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A brief review of the structure and extraction methods of lignin

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ABSTRACT

One of the three basic components of lignocellulosic biomass (LB), lignin is the second most plenty of natural polymer on the planet after cellulose. It has the power to become the aromatic raw material of the future for industry. However, this potential has not been fully realized due to its structure has not yet been fully elucidated and its largely underexploited industrial availability. Lignin extraction methods are divided into two main processes are sulfur-containing and non-sulfur-containing. Sulfur processes contain sulfite and kraft (alkaline) pulping methods; the main types of lignin obtained by these methods are kraft lignin and lignosulfonates. Non-sulfur processes contain solvent-based pulping (organosolv) and soda pulping methods; the main types of lignin obtained by these methods are organosolv lignin and soda lignin. In addition to these methods, there are also other chemical-based delignification processes, such as acid hydrolysis from bio-ethanol production and vapor spraying methods with the application of high-temperature and high-pressure steam. In this context, understanding the structure and extraction of lignin is important to fully exploit its potential. This review provides an overview of the structure, chemical properties, and extraction methods of lignin.

1. Introduction

Today, due to the depletion of fossil fuel reserves and increasingly stringent environmental regulations, the production and use of renewable and sustainable materials has gained global importance. This has led to an increased interest in the development of bio-based products and technologies [1]. As environmental awareness increases and raw material resources become scarce, research into the utilization of alternative sources of industrial raw materials has become increasingly important.

Due to renewable resources for bio-based materials and chemicals, lignocellulosic biomass has attracted more attention and is composed of cellulose (30–50 %), hemicellulose (15–35 %), and lignin (10–20 %) [2,3]. The efficient usage of these biomass components is of importance for the sustainable use of renewable resources [4]. Lignin is an amorphous and complex aromatic polymer composed of randomly cross-linked phenylpropanoid units. These units are coniferyl alcohol, sinapyl alcohol and *p*-coumaryl alcohol. [5]. Lignin content can vary between 18 % and 33 % in softwoods, 15–30 % in hardwoods and 5–30 % in herbaceous plants [6]. The total amount of lignin in the biosphere is increasing by about 7 % per year and now exceeds 300 billion tons [7].

Lignin has great potential as an alternative raw material to petroleum-based products and phenolic resins. Despite its promising potential as a renewable source for the production of bio-based materials, fuels, and value-added chemicals, lignin is currently largely underutilized compared to cellulose. In the pulp and paper industry, large quantities of lignin are generated as waste. Although global lignin production is estimated to be around 70 million tons per year, less than 2 % is used in value-added products, and this amount is projected to reach 225 million tons annually by 2030 [8]. At present, the majority of lignin is utilized as an energy source in paper mills, which constitutes a low-value application [9].

Due to the variation in lignin's chemical structure depending on the type of lignocellulosic feedstock and its association with hemicelluloses via physical and covalent bonds within the plant cell wall, its structure has not yet been fully elucidated. As a result, its full potential has yet to be realized. The major disadvantage of lignin assessment is its complex chemical structure and variability [10]. Currently, extensive efforts are underway to valorize lignin [11–15].

The purpose of this review is to present an overview of the structure, chemical properties, and extraction process of lignin.

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2. Discovery, structure and chemical properties of lignin

In 1838, Anselme Payen discovered cellulose and demonstrated that it is commonly found in all plants, particularly in their cell walls. Payen is also recognized in the literature as the first researcher to isolate cellulose. Additionally, while studying the chemical composition of wood, he attempted to separate it into its components using nitric acid and alkaline solutions, during which he succeeded in isolating lignin. He referred to the compound he obtained as "incrustant." In 1857, the German scientist Schulze identified this compound as lignin. Lignin was generally defined as a natural polymeric substance formed by the enzymatically initiated dehydrogenative polymerization of three primary phenylpropanoid precursors: *trans*-coniferyl, *trans*-sinapyl, and *trans-p*-coumaryl alcohols.

