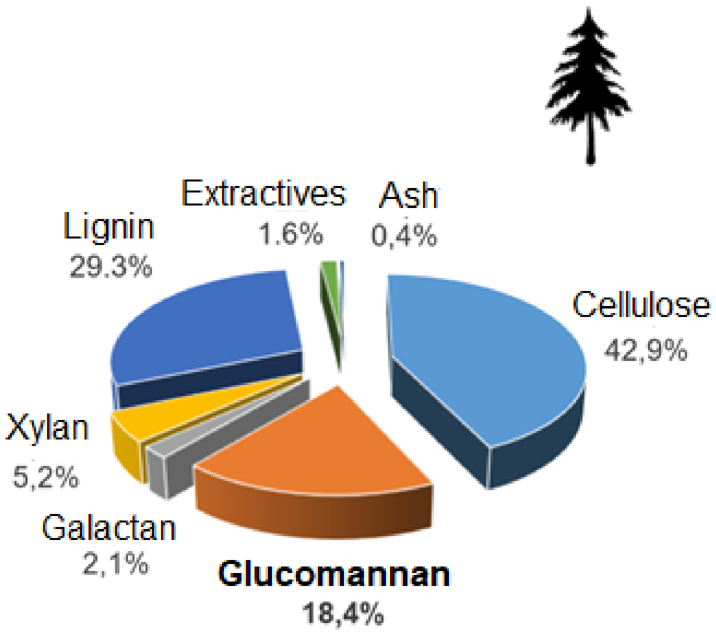
Content of basic components in wood

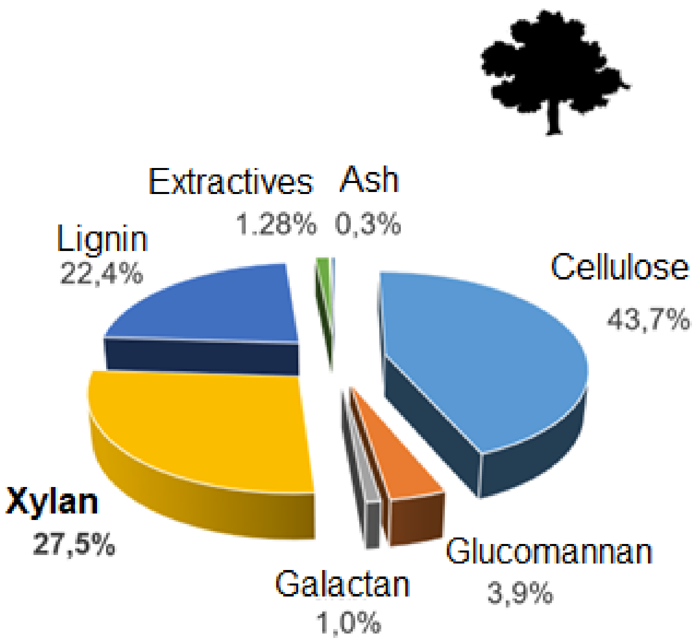
Spruce *(Picea abies L.)*

main component of conifers
glucomannan



Beech *(Fagus sylvatica L.)*

main component od deciduous trees
xylan



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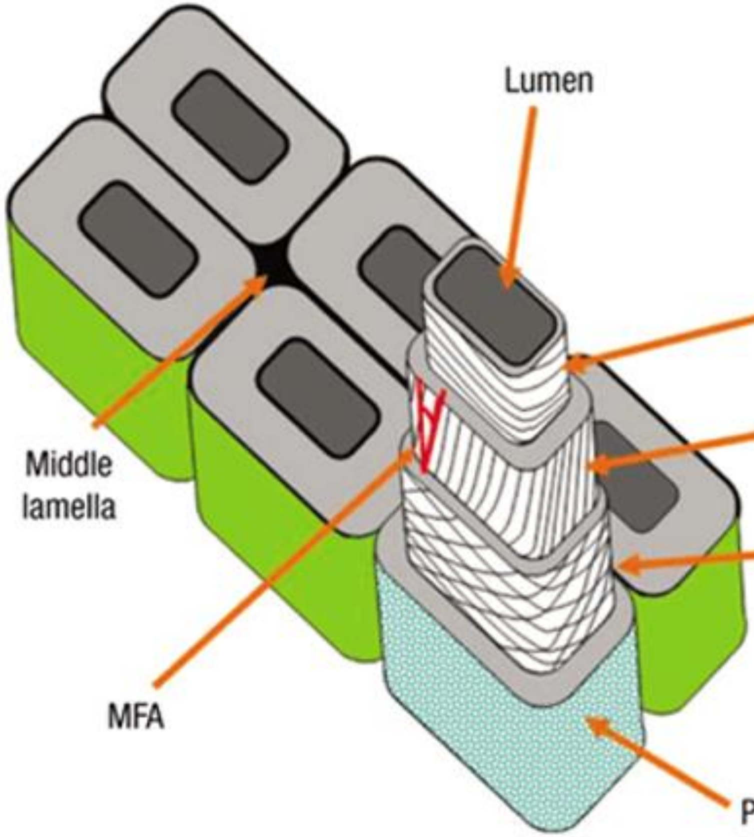
Microscopic aspect

