Review Article

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# Pretreatment of lignocellulosic biomass conversion into biofuel and biochemical: a comprehensive review

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**Mohammad Siddique,[1,2] Ali Nawaz Mengal,[3] Suleman khan,[3] Luqman Ali khan,[4] Ehsanullah khan Kakar[5]**

[1]Department of Chemical Engineering, Mehran University of Engineering &Technology, Jamshoro, Pakistan
[2]Department of Chemical Engineering BUITEMS, Quetta, Pakistan
[3]Department of Mechanical Engineering BUITEMS, Quetta, Pakistan
[4]Institute of Petroleum & Natural Gas, Mehran University of Engineering &Technology, Sindh, Pakistan
[5]Department of Civil Engineering BUITEMS, Quetta, Pakistan

**Correspondence:** Mohammad Siddique, Department of Chemical Engineering, Mehran University of Engineering and Technology, Jamshoro, Pakistan,
Email siddiqnasa786@gmail.com

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## Abstract

The most potential feedstock for industrial civilizations is lignin derived from biomass. The most prevalent aromatic polymer on earth and one of the most difficult materials for commercial application is lignin. Reducing sugars, which can be used to make biofuels and some other products, are among the many chemicals that lignocellulose biomass releases during pretreatment. Lignocellulosic material (LCMS) is a material that is easily accessible, renewable, recyclable, and plentiful. Sustainability has gained traction as a result of climate change and environmental harm. The need for a flexible strategy to meet rising global energy demands has led many academics to concentrate on renewable biofuel made from sustainable sources. Construction of industrial biorefineries using lignocellulose feedstock for biofuel production and other bioproducts. The effective and scalable valorization of lignin is one of the main issues. Its presence prevents the biochemical conversion of lignocelluloses into fuels and chemicals, which depends on the extraction of cellulose and hemicellulose. To produce sustainable energy, lignocellulosic biomass must undergo pretreatment to speed up fragmentation and reduce lignin content. Temperature, time, particle size, and solid loading are the controlling factors for lignin extraction. This study covers the working conditions, parameters, yield percentages, techno-economic evaluations, challenges, and recommended next steps for the direct conversion of biomass to hydrogen. It detailed how green pre-treatment techniques can be used to produce green biofuels, and prospects for the application of green pre-treatment technologies on an industrial scale are also provided. The sustainable lignocellulose biorefinery has a path forward thanks to effective lignin recovery and valorization techniques.