Wood, a natural material, as well as annual plants, is composed of cells. The cells that make up softwood, hardwood and annual plants are morphologically different from each other. Despite these morphological differences, these cells fundamentally contain three primary macromolecular components: cellulose, hemicellulose, and lignin [16]. For this reason, they are generally referred to as lignocellulosic biomaterials. In general, cellulose forms the structural framework of the cell wall, while hemicellulose and lignin constitute the matrix that surrounds and fills the spaces within this framework. Lignin is a complex aromatic polymer that is one of the major components of the cell wall in all vascular plants, including hardwood and softwood species [17]. This component provides trees with mechanical strength and flexibility [18,19].

The structure of biomass is shown in Fig. 1.

Another important property of lignin is its function as a natural adhesive that permanently bonds cells together in woody stems. Thanks to this structural characteristic, stems become mechanically reinforced and more resistant to external factors, particularly impacts.

Table 1 provides an overview of the average cellulose, hemicellulose, and lignin content in hardwoods, softwoods, bast fibers, and seed fiber materials.

The chemical structure of lignin is not fully understood due to its complex nature resulting from the different bonding patterns of its monomers, as well as its variability depending on its source and species.

Fig. 2 shows the schematic representation for the macromolecular structure of lignin.

All lignins, regardless of their source, are polymerization products of coumaryl alcohol, coniferyl alcohol and sinapyl alcohol. Fig. 3 shows the structures of the monolignol precursors of lignin.

Table 1
Average cellulose, hemicellulose, and lignin composition of various lignocellulosic materials.

Type	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Softwood	41–44	26–33	26–31
Hardwood	42–51	23–38	19–24
Bast Fiber	75.9–84	15.7–20.7	0.3–3.4
Seed Fiber	46.7–48.7	0.2–0.3	51–53

The main strong bonds in the structure of lignin are biphenyl (C-C) bonds between aromatic rings, alkyl-aryl (C-C) bonds between aliphatic and aromatic carbons, and ether bonds that show high resistance to hydrolysis. In contrast, α-aryl ether bonds are sensitive to hydrolysis and are relatively weaker compared to other bonds in the lignin structure. It is evident that the β-O-4-C bond occurs more frequently compared to other bonds.

Softwood lignin includes predominantly of coniferyl alcohol groups linked to aromatic rings, hardwood lignin includes of both coniferyl and sinapyl alcohol groups linked to aromatic rings, grass lignin is stored in grass plants and consists of coniferyl, sinapyl and *p*-coumaryl alcohol groups linked to aromatic rings. These three aromatic alcohol groups are called monolignols [23]. The phenolic natural forms of monolignols in the lignin structure are *p*-hydroxyphenyl (H), guaiacyl (G) and syringyl (S) groups. [24].

3. Extraction techniques of lignin

The isolation of lignin from lignocellulosic feedstocks can generally be divided into two main categories. In the first method, the polysaccharide components of the lignocellulosic structure (cellulose and hemicellulose) are removed through hydrolysis or dissolution processes, resulting in lignin being obtained as a residue. In the second method, commonly used in pulp and bioethanol production, delignification processes solubilize lignin, thereby separating it from cellulose [25].

In general, lignin extraction methods are divided into two main processes: sulfur-containing and non-sulfur-containing. Sulfur processes contain sulfite and kraft (alkaline) pulping methods; the main types of lignin obtained by these methods are kraft lignin and lignosulfonates. Non-sulfur processes contain solvent-based pulping (organosolv) and soda pulping methods; the main types of lignin obtained by these methods are organosolv lignin and soda lignin. In addition to these