- Cell wall:
  - Lumen
  - Middle lamella
  - Primary wall
  - Secondary wall: S1, S2, S3



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Cellulose

- the basic material of plant cells
- important material for all living matter
- the percentage of cellulose in plant material varies according to its origin

Cellulose

- isolation of cellulose highly dependent on the association of substances in the cell wall
- in wood, cellulose is associated with hemicelluloses and lignin
- isolated cellulose contains impurities

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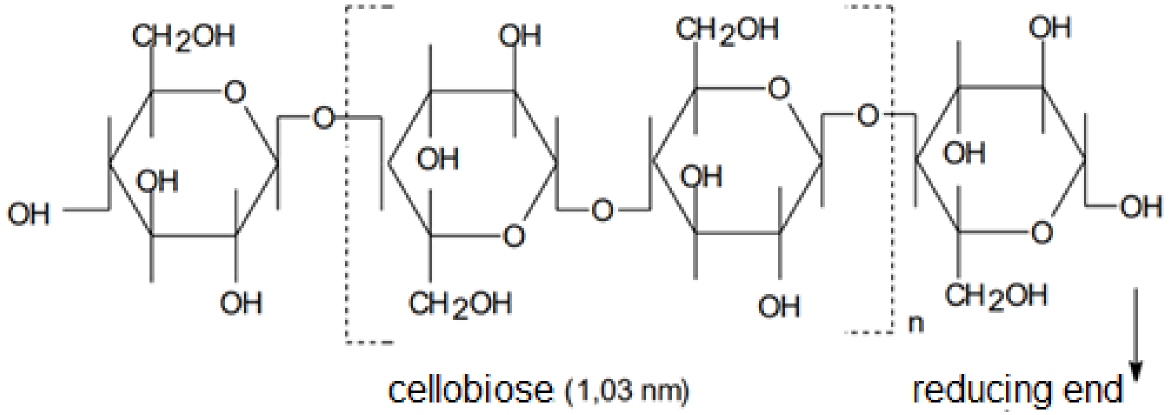
Constitution and configuration of cellulose

- cellulose composed of anhydroglucopyranose units
- linear polymer with a uniform structure
- the units are bound to each other by β-(1-4) glycosidic bonds



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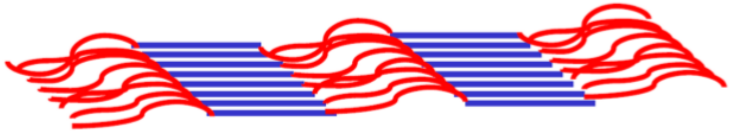
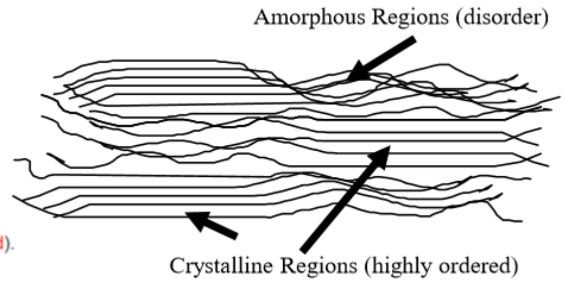
Crystalline and amorphous cellulose fraction

- crystalline (inaccessible) share - ordered
- amorphous (accessible) moiety - disordered
- the relative proportions depend on the origin and preparation
- native cellulose - about 70 % of the crystalline fraction



Cellulose consists of crystalline regions (blue) and disordered or amorphous regions (red).



