Pretreatment Methods for Lignocellulosic-Based Biomass to Provide Sustainable Biofuel and Environmental Benefits. A Review

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Abstract

Agriculture, food and food-processing sectors produce 1.3 billion tonnes of lignocellulosic-based biomass (LCB) globally each year. LCB is a viable alternative energy source, due to its economical nature and minimal waste production. LCB may be efficiently transformed into fuels like bioethanol, biodiesel and biogas, along with higher-value products, such as organic acids, enzymes, biopolymers and charcoal. Nonetheless, owing to its structural intricacy and resistance, LCB presents difficulties for large-scale application. Consequently, its preparation is essential for effective delignification. The approach isolates hemicellulose and cellulose (CL) from lignin within a complex polymer matrix. This diminishes matrix size, while augmenting surface area of CL molecules, hence enhancing their accessibility to microorganisms and enzymes for hydrolysis and fermentation, respectively. Pretreatment procedures may be classified as chemical, physical, chemical/physical or biological. Physical methods include mechanical grinding, ultrasound and electromagnetic radiation. Chemical methodologies comprise acid/alkaline hydrolysis, organosol processes, ionic liquids and ozonolysis. Physical-chemical processes encompass ammonia fibre explosion, CO_2 detonation, moisture explosion and liquid hot water filtering. The use of particular yeasts, fungi and bacteria in biological pretreatment processes is not yet extensively implemented.

Keywords: biofuels; biomass; hydrolysis; pretreatment procedures; value-added goods.

Introduction[•]

The global energy crisis, driven by the increasing reliance on nonrenewable fossil fuels to meet industrial development demands, jeopardises human society. World

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[•] The abbreviations list is in page 113.

population of 7.3 billion is projected to rise to 8.5 billion, by about 2030, and 9.7 billion, by 2050. Worldwide energy consumption has been estimated to increase by 28%, since 2015 (around 600 kJ) to 2040 (about 768 kJ) (Environmental Impact Assessment, 2017). These projections present issues about oil reserves, gasoline pricing, greenhouse gas emissions and pollution [1]. Concerns regarding ramifications of global warming, instability and, particularly, national security, have renewed interest in renewable energy.

Consequently, research focused on identifying and utilising alternate energy sources has been on the rise [2]. Renewable power, commonly referred to as energy derived from plants, has emerged as a feasible alternative. Solar, wind, tidal, geothermal and biomass renewable energy sources are employed to mitigate greenhouse gas emissions and enhance energy efficiency. Biomass possesses considerable potential for biofuel production, diminishing dependence on fossil fuels, and ensuring food security.

Renewable fuels, or biofuels, are a cost-effective and environmentally sustainable alternative to fossil energy, accounting for approximately 1.5% of global demand. They generate markedly reduced greenhouse gas emissions relative to fossil fuels, and include ethanol, bioethanol, biodiesel and biogas. Biofuels are classified into first and subsequent generations, according to their origin. Preliminary forms originate from consumable plant constituents, such as sugars, particles and seeds cultivated on agricultural soil [3]. They represent advancement towards attaining energy autonomy and a shift from fossil fuel combustion for energy generation.

However, the advancement of first-generation biofuels is problematic, as economists and ecologists oppose it, due to its potential danger to food supplies.

Non-consumable plant components, such as agricultural wastes, woody biomass and lignocellulosic biomass (LCB), represent second-generation bioenergy sources. This type of biomass demonstrates greater promise than first-generation biofuels, due to the lack of competition for basic resources and direct utilisation of arable land [4].

The present study examines pretreatment methodologies for the production of biofuels and value-added products from LCB. It delineates most recent breakthroughs and limitations from each methodology [5].

Lignocellulosic biomass

Lignocellulosic biomass (LCB) is a sustainable and abundant organic bioresource derived from waste byproducts in food and agricultural sectors, of which composition includes 40-60% cellulose (CL), 25-35% hemicellulose (HCL) and 15-40% lignin [6].