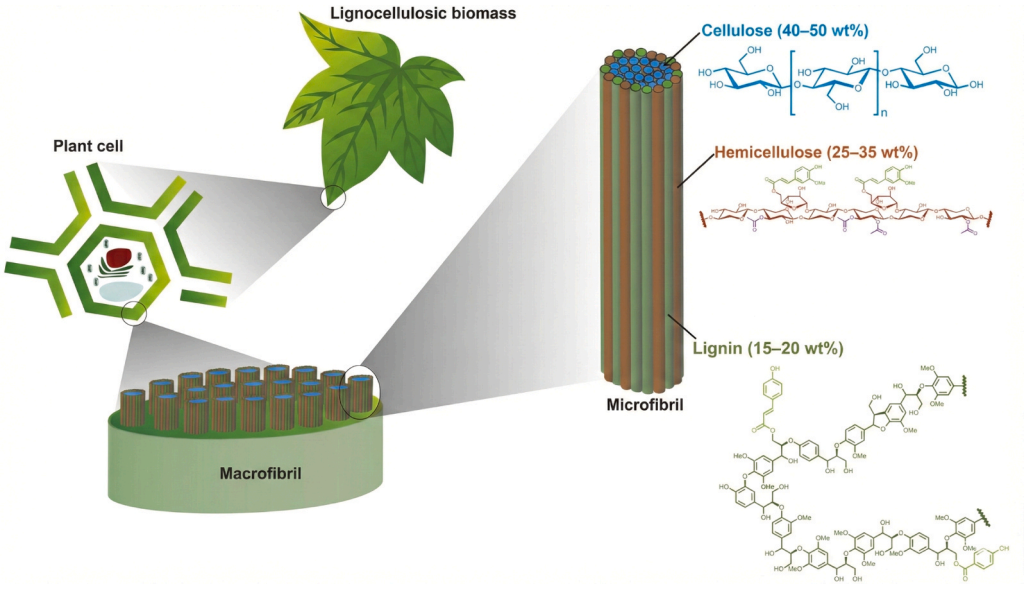

Fig. 1. The structure of lignocellulose biomass [20].

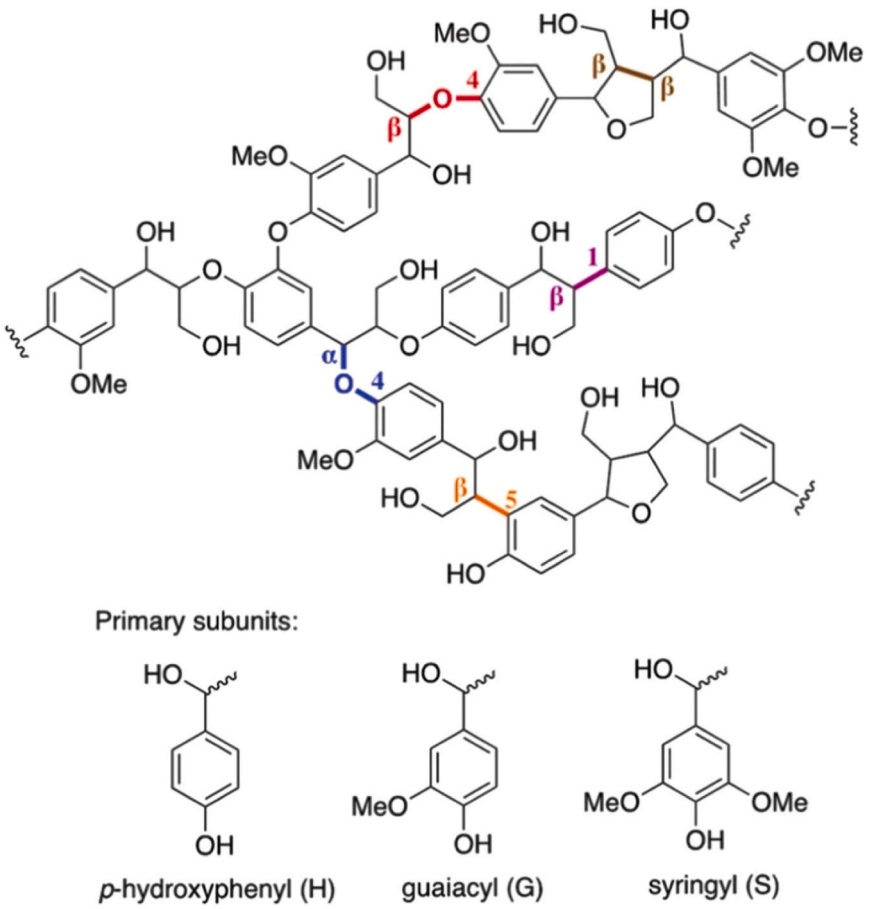

Fig. 2. Schematic representation of the macromolecular structure of lignin. Major monolignol units are color coded as follows: sinapyl alcohol (red), guaiacyl alcohol (blue), and *p*-coumaryl alcohol (green). [21].