**Keywords:** pretreatment, delignification, lignocellulosic biomass, biofuels, agricultural crop, renewable

## Introduction

The most prevalent type of biomass and least priced feedstock is lignocellulosic. It helps plants maintain their structural integrity. It was composed of cellulose, hemicellulose, and lignin, three different components. Due to excessive use in the energy conversion, industrial, residential, and transportation sectors, fossil fuels are arbitrarily running out. Fossil fuels will be fully depleted by the middle of this century; natural gas is currently a scarce and valuable resource that won't be discovered in 50 years, and fossil coal will be completely exhausted in 114 years.[1] In addition to the issue of supply, traditional fossil fuel combustion pollutes the air and the environment. Climate changes occur as a result of the emission of carbon dioxide into the atmosphere during the combustion of this fuel. Burning exposes nitrogen and its oxides, which causes rainwater to become more acidic.[2] Although greenhouse gases' main job is to maintain the earth's temperature, the climate cycle is collapsing as a result of their rising concentrations. Tons of lignocellulose are created every year from the enormous lignocellulosic biomass that exists across the planet. The way these services are delivered must undergo a major change to fulfill the increasing chemical and energy demands in a climate-constrained environment. In this context, renewable resources serve a crucial role as a sustainable source of chemicals and fuels, contributing to significant regulatory and industrial advancements. The applications for lignin, the second-most prevalent natural polymer and the only source of renewable aromatic compounds are numerous.[3] Biomass made of lignocellulose is a renewable resource that is plentiful and developing into a viable precursor to the carbon cycle. Recent research has focused on numerous agricultural leftovers as prospective sources of lignocellulosic biomass because of their availability, low startup costs, and quick biomass accumulation. Lignin is the third main component of lignocellulosic materials. Lignin has historically been used in the paper and pulp industries for power and heat because of its high calorific value. Different sources, including trees, crops, and plants, can provide lignin of various chemical compositions and structures with various properties.[4] Lignin is one of the by-products of lignocellulosic biomass processing for biofuels in the pulp and paper industry. During the pretreatment process, cell wall connections are broken down, making cellulosic and hemicellulosic fractions more accessible for subsequent applications and eliminating lignin fractions as insoluble leftovers. Various pretreatment methods have been created based on the varieties and traits of biomass sources. The utility of lignin, an abundant organic polymer, is discussed in this review study along with its composition for biofuel and bioproducts, applications, and potential future development. Neem tree bark and babul tree bark are two examples of biomass that have antibacterial potential and characteristics.[6] Green plants are the best source of lignocellulose biomass (LB), and between 60 and 90 percent of the plant, debris is used in the extraction procedure. Nearly every part of a green plant, including the leaf, root, stem, and bark, contains lignocellulose.[7] A green plant produces two billion tonnes of LB annually. Three categories of sources are used to categorize LB, including Virgin sources, energy sources, and trash sources are listed in that order. Apparently from a new source, there are a lot of trees, bushes, and green grasses to choose from in daily life. Energy biomass, which includes Panicum virgatum and Miscanthus Gigantes, is a groundbreaking LB source that yields an exceptional amount of secondary biofuel.[8] Secondary byproducts or trash from numerous industries are the waste source. Garbage is dumped and gathered on land and adjacent to water sources in order to clean up the area and develop a bioeconomy. This garbage includes municipal waste, agricultural waste, vegetable waste, corn waste, and waste from growing corn and maize. Biomass is produced using these byproducts.[9] The pretreatment for the separation of LB, which has as its major goals the removal of lignin and opening up of cellulose to make cellulosic sugar available for fermentation, is the high-impact step in the biohydrogen synthesis process.This step, which costs 30% more than the previous stage, makes up the majority of the production process. Lignin is extracted using a variety of techniques, such as thermal, mechanical, chemical, and biological ones.[10] Uniquely built apparatus, trained workers, and high energy sources are needed for physical and mechanical approaches. Hazardous and pricey chemicals are used in the chemical process. The generation of lipids is increased during the subsequent hydrolysis process, which also uses biotreatment to decrease hazardous components. The viability, dependability, and highly commercial character of biological technologies for pretreatment and hydrolysis have led researchers to advocate them for use in mass production.[11] The main challenges of this extraction technique are: 1) separating lignin, 2) creating the conditions for cellulose and hemicellulose hydrolysis and optimizing pressure and temperature, 3) appropriately bio-fermenting sugar, and 4) increasing production without the use of hazardous boosters.[12]

### Biomass made of lignocellulose: Properties and composition

Wooden biomass sometimes referred to as lignocellulosic biomass, can be easily obtained from forests and green trash. The total amount of dry biomass produced in the modern world is 820 million tonnes. As was already noted, cellulose, hemicellulose, and lignin make up the majority of LB, with minor amounts of phenolic and acetyl groups as well as minerals based on the source. Figure 1 demonstrates the features of lignocellulosic biomass conversion and the composition of biofuels. Around 50% of the makeup of the LB is made up of cellulose, a significant crystalline-shaped molecule disaccharide A repeating unit of the cellulose chain called cellobiose is tightly joined to glucose by powerful hydrogen bonds. Along with these three main components, there are traces of proteins, pectin, amino acids, metals, and ashes.[13]

