



## Minireview

# Polysaccharides and lignin from oak wood used in cooperage: Composition, interest, assays: A review



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## ABSTRACT

It is widely accepted that alcoholic beverage quality depends on their ageing in premium quality oak wood. From the choice of wood to beverage ageing, through the different steps in cask manufacturing, many factors should be considered. One of the biggest challenge in cooperages is to take into account all these factors. Most of the studies are interested in phenolic compounds, extracted during ageing and especially involved in wine oxidation, colour, and sensory properties such as astringency and bitterness. Oak aroma volatile compounds have also been the subject of numerous studies. These compounds of interest are part of low molecular weight compounds which represent 2%–10% of oak wood composition. However, three polymers constitute the main part of oak wood: cellulose, hemicellulose and lignin. As far as we are aware, few studies concerning the role of these major macromolecules in oak wood have been published previously. This article reviews oak wood polysaccharides and lignin, their potential interest and different assays used to determine their content.

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## 1. Introduction

Oak wood presents a special interest in the Oenology field, considering that wood barrels have been used in winemaking since the pre-Christian time. Firstly intended for fermentation, storage, and wine shipping, winemakers realized over time that wine storage in barrels improved wine organoleptic quality, leading them to realize the importance of barrel making process. To fabricate barrels, the wood needs to have certain required properties. It should be either straight-grained or even strong, flexible enough to be bend and without defects that may cause leaks. It is preferable at the same time to be free of undesirable flavours. Anatomical features may be correlated with chemical properties that could affect wine or spirits maturation. Different types of woods, such as acacia, chestnut and oak wood, have been tested. Among them, oak wood appeared to be ideal for beverage ageing.<sup>1</sup>

Moreover, to offer all the qualities required, in the course of barrel production, oak used for barrels must pass through some important processing stages including seasoning and toasting.<sup>2</sup> During toasting process, chemical changes occur extensively and chemical bonds between polymers, in particular hemicellulose and lignin,<sup>3</sup> are degraded. Several studies have correlated the influence of wood extractives, primarily ellagitannins,<sup>4,5</sup> and some aromatic precursors<sup>6</sup>

with wine quality. However, to date, the contribution of polysaccharides, lignin and some oak wood oligosaccharides on wine organoleptic properties has not been highlighted. Particularly in woody plants, these polymers represent the major compounds in cell walls forming an insoluble network,<sup>7</sup> which produce wood. This natural tissue is composed mainly of cellulose, hemicellulose, and lignin, forming together a composite material of rigid cellulose fibres embedded in a cross-linked matrix of lignin and hemicelluloses that bind the fibres.<sup>8–10</sup>

Table 1 shows some results obtained from different authors on quantitative analysis of oak wood macromolecule composition.<sup>11–17</sup> According to this table, cellulose, hemicellulose and lignin proportions are 41%, 26.35% and 25.71% respectively.

Polysaccharides are widespread in plant kingdom, and account for around 66% of all global bound carbon.<sup>18</sup> They are polymeric carbohydrate molecules composed of long chains of monosaccharide units bound together by glycosidic linkages, their hydrolysis liberates the monosaccharide or oligosaccharide constituent. Their structure diversity ranges from linear to highly branched oligomeric chain. Not all the functions of individual polysaccharides are known, but they may act as storage material, structural components and protective substances. Starch, glycogen, cellulose, fructans, some  $\beta$ -glucans, and some galactomannans are examples of storage polysaccharides. Structural polysaccharides are either fibrous polysaccharides—mainly cellulose in higher plants and some algae or chitin in yeast and fungi—or matrix polysaccharides, for example arabinoxylans, galactomannans or pectins in plants. Protective polysaccharides include extracellular polysaccharides from

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**Table 1**  
Oak wood chemical composition (% of dry weight)

Species	% Cellulose	% Hemicellulose	% Lignin	% Extractables	Benzene-ethanol	Hot water	Ashes
Aubier	39.9	27.6	24.9	—	2.4	—	—
Duramen	37.6	28.6	24.5	—	4.4	—	0.3
European oak	38	29	25	4.4	—	—	0.3
French oak	22–50	17–30	17–30	2–10	—	—	0.2
Quercus sp.	40.5	23.3	22.2	—	—	—	—
Quercus robur	41.1	22.2	29.6	3.8–6.1	0.4	12.2	0.3
Quercus robur	46.37 ± 0.46	18.89 ± 0.24	29.12	5.53 ± 0.26	—	—	0.52 ± 0.01
Quercus alba	42	28	25	5.3	—	—	—
Quercus prinus	41	30	22	6.6	—	—	0.4
Quercus stellata	38	30	26	5.8	—	—	0.5
Quercus rubra	45.7	24.8	27.6	—	—	—	0.41
Quercus lyrata	42.4	24.8	27.6	—	—	—	0.41

microorganisms or exudate gums from plants.<sup>19</sup> Cellulose, the main polysaccharide in wood, has long been studied to elucidate its structure and function in plants. The structure and role of hemicelluloses and pectins, which also represent a large proportion of polysaccharides in wood and pulp fibres, have been widely studied in the 1970s and these investigations are still under progress. In addition, minor polysaccharide components play also an important role for the enrichment of acidic polysaccharides in the circulating water of paper machines and polysaccharide components that retain lignin in chemical pulp fibres during cooking and bleaching.