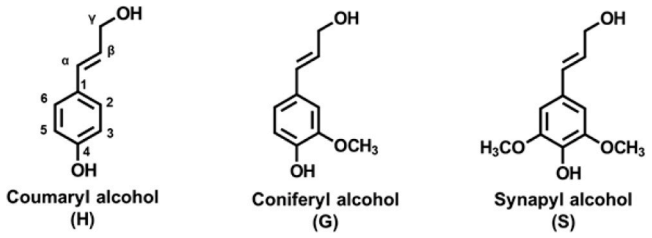

Fig. 3. Structures of the monolignol precursors of lignin. [22].

methods, there are also other chemical-based delignification processes, such as acid hydrolysis from bio-ethanol production and vapor spraying methods with the implementation of high-temperature and high-pressure steam. [26].

Common lignin extraction techniques are shown in Fig. 4.

The properties of different lignins with different properties and reactivity depending on the pulping method are presented in Fig. 5.

3.1. Sulfur-containing process

3.1.1. Kraft process

The kraft process is the most commonly used pulp production technology worldwide, accounting for approximately 80 % of chemically produced fibers today, with an annual production capacity reaching around 500 million tons [28,29].

In the kraft process, lignocellulosic raw material is processed in digesters at 170 °C in a strong alkaline solution containing sodium sulfide (Na_2S) and sodium hydroxide (NaOH), commonly referred to as white liquor [30]. Under these conditions, especially the α-aryl ether (α-O-4) and β-aryl ether (β-O-4) bonds in the lignin structure are broken; the resulting reactive groups (HO^- and HS^-) facilitate the depolymerization and solubilization of lignin molecules [31]. Approximately 350–500 kg of lignin is present in the black liquor for every ton of pulp produced via the these process. The intensive cleavage of β-O-4 bonds is a significant characteristic that distinguishes kraft lignin from other types of lignin. These lignins contain higher amounts of phenolic hydroxyl groups and have lower proportions of biphenyl and other condensation products compared to soda lignin. The black liquor generated after cooking contains ash content that can reach up to 30 % [27]. However, since other components such as inorganic compounds and sugars remain soluble over a wide pH range, lignin can be recovered by precipitation through acidification. This process results in kraft lignin as a product of high purity with low ash and carbohydrate content [32]. The main reasons for its widespread use include the ability to process of lignocelluloses, enabling the manufacturing of high-strength paper, efficient chemical recovery, and high energy efficiency.

3.1.2. Sulfite process

The sulfite pulping method is primarily based on the sulfite process, which involves treatment with sulfur dioxide [33]. These process is performed at temperatures between 125 °C and 150 °C with reactions lasting 3–7 h [27]. Due to the presence of sulfurous acid (H_2SO_3) in the sulfite solution, only certain wood species can be effectively processed using this method. When the same raw material is used, the sulfite process generally provides a higher pulp yield compared to the kraft method. In addition, the pulp obtained is naturally lighter in color and can achieve a high level of brightness without the need for extensive