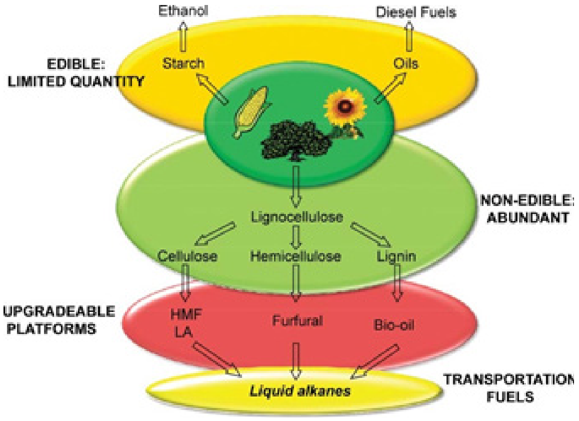

**Figure 1** Biomass-derived feedstock, platforms for biofuel conversion.[13]

## A multitude of physico-chemical techniques for delignifying LCB

The biochemical conversion of LCB is significantly hampered by the refractory nature of the lignin present in this substance. Since lignin prevents enzymes from accessing hemicellulose and cellulose during saccharification, delignifying LCB by pretreatment is an important step. Lignin can be recovered from biopolymers using a variety of chemical solvents, including alkaline reagents, ammonia, organosolv, deep eutectic solvents (DESs), and ionic liquids (ILs). To achieve a cost-effective bioenergy process, an efficient pretreatment procedure with less carbohydrates loss and maximum delignification is preferred.

However, since each component needs a particular set of reaction conditions to be depolymerized, it is difficult to meet all the aforementioned parameters in a single pretreatment step. Thus, for effective solubilization, multi-stage pretreatment techniques are frequently used. This section gives an overview of a number of delignification treatment techniques, covering both standalone and combined approaches.

## Techniques for lignocellulosic biomass pretreatment

To separate the lignin and hemicellulose parts of lignocellulose, several pretreatments are used. They can be classified as advanced pretreatment, biological or microbiological pretreatment, chemical pretreatment, physical-chemical pretreatment, or chemical-physical pretreatment. Figure 2 shows various lignocellulosic biomass pretreatment methods. This process is a very important preliminary step to hydrolysis. The following are some important considerations for pretreatments: (I) low cost and energy consumption with high feedstock yield; (ii) no harm to sugars, cellulosic compounds (iii) preference for non-hazardous chemicals and low pollution emitting treatments; (iv) absence of chemicals or agents that prevent fermentation and hydrolysis; and (v) minimal equipment and labour requirements. The type of pretreatment, environmental factors, and bioconversion method for generating hydrogen from lignocellulosic biomass are shown in Table 1 and lignocellulosic biomass in figure 3.[14]

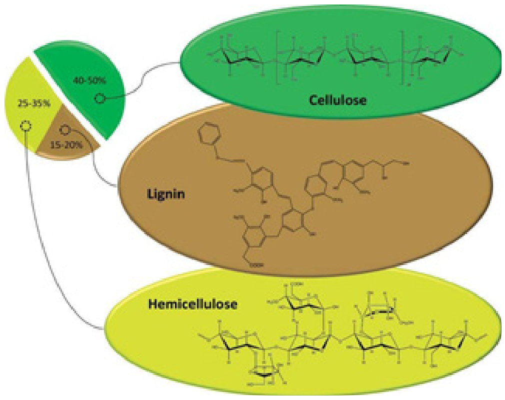

**Figure 2** Lignocellulosic compositions: Cellulose, hemicellulose, lignin.[13]

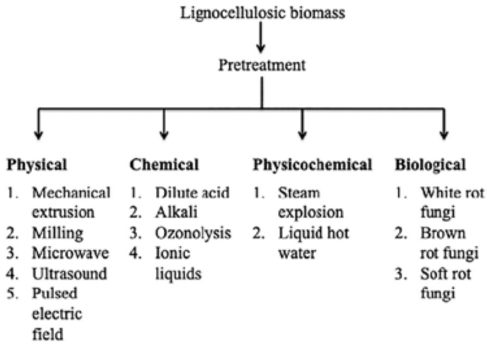

**Figure 3** Various lignocellulosic biomass pretreatment methods.

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**Table 1** Type of pretreatment, environmental factors, and bioconversion procedure for lignocellulosic biomass to produce hydrogen[15]