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Hemicelluloses

- cellulose precursors
- extractives obtained by alkaline extraction of chemically pure cellulose consist of hemicelluloses and short-chain celluloses
- hemicelluloses defined as low molecular weight celluloses
- composed of various monosaccharides, have a shorter chain molecule and contain short side chains
- the monosaccharides forming hemicelluloses can be divided into groups such as pentoses, hexoses, hexouronic acids and deoxy-sugars
- the main chain may be formed by one sugar species, e.g. xylan, or by two or more sugar species, e.g. glucomannan

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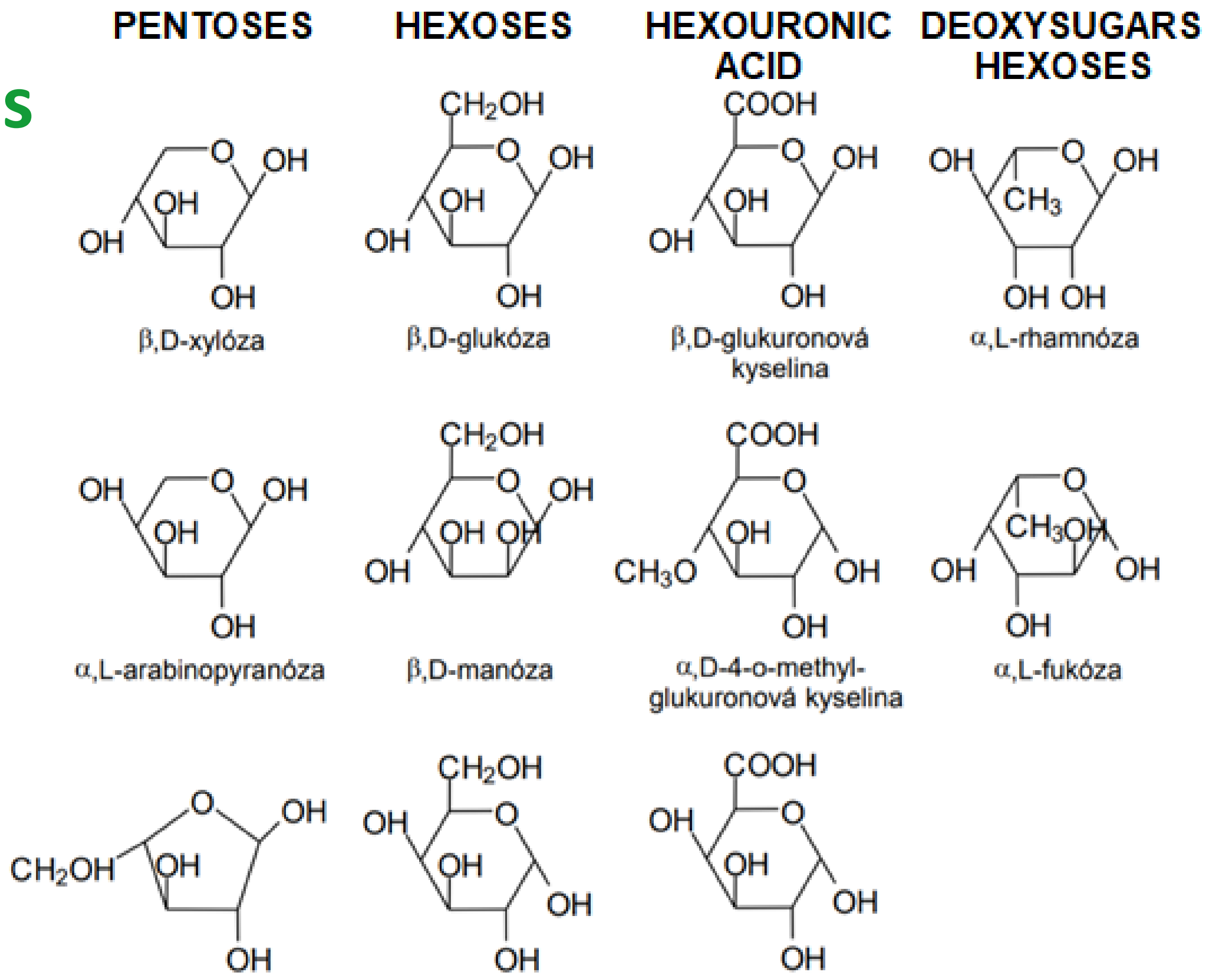
Hemicelluloses