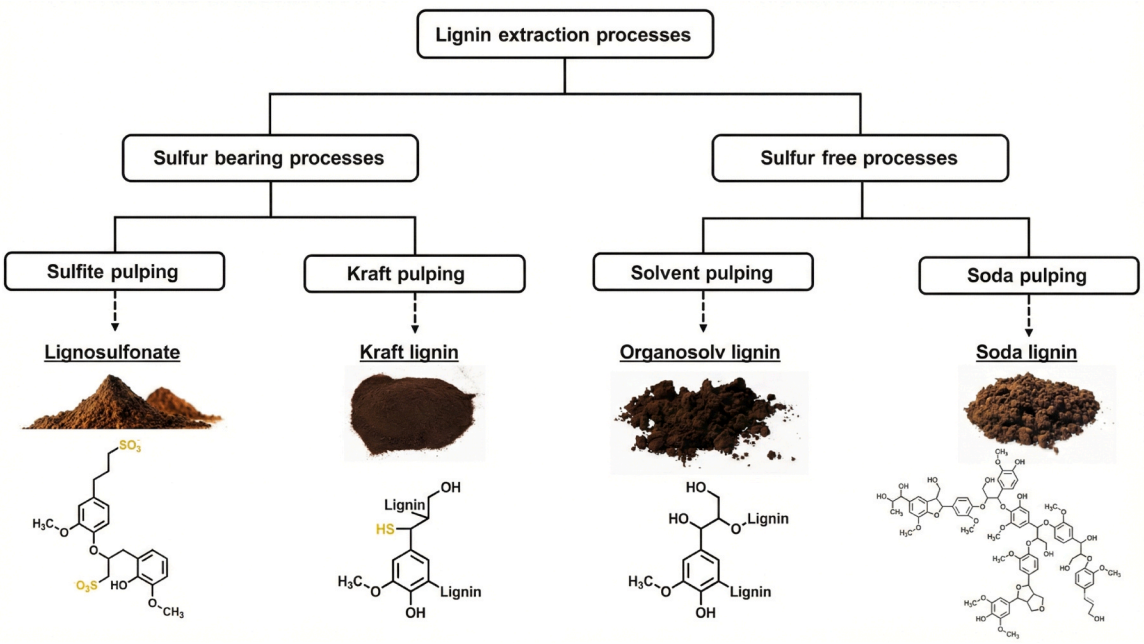

Fig. 4. Common lignin types, lignin extraction techniques and representative structures [22].

Lignin type	Sulfur-lignins		Sulfur-free lignins	
	Kraft	Lignosulfonate	Soda	Organosolv
Aspect				
Raw materials	Softwood Hardwood	Softwood Hardwood	Annual plants	Softwood Hardwood Annual plants
Solubility	Alkali Organic solvents	Water	Alkali	Wide range of organic solvents
Number-average molar mass (M_n – g mol^{-1})	1000–3000	15,000–50,000	800–3000	500–5000
Polydispersity	2.5–3.5	6–8	2.5–3.5	1.5–2.5
T_g (°C)	140–150	130	140	90–110

Fig. 5. The properties of different lignins [27].

bleaching. The pulp produced by this method can be easily bleached with mild bleaching agents to achieve high brightness levels.

However, there are also several disadvantages to this method. The cooking time is longer compared to the kraft process, the mechanical resistance of the resulting fibers is lower, and recovery of the chemicals from the spent liquor is relatively difficult. The solution obtained at the end of the cooking process, referred to as brown liquor, is rich in lignin content. This lignin is precipitated on an industrial scale using the howard process [34]. The lignin isolated through this method is structurally sulfonated and is therefore referred to as lignosulfonate lignin. Their high water solubility is the most important reason for their wide range of applications. [35,36].

3.2. *Non-sulfur-containing process*

3.2.1. *Soda process*

The soda pulping method is a chemical pretreatment process commonly used in the production of paper from non-wood

lignocellulosic raw materials. Unlike the kraft process, this method uses only sodium hydroxide (NaOH). The lignocellulosic material is treated with sodium hydroxide (NaOH) solution (13–16 %) at between 140 and 170 °C, resulting in the extraction of lignin [37,38].

During the soda pulping process, various delignification reactions occur that contribute to the structural breakdown of lignin molecules. Although these reactions vary in importance and impact, the most critical mechanism is the cleavage of non-phenolic β-O-4 aryl ether bonds under alkaline conditions. The breaking of these bonds occurs through the nucleophilic action of NaOH, leading to the formation of epoxide intermediates and phenolic end groups. This, in turn, makes the lignin more reactive and susceptible to further transformations [37]. In addition, alkaline conditions during the cooking process can cause hydrolysis and peeling reactions in the carbohydrate fractions, potentially affecting the structural integrity of cellulose and hemicellulose. Due to the absence of sulfur, the soda pulping method is considered more environmentally friendly. It is particularly preferred in applications where the prevention of sulfur gas emissions and the production of sulfur-free lignin are desired.