It is therefore essential to learn more about polysaccharides from wood origin role and behaviour during oak barrel manufacturing in order to control these processes and optimize beverage quality aged in contact with oak wood.

## 2. Polymers' discovery

The 19th century was particularly important for wood polymer understanding and describing. In 1838, the French chemist, Anselme Payen was the first to discover the most abundant molecule on earth: the cellulose.<sup>20</sup> He isolated it from plant matter and determined its chemical formula. He also noted that the carbon content of wood and, consequently, that of the removed material was higher than the cellulose which had the same composition as starch. He defined the carbon-rich substance as an “encrusting material” which embedded the cellulose in the wood. Later, Schulze (1865)<sup>21</sup> introduced the term “lignin” and Erdtmann observed that catechol and protocatechuic acid were formed on alkali fusion of wood; he concluded that the non-cellulosic constituent was aromatic. Tiemann and Mendelsohn<sup>22</sup> suggested the possibility that coniferin, a glucoside of coniferyl alcohol, might be a transformation product of the “aromatic atom complex” present in wood. In 1890, Benedikt and Bamberger demonstrated that methoxyl groups were present in woody tissues but were lacking in pure cellulose.<sup>23</sup> The development of new pulping processes, especially the sulfite process, strongly stimulated interest in the reactions involved and in connection with his studies on the composition of the lignin sulfonates, Klason advanced the idea that lignin was chemically related to coniferyl alcohol.<sup>24</sup> He supported this view by observing that the heat of coniferyl alcohol with acidic bisulfite solutions produced a sulfonic acid which he believed was similar to lignin sulfonate. Remarkably enough, as early as 1907 Klason considered lignin to be a high-molecular substance and ten years later he proposed that the coniferyl alcohol units might be linked together by a continuous condensation between the alcoholic and phenolic hydroxyl groups.<sup>23</sup> He also assumed that part of the lignin was made up of coniferaldehyde units or of a hydroxyconiferyl alcohol rather than coniferyl alcohol. Although Klason was unable to produce solid experimental evidence for his structural views, the basic coniferyl

alcohol idea has undoubtedly greatly influenced the later lignin chemists. It needs to be emphasized that the lignin artistic representations of macromolecular configuration have changed enormously over five decades. Originally it was being envisaged as a complex three-dimensional (Bakelite like) phenolic polymer<sup>25,26</sup> afterwards as amenable for a computer-programmable simulation,<sup>27,28</sup> and more recently it was considered as linear macromolecular entities.<sup>29</sup> In parallel other researches occurred; crude fibre analysis (involving a mixture of high molecular weight polymers) was developed in Weende agricultural research station in German by Henneberg and Stohmann (1859). They found that this method had already shown its limits. This analysis did not quantify only cellulose but also fibrous residue. Few years later, cellulose was used to produce the first successful thermoplastic polymer, celluloid, by Hyatt Manufacturing Company in 1870. Hermann Staudinger determined the polymeric structure of cellulose in 1920.<sup>30</sup> The compound was first chemically synthesized (without the use of any biologically-derived enzymes)<sup>31</sup> in 1992. After 100 years of the crude fibre analysis first development, a group of researchers has developed in the USA a new and efficient method that can distinguish between two cellulosic fractions:<sup>32</sup> the walls (Neutral Detergent Fiber or NDF) and the lignocellulose (Acid detergent Fibre or ADF).

Weende or Van Soest methods for determination are similar. Francou, in 2003, observed the same type of relationship.<sup>33</sup> These methods are described in literature as “forage fiber techniques”, in 1989 Ryan et al.<sup>34</sup> made a comparison between forage fibre techniques inspired by Goering and van Soest and “forest products techniques” related with a series of digestions using the procedures outlined in Fig. 1.

They highlighted that both methods appeared to be closely related and the forage fibre techniques estimated remarkably well the proximate carbon fractions of forest litter. However, this study was conducted on birch, alder, aspen, fir, fresh red pine needles, dogwood leaves, white oak leaves, red maple leaves, but not on oak, a wood characterized by its very high density. That may let suggest that forage techniques are not adapted methods in the case of oak wood samples. This could be linked with the unique oak wood anatomical properties that make it more appropriate for barrel fabrication: essentially its sustainability.

As outlined above, methods to analyze polymers evolved as researches and discoveries progressed. Wood polymer fraction (including, cellulose, hemicellulose and lignin) is considered as insoluble residue after a full extraction of other wood constituent using chelating agent and/or mineral bases and are often called CWR (cell wall residue). This is well illustrated in Fig. 2 which represents general schema for wood constituent separation according to Baeza and Freer.<sup>35</sup> Table 2 summarizes the most commonly referenced methods with their principles.<sup>17,32,36–40</sup> The most recent analysis methods are mainly based on carbohydrate decomposition.

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