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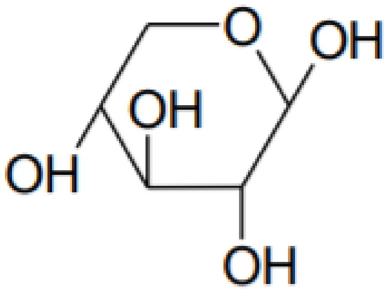
Xylans

- hemicelluloses usually consisting of a homopolymer chain based on xylose linked together by a β-(1-4)glycosidic bond



β,D-xylóza

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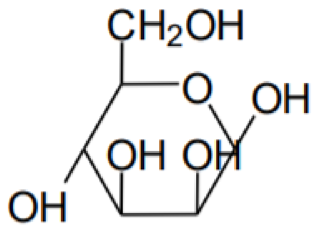
Manans

- the basic chain is heteropolymeric consisting of mannose and glucose
- the ratio of mannose to glucose is about 3:1
- galactose is bound to the base chain by a β-(1-6)glycosidic bond



β,D-manóza

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Glucans

- besides cellulose, other polysaccharides containing glucose units
- the most important is **starch** (fruits, seeds, etc.) found in parenchymatous wood cells
- starch is made up of various components differing in molecular weight and molecular structure
- linear **amylose** and branched **amylopectin**
- another wood glucan is **callose** - a component of the seed cells in the cotyledon, also found in parenchymatous cells

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Galactans

- the hemicelluloses of the galactan group have been known for a long time, especially the arabinogalactans isolated from larch (conifer)
- these hemicelluloses are water-soluble and can be isolated in quantities of 10-25%
- in general, galactans are highly branched
- arabinogalactan isolated from larch has a basic chain consisting of galactose linked by β-(1-3) glycosidic bonds

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Pectins

- pectins include galactouronates, galactans and arabanes
- galacturonans of various compositions are present in many plants, and are also found in fruit skins and gums
- these hemicelluloses are present in both deciduous and coniferous plants in quantities of less than 1%
- they are mainly found in the middle lamella

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Lignin

- another important polymeric organic substance in the plant world
- incorporated into plant cell walls
- increases mechanical strength properties
- the amount of lignin present in different plants is highly variable
- high lignin content is characteristic of the highest, lowest and inner parts of the trunk, conifer branches, bark and compression wood

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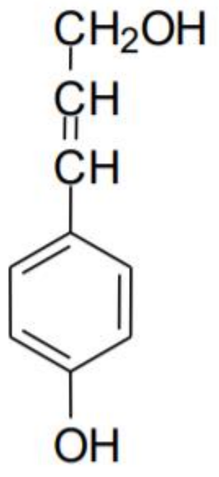
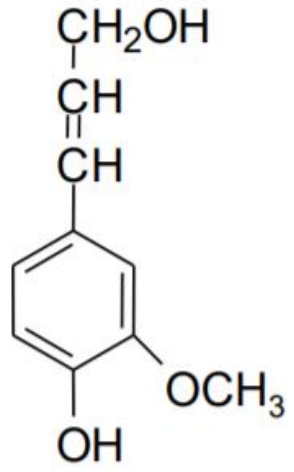
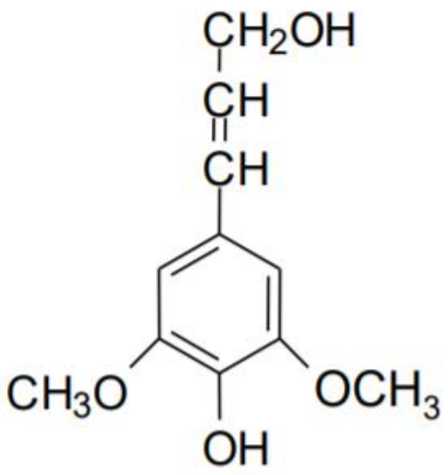
Lignin



kumarylalkohol (I)



koniferylalkohol (II)



sinapylalkohol (III)

Wood analysis – the main problems

- Very tight structure
- Chemical interconnection between cell wall macromolecules
- It is never possible to completely isolate the individual components

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Wood analysis - Preparation of holocellulose - delignification methods