3.2.2. Organosolv process

Delignification processes used in pulp production and bioethanol generation have gained importance with the development of alternative methods aimed at reducing environmental impact. In this context, key criteria include the environmental friendliness of the method, its economic feasibility, its potential to yield high-quality products, and its effectiveness even in small-scale production systems.

Organosolv pulping methods were developed as environmentally friendly alternatives to the kraft and sulfite processes in response to these needs. The principle of this method is the hydrothermal treatment of lignocellulosic biomass using a mixture of additives such as water, organic solvents and acids to obtain high-quality and sulfur-free lignin [39]. The organosolv process is carried out in the temperature range 130–200 °C. In this method, organic solvents like ethanol, methanol, acetone or their mixtures are combined with inorganic chemicals or water in specific proportions to form solutions used for delignification of lignocellulosic raw materials [40,41]. Lignins isolated via organosolv methods are named according to the solvent used in the process. For example, varieties such as alcohol lignin, acidified dioxane lignin, acetic acid lignin, thioglycolic acid lignin, phenol lignin, and hydrotropic lignin are classified based on the chemical structure of the main solvent. Because lignin dissolves in the solvent and can then be precipitated using relatively simple chemical procedures, complex purification steps are generally not required for lignin isolation in this method. Organosolv lignin is more hydrophobic than kraft lignin and lignosulfonates.

3.3. Other chemical-based delignification processes

3.3.1. Steam explosion method

The steam explosion method works on the principle of breaking chemical bonds between lignocellulose (cellulose, hemicellulose and lignin) by applying high temperature ans pressure steam followed by rapid pressure relief [42]. This pretreatment technique involves subjecting lignocellulose to high-pressure steam at temperatures between 180 and 250 °C, typically in the presence of an acidic catalyst. During the process, hemicellulose and cellulose are hydrolyzed into monomeric sugars, while the cleavage of aryl-ether and carbon–carbon bonds in lignin leads to secondary reactions such as demethoxylation, alkylation, and condensation [43–45]. Following steam explosion, enzymatic hydrolysis is usually applied to further break down the cellulosic fraction. The resulting lignin fraction exhibits characteristics similar to organosolv lignin due to properties such as low molecular weight and high solubility in organic solvents, and this method is compatible with green chemistry [46,47].

3.3.2. Acid hydrolysis process

Lignin can be obtained as a residual fraction remaining after the dilute acid hydrolysis process applied prior to bioethanol production from lignocellulosic feedstocks [48]. In this method, polysaccharide hydrolysis is performed using dilute or concentrated acids like sulfuric acid, nitric acid, hydrochloric acid, or phosphoric acid. The lignocellulosic material is typically treated with a dilute acid solution at temperatures ranging from 165 to 195 °C for a duration of 3–12 min [49,50]. Following this pretreatment, a second separation step is performed through enzymatic hydrolysis to further break down cellulose and hemicellulose more effectively. Lignin obtained via the dilute acid hydrolysis method exhibits high solubility characteristics and offers advantages in terms of cost-effectiveness.

3.4. Properties of lignins according to extraction method

The chemical structure, functional groups, molecular weight, purity level, and reactivity of lignin vary substantially depending on the extraction method employed. Lignin obtained through the kraft process undergoes extensive cleavage of β-O-4 linkages, resulting in a high phenolic hydroxyl content, increased reactivity, and a structure that is markedly altered from native lignin; additionally, its low carbohydrate and ash contents contribute to relatively high purity [31]. Lignosulfonates produced via the sulfite process are strongly sulfonated, highly water-soluble, and commonly used as dispersants and plasticizers; however, they are among the most chemically modified lignin types and differ considerably from native lignin [36]. Soda lignin also extracted under alkaline conditions but without sulfur-containing chemicals offers a more environmentally friendly profile; its degree of condensation tends to be higher and its reactivity slightly lower than that of kraft lignin [37]. Organosolv lignin, isolated under solvent-based and comparatively mild conditions, retains a structure closest to native lignin and exhibits high purity with minimal ash and carbohydrate content [41]. Lignins derived from acid hydrolysis or steam explosion methods generally display lower molecular weights, partially condensed structures, and variable solubility, reflecting the harsher chemical or thermo-mechanical conditions of these treatments [46,48]. Overall, the diversity of extraction methods leads to pronounced differences in reactivity, solubility, thermal stability, and potential for chemical functionalization, making each lignin type suitable for distinct value-added applications.