| Lignocellulose feedstock | Type of Pretreatment | pretreatment condition | Inoculum | bioconversion process | | | | Hydrogen production rate | Reference |
|---|---|---|---|---|---|---|---|---|---|
| | | | | Type of fermentation | pH | Temp (0c) | Time (h) | | |
| Municipal waste | Alkaline pretreatment | Agent: NaOH;<br>Temp:85°C<br>Time: 2h | *Clostridium lentocellum* | Dark fermentations | 7.6 | 56 | 2 | 5.429mL/g | Zhang et al,. 2019 |
| Seaweed | Thermal Treatment | Temp: 121°C;<br>Yield: Releases | *Ascophyllum nodosum* | Anaerobic Digestion | 4.48 | 41 | 4 | 5.8mL/g | Aneein et al,. 2018 |
| Wheat straw | Acid treatment | Agents: Dilute acid | *Escherichia coli* | Batch dark fermentations | 8.3 | 30 | 3 | 23.3mL/g | Lopez-Hidalgo et al,.2017 |
| Corn | Milling and grinding | Bead size: 0.2–2 mm; Temp: room temp; Time: 2 h | *Candida guilliermondii* | Dark Anaerobic fermentations | 6-8 | 38 | 24-48 | 34.17 g | Jatoi et al,.2022 |
| sawdust willow | Chemical | Agent: NaOH or H2O2; Temp: 80°C ;Time: 24 h | Pachysolen tannophilus and Wickerhamomyces | batch fermentation dark | 4.8–6 | 31 | 23 | 13.82 ± 0.20 g | Ben Atitallah et al,.2022 |

## Method for chemical pretreatment

The pretreatment method using chemicals such as alkalis, acids, and various mixtures of chemicals is more efficient.

### a. Alkaline treatment process

The base solution is successfully used in the alkaline treatment procedure to treat and break down the lignin component. This method causes the LB to swell while decreasing the crystallinity and polymerization rate. When CaOH, KOH, and NaOH are utilized as chemicals to treat the amorphous surface area, the method to reduce the amount of lignin is more effective. Even when bases prevent it, hydrolysis is used to treat agricultural, corn, and sugarcane waste.[16] The alkaline approach's basic operating premise is cellulose's propensity to swell when submerged in an alkaline solution. The technique produces the highest biomass when lignin is absent. When breaking carbon bonds in green biomass, they are highly selective. At 30°C, where 73% of the product is produced, even gaseous ammonia performs better throughout the delignification process.[17]

### b. Acid treatment process

Acid pretreatment dissolves hydrogen bonds and van der Waals forces in hemicellulose and xylan content. HCl and $H_2SO_4$ are frequently utilized because they accelerate cellulose hydrolysis, hydrolysis of other organic materials, and reaction speed. Rarely dissolved, lignin is severely harmed by high pH and temperature conditions. After $H_2SO_4$ and $HNO_3$ treatment, glucose output increases, however, these chemicals also release hazardous compounds called sulfate or nitrate. Acid therapies are employed to stop the recalcitrance reaction, which cleaves the Vander wall attraction by deteriorating carbon bonds and breaking carbohydrates. The initial step in the synthesis of biohydrogen is pretreatment, and 77.5% of the biohydrogen is produced as a result.[18]

### c. Polymeric substances based on lignin

Lignin, which takes up between 15 to 40% of the dry biomass weight in plant cell walls, is the second most common biopolymer.[19] Given that it is a complex, highly branching polymer with several aromatic moieties and functional groups, including hydroxyl, methoxyl, carbonyl, and carboxyl groups, it has the potential to be used in a variety of polymeric materials. For lignin to be reactive, the aliphatic and phenolic hydroxyl groups in particular are required.[20] Still, they are thought of as reactive zones. Included are the open ortho locations, the C5 positions on guaiacyl units, and the C3 positions on the p-coumaryl type units on the phenolic rings of the phenylpropane units. Due to the numerous stiff aromatic groups found in lignin, it has a reinforcing effect and can provide stiffness and strength.[21] Lignin has frequently been used in the past as a filler or physical blending component in plastic composites. We will focus on complex chemical processes in polymers made from lignin.[22] Table 2 summaries lignin materials.