Therefore, LCB is the most economical, plentiful and sustainable alternative fuel, since it is able to produce an array of value-added products, such as organic acid-based biopolymers, rodenticides, animal feed, biological fertilisers, seafood nutrition, fish oil, face toner, fragrances and compost conditioners (Table 1).

Table 1: Detail of LCB leftovers yield biofuels and value-added goods [7].

LCB source	LCB residues	Generated biofuels	Value added goods	Ref.
Forest vegetation	Logs, bark, leaves	Natural gas	Biochar, syngas, soil fertiliser	[8]
Vegetables, fruits	Seeds, peels	Ethanol from plants,	Flavors, one oil, face toner,	[9]
Cotton	Seeds, stalk, fibers	Biodiesel	Biofertilizer	[10]
Oilseed plants	Shells, lint, husk	Biodiesel	Biofertilizer, animal feed	[11]
Sugarcane	Bagasse	Bio methanol	Paper products feed animals	[12]
Wheat	Straw	Bio methanol	Biopolymers	[13]

Anaerobic fermentation of LCB generates bio-methane, which is economically advantageous and environmentally friendly [14]. It may be a substitute for natural gas, since it is a cost-efficient energy source across various sectors. LCB also produces bio-butanol, which is used for internal combustion and petrol blending. It is presently widely employed in cleaning formulations, pharmaceuticals and cosmetics. Additionally, LCB generates ethanol, which is a feasible substitute for petroleum fuel in transportation, power generation, oil spill remediation, paint degradation and lubrication. Production of bioethanol from sugarcane, rice waste and maize straw constitutes around 65% of total biofuels. Limited literature exists on sustainable synthesis of bioethanol from LCB. Researchers are exploring its possible use in petrochemicals, synthesised hydrocarbons and petrol formulation. The utilisation of an auxotrophic strain for bioethanol production from indigenous biomass remains under investigation.

Byproducts from bioethanol production can be utilised as soil conditioners, hence diminishing the need for organic fertilisers. LCB additionally assists in wastewater treatment. Nano CL, a more straightforward variant of CL derived from LCB, can be employed to produce translucent paper [15].

LCB pretreatment

LCB‘s intricate structure and resilience hinders its enzymatic hydrolysis. Therefore, its pretreatment, which isolates HCL, cellulose CL and lignin (Fig. 1), is a viable option for large-scale production of biofuels and value-added products, since it improves epidermal accessibility for enzymatic hydrolysis [16], reducing operational and capital costs [17].

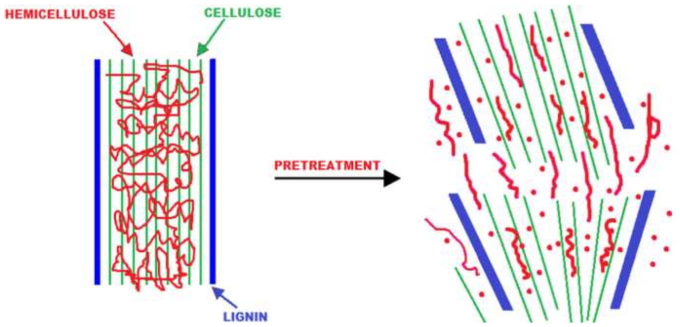

Figure 1: Disaggregation of CL and HCL from lignin.

The approach aims to decompose HCL into smaller fragments, hence augmenting the surface area accessible for enzymatic hydrolysis.
As seen in Table 2, chemical, psychological, physical and biological techniques are the main methods used for LCB's pretreatment [18].

Table 2: Key pretreatment methods for LCB.

Pretreatment methods for LCB			
Psysical	**Chemical**	**Physicochemical**	**Biological**
Mechanical extrusion	Dilute acid	Steam explosion	White rot fungi
Milling	Alkali	LHW	Brown rot fungi
Microwave	Ozonolysis		Soft rot fungi
Ultrasound	Ionic liquids		
PEF			

Table 3 outlines merits and demerits of LCB pretreatment methodologies. Prior to employing a pretreatment method, the following criteria must be