- In the preparation of holocellulose, the following applies
  - Low residual lignin content
  - Minimum loss of polysaccharides
  - Minimal oxidative and hydrolytic degradation of cellulose
- Two general methods
  - Chlorination, including alternate extraction with a hot alcoholic solution of an organic base
  - Delignification with acidic sodium chlorite solution

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Wood analysis - Isolation and determination of cellulose

- Three main types of methods to obtain pure cellulose
  - Separation of the major portion of hemicelluloses and residual lignin from holocellulose
  - Direct isolation of cellulose from wood (including purification procedure)
  - Determination of the cellulose content of complete hydrolysis of wood, holocellulose or α-cellulose, followed by determination of the sugars produced

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Wood analysis - Differentiation of α, β, γ-cellulose - different solubility in alkalis

- α-cellulose - insoluble
- β-cellulose - soluble, precipitates after neutralisation
- γ-cellulose - soluble even after neutralisation

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Wood analysis - Isolation of hemicelluloses

- Different solubility in alkalis

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Wood analysis - Isolation and determination of lignin

- Its isolation in unchanged form and its precise determination impossible
- All isolation methods substantially alter the natural (native) structure of lignin or release only relatively unaltered portions of lignin
- Methods of lignin isolation
  - Methods obtaining lignin as a residue
  - Methods in which lignin is dissolved
    - without reaction, with the solvent used for extraction
    - to form soluble derivatives

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Analysis of cellulose - Metal complexes are often used to dissolve cellulose (determination of polymerization degree)

- Copper complexes - cuoxam, cuen
- Cobalt complexes - cooxen
- Cadmium complexes - cadoxene
- Sodium theatrate with iron complexes - FeTNa

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Analysis of cellulose - Cellulose dissolution process

- Degradation of fibres and fibrous structures
- Disintegration into single molecules of unchanged chain length
- Degradation of intermolecular bonds occurs with botting and with the ingress of chemical groups that break intermolecular bonds and solvate single molecules
- The arrangement of macromolecules depends on the concentration of the solution - swollen gel - highly diluted solution

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Analysis of lignin – Solubility

- With the exception of acid lignins, which are insoluble in all solvents, analytical lignins or their fractions may be soluble in methanol, ethanol, acetone or mixtures thereof
- Technical alkyl lignin and lignosulfonates are generally soluble in water, dilute alkalis, buffer and salt solutions, as well as in certain basic and neutral polar solvents

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Analysis of lignin - UV spectroscopy

- UV absorption is a well-known and used method for lignin identification
- Quantitative and qualitative determination as well as characterization of changes in lignin structure and properties
- The distinct absorption of lignin in the UV region is based on its aromatic character (sum of phenylpropane units)
- A typical lignin spectrum includes a maximum at 280 nm, followed by a decrease to lower wavelengths, with a more or less distinct slope in the 230 nm region

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Analysis of lignin - IR spectroscopy

- IR spectroscopy in the near-IR region (2.5-15 μm, 4000-600 cm^{-1}) is another physical method used to characterize lignin and its derivatives
- IR spectra of lignins show several major absorption bands that are assigned to structural groups
- The characteristic absorption bands of lignin are most commonly found in the region around 1510 and 1600cm^{-1} (C-H deformation and aromatic ring vibration)

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Wood morphology – cell wall

- Cell membrane - osmotic pressure inside the cell (MPa)
- Cell wall (10-500 nm)
    - Mechanical function
    - Composition
        - In the early stages of cell life, made of pentosan-type polysaccharides
        - cellulose
        - in wood cells, later also from lignin
- Fibrillar structure - chaotic arrangement of fibrils
- most important cell wall in terms of paperwork - much thicker

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Wood morphology – Cytoplasmic membrane

- 5-10 nm, gel-like bilayer
- Transport of substances - selective permeation
- Proteins and lipids - matrix arrangement
- By changing the spatial configuration change selectivity, pore size approx. 0.1 nm (molecule) filter for cell

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Wood morphology – Cytoplasm

- Gel - proteins, polysaccharides, fats, inorganic compounds, water-diffusely dispersed - not free
- Water in cytoplasm - hydrophilic envelope of macromolecules, hydrates salts present, bound to proteins present
- Cytoplasm thermolabile and sensitive to changes in pH and salt concentrations

Wood morphology - Mitochondria

- 500 × 1200 nm to 700 × 2000 nm
- Double layer of lipids and proteins with as large a surface area as possible
  - Transport and oxidation-reduction processes
  - Pigment content (chlorophyll) - amount depends on cell function

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Wood morphology - Core

- 700 to 3000 nm - contains a nucleus (3 nm)
- Separated by a cytoplasmic membrane with pores of 40-70 nm
- DNA - deoxyribonucleic acid
- RNA - ribonucleic acid
- Lipids ...
- Control of cellular processes and hereditary information, division ...