**Table 2** Common agricultural residues and wastes: hemicellulose, cellulose, and lignin content[23]

| Lignocellulosic material | Cellulose (%) | Hemicellulose (%) | Lignin (%) |
|---|---|---|---|
| Grasses | 25-45 | 35-55 | Oct-35 |
| Wheat straw | 30 | 55 | 10 |
| Nut shells | 20-35 | 25-35 | 30-40 |
| Leaves | 25-Oct | 85-90 | 0 |
| Cotton seed hairs | 80-95 | 20-May | 0 |
| Newspapers | 40-55 | 25-40 | 20-35 |

### d. Lignin based polyesters

Common methods for producing polyesters include polyesterifying hydroxycarboxylic acids on their own, polyesterifying dicarboxylic acids with diols or dihalides, and ring-opening polymerization of lactones and cyclic esters. The polymer backbone of polyesters has repetitive ester bonds.[24] Grafting polymer chains to lignin's hydroxyl groups is a standard technique for creating lignin-based copolymers. This method has two subcategories: "grafting-from" and "grafting-to.[25]

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### e. Epoxy resins based on lignin

Epoxy resins are thermosetting polymers containing an oxirane ring. They may need extra curing agents, most often amines, or they may self-crosslink on their own. They have a wide range of applications in coatings, adhesives, high-performance composites, coatings for electronics, etc. because of their highly tuneable characteristics and generally great heat resistance.[26] Due to the presence of aromatic rings in its molecular structure, lignin can serve as an appealing bio-based substitute for the vast majority of commercial epoxies made from bisphenol A (BPA), which is harmful to human health and the environment [27].There are several methods for adding lignin to epoxy resins, however the two that are most frequently used are (1) producing epoxy prepolymers from lignin directly or (2) making curing agents from lignin or modified lignin.[28]

### f. Phenolic formaldehyde resins based on lignin

When phenol and formaldehyde step-growth polymerize in the presence of an acid or base catalyst, phenol-formaldehyde (PF) resins are created. Due to their many advantageous qualities, such as strong adhesion, thermal stability, and resistance to water and chemicals, this class of resin is widely utilised in the wood industry as a bonding medium for plywood, oriented strand boards, and other engineered wood products. Some investigations look for bio-based substitutes for crosslinkers in lignin-based PF resins as formaldehyde is a recognised carcinogen. In a recent work, Hussin et al. created PF resins by glyoxal, a dialdehyde derived from a range of natural resources, in place of formaldehyde in a ratio of 50%.[29] In organosolv lignin phenol glyoxal (OLPG) resins that were employed as wood adhesives in plywood samples, up to 50% by weight of phenol was substituted with organosolv lignin. In comparison to market PF wood adhesives, the plywood made with 50% OLPG showed noticeably greater tensile strength and modulus of elasticity in both dry and wet circumstances. Another biobased substance that is frequently utilised in PF resins as a formaldehyde substitute is a hydroxymethylfurfural (HMF). It is an aromatic aldehyde generated from lignocellulose and cellulose that is used to make a variety of compounds.[30] (Figure 4)

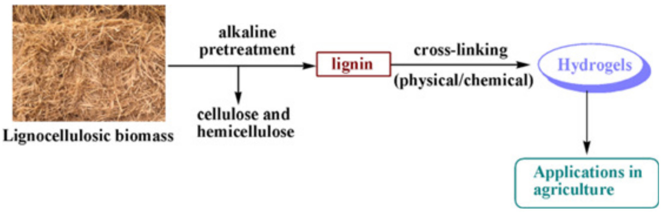

**Figure 4** Extracted lignin and synthesis of lignin-based materials.[31]

## Conclusion

This paper summarizes developments in lignocellulosic biomass chemical pretreatment techniques. Green biomaterials are in higher demand than ever thanks to their affordable production costs. Lignin has stabilizing characteristics and is a part of new green material. Lignin biomass is a naturally occurring resource that can serve as an alternate application concerning economic importance. The preparation of the lignocellulosic biomass pretreatment method in terms of liquid and chemical loading is reviewed in this research. The overall goal of this article is to construct a whole biohydrogen synthesis from waste lignocellulosic biomass, starting with pretreatment techniques and ending with the final product. Several pretreatment techniques, as well as outdated and best economically safe procedures, are discussed. Numerous LB applications, their ability to produce hydrogen at low production rates, and several methods for increasing yields to 83% are discussed in this study. Operation settings, biological synthesis methods, and their socioeconomic advantages have all been investigated. Future perspectives with real-world issues are highlighted, as are strategies to boost yield rates by 20–30%. Lignin can be recovered using organosolv and alkali pretreatments, which need fewer unit operations. Lignin can subsequently be valorized using thermochemical and biological platforms for use in beneficial applications.

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## Conflicts of interest

The author declared no conflicts of interest concerning the research work.

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