Table 2 provides a comparative overview of selected studies in the literature, highlighting key characteristics of lignins obtained through different extraction methods.

4. Conclusion

With significant potential for producing bio-based chemicals, biofuels, and biocomposite additives, lignin is the aromatic raw material of the future for industry. In this context, understanding lignin's structure, chemical properties, and extraction processes is essential to fully harness its potential.

The features of lignin are affected by variables like the type of lignocellulose used, the cooking method applied, and temperature, pH, pressure process parameters. Depending on the type of lignocellulosic materials, the structural compounds it contains vary, and this directly affects the properties of lignin. Cooking method is another important variable on lignin properties. Many properties of lignin change due to the chemical reactions that take place depending on the cooking conditions. Most of the available isolated lignins are derived from kraft and sulfite processes. Lignins obtained by alkaline cooking methods (kraft and soda) generally have a more stable structure and contain lower levels of impurities compared to lignins obtained by acidic cooking processes. Lignins obtained through the organosolv process (a sulfur-free method) are structurally closer to natural lignin and present higher purity. In contrast, lignin isolated by the sulfite cooking method

Table 2
Comparative overview of selected studies in the literature.

Lignin type	Key characteristics	Advantages	Disadvantages	Reference
Kraft lignin	- High phenolic –OH content - Extensive β–O–4 bond cleavage - Low ash and carbohydrate content - Strongly condensed structure compared to native lignin	- High reactivity - Widely available and low-cost industrial source	- Highly condensed and chemically modified structure - Less similar to native lignin - Tends to char under heat	[31]
Lignosulfonate	- Strongly sulfonated structure - Very high water solubility - Low hydrophobicity - Chemically modified lignin backbone	- Excellent dispersant and binder properties - Broad industrial use (concrete additives, dust suppressants, etc.)	- Low thermal stability due to sulfonate groups - Highly modified structure - Limited compatibility with hydrophobic polymer systems	[36]
Soda lignin	- Sulfur-free - Alkaline β–O–4 cleavage - Condensation degree varies with process severity	- Environmentally friendly sulfur-free lignin source - Often exhibits higher purity than kraft lignin	- Typically lower reactivity than kraft lignin (but process-dependent) - Less available at large industrial scale	[37]
Organosolv lignin	- Very low ash (<1 %) and sugar content - Closest structural resemblance to native lignin - Low condensation - High homogeneity and purity	- Ideal for chemical functionalization - High reactivity - Excellent performance in polymer and composite applications	- High production cost - Requires solvent recovery - Industrial scale-up is challenging	[41]
Steam-exploded lignin	- Low to medium molecular weight - Partially condensed structure - High heterogeneity - Contains soluble and insoluble fractions	- Produced without chemicals (greener process) - Certain fractions may show higher reactivity	- Strong heterogeneity - Often requires additional purification or fractionation - Less uniform and pure than organosolv lignin	[46]
Acid hydrolysis lignin	- Strongly condensed and recalcitrant structure - Very low solubility - By-product of biofuel and carbohydrate conversion processes	- Low cost and abundantly produced - Useful for carbon materials, fillers, and energy applications	- Extremely limited reactivity - Extensive structural degradation due to harsh acid conditions - Limited suitability for chemical modification	[48]

is significantly chemically modified and exhibits distinctly different properties from native lignin.

CRediT authorship contribution statement

Mert Yildirim: Writing – review & editing, Writing – original draft, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

No data was used for the research described in the article.

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