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Wood morphology – ribosomes, starch grains

- Ribosomes
  - Protein synthesis for cell growth
- Starch grains
  - Energy storage

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Wood morphology – Lysosomes, Vacuola

- Lysosomes
  - Polymerization and depolymerization of proteins and other substances
- Vacuola
  - Air bubble, in a wood cell all organs are pressed against the cell wall (lumen)

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Wood morphology – wood cells

- The information above applies to wood cells only at the point of wood cell formation, i.e. the cambial layer dividing mesh
- Significantly varies - wall thickness 7-25 μm (7,000-25,000 nm) due to the formation of S1 and S2 layers between the T and P layers
- Thinning of cell wall - transport of fluids tracheids × libriform cells
- Enlargement of the vacuole (lumen) - receding cytoplasm - in the nucleus of the stem, death of wood cells

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Wood morphology - mesh

- Units of cells with the same function in a plant
- Terminal meristem at the top of the plant
- Thin walled, semi-permeable, growth, respiration, cell division, tree growth to height
- Primary meristem separates and divides:
  - Dermatogema - later epidermis
  - Priblema - later cortical mesh
  - Preloma - later the phloem and the woody part (xylem)

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Wood morphology - mesh

- Bark
  - Protective function, outer cracked part called bark
- Bast
  - Fibrous, living mesh, inner side of the bark, transport of photosynthetic products to the cambial layer - formation of cells
- Pith
  - Mid-trunk from root to top, true pith rays - radially, distribution of photosynthesis products across the width of the tree

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Wood morphology - mesh

- Cambial layer
  - Layer between wood and bark, secondary growth of wood, cells are prism-shaped, vertically arranged, cells do not harden or thicken, cells divide radially and tangentially into:
    - Wood cells (secondary xylem)
    - Bark cells (secondary phloem)
  - False marrow ducts - do not go all the way to the marrow
  - Formation of leaf rings - periodicity

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Wood morphology - Functional division of cells

- Parenchymatous cells
  - Functional meshes, storage meshes, living protoplasm, starch
    - Shape - ovoid, brick-shaped, fine-walled, pores
    - Meshes - marrow, marrow rays, wood parenchyma, resin tubules

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Wood morphology - Functional division of cells

- Prosenchymatous cells
  - Building mesh, fluid transport, mechanical function
    - Tracheae i.e. blood vessels - connected to each other, transport of fluids
    - Tracheids i.e. blood vessels - elongated closed, fluid flows through pores, fluid transport and strength
    - Prosenchymatous cells for mechanical strength only
      - Sclerenchymatous cells - coniferous trees
      - Libriform cells - deciduous trees

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Wood morphology - Coniferous wood

- Parenchymatous cells
  - Root rays, resin canal walls, diffuse and terminal parenchyma
  - 5 % of mass
- Distinct annual rings
  - Different density of tracheids (spring and summer)

Wood morphology - Coniferous wood

- Tracheal rays thin
    - Unicellular
- Resinous ducts
    - Both horizontal (through the centre of the pith ducts) and vertical
    - Varying amount of resin canals (pine × spruce)
    - Channels full and empty

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Wood morphology - Deciduous wood

- Liquid transport - tracheae - width up to 0.5 mm
- Mechanical function - libriform cells very dense network
- More complex cell structure in deciduous than in coniferous trees
- Leaf rings of deciduous trees inconspicuous
- Greater number of parenchymatous cells (non-fibrous)
  - Diffuse parenchyma
  - Terminal parenchyma
  - Paratracheal parenchyma

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Wood morphology - Deciduous wood

- Physical and mechanical properties of hardwood
  - Bulk density higher than conifers
  - Most cells smaller lumen than conifer cells, libriform fibres thicker than tracheids
  - Libriform fibres - hardness of wood, 60% of volume
  - Heartwood rays - large number, poor splitting, good machinability
  - Paper with high opacity, high smoothness, suitable for chemical processing

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Experiments of students at FFWS

- Moisture content
- Ash and silicates
- Extractives
- Lignin
- Cellulose

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Moisture content

- Wood is made up of 90 to 97% of the main components such as lignin and the carbohydrate part (cellulose and hemicelluloses)
- In addition to these substances, wood also contains 'accessory substances' such as inorganic compounds and organic monomers and polymers

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Moisture content

- Determination of dry matter, %

$$x_{OD} = \frac{m_{OD}}{m_{AD}} \cdot 100$$

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Moisture content

- Determination of moisture content, %

$$x_{MC} = 100 - x_{OD}$$

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Ash and silicates

- Inorganic substances, such as mainly sodium salts, calcium salts, etc., are separated from the wood by mineralization or combustion - the formation of ash
- The amount of these substances depends on the age of the plant, the position of the wood in the trunk, the type of wood, the location where the plant grew and its health

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Ash, %

$$x_A = \frac{m_A}{m_{OD}} \cdot 100$$

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Silicates

- The inorganic content of wood tells us not only how much ash is in the plant
- Specifically, the inorganic fraction insoluble in hydrochloric acid tells us the amount of silicate in the plant, which is another companion component of wood

Silicates

- Relative to ash

$$x_{Si(A)} = \frac{m_{Si}}{m_A} . 100$$

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Silicates

- Relative to original sample

$$x_{Si} = \frac{m_{Si}}{m_{OD}}$$

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Extractives

- Extraction is performed to remove unwanted substances from the sample
- Extractables can be determined using non-polar solvents such as ether, petroleum ether, benzene or toluene
- These solvents are mainly used to remove fats, fatty acids and their esters, resins, resin acids or waxes and sterols
- In addition to non-polar solvents, water (polar solvent) is used, either hot or cold, into which mainly salts and carbohydrates are transferred
- Other polar solvents include, for example, ethanol, which removes tannins, glucosides or dyes from the sample
- Acetone is also a polar solvent that removes fatty and resinous acids and sterols from wood

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Extractives

$$x_e = \frac{\Delta m}{m_{OD}}$$

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Lignin

- Wood consists of main components and supporting components. Lignin is one of the main components of wood, 15-35%
- Its distribution in the cell wall or in the different parts of the tree is not uniform
- Lignin is a characteristic chemical and morphological component of the tissues of higher plants, where it is predominantly found in the conductive laminae, which provide both fluid transport and strength properties

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Lignin

- In terms of chemical composition and structure, it is a highly irregular, randomly cross-linked three-dimensional polymer composed of phenylpropane units linked by two types of bonds, i.e. carbon-carbon (C-C) and ether-type (C-O-C), and containing two basic types of functional groups, i.e. hydroxyl (-OH) and methoxyl (-OCH_3)

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Lignin

- The isolation of lignin in its unaltered form and without the destruction of the macromolecule itself is not yet known
- Therefore, delignification procedures are used
- Delignification is an irreversible process whose final product is lignin. It is most commonly determined in the laboratory by the Klason method, the so-called Klason lignin

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Lignin

$$x_{KL} = \frac{\Delta m}{m_{OD}}$$

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Cellulose

- Wood consists of main components and supporting components. Cellulose is one of the main components of wood, which is almost half of the wood, 35-50%
- Of course, it depends on the type of wood (conifers generally contain more cellulose than deciduous trees), its age, location, etc.
- In terms of chemical composition and structure, cellulose is a natural macromolecular substance composed of β-D-glucopyranose units that are linearly linked at the 1,4 position by a β-D-glycosidic bond
- This linkage allows linear chain extension

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Cellulose

- Although cellulose is a chemically stable compound, it is subject to various types of degradation, such as exposure to acids or alkalis, elevated temperature, mechanical action or radiation
- The isolation of cellulose is highly dependent on the association of substances in the cell wall
- Chemical compounds such as fats, waxes, proteins and pectins can be easily removed by extraction with organic solvents and dilute alkalis
- In our case, a sample after acetone extraction is used for analysis
- Cellulose in wood is bound to hemicelluloses through various polysaccharide bonds and to lignin through hemicelluloses
- Their separation requires intensive chemical action

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Cellulose

$$x_{SC} = \frac{\Delta m}{m_{OD}}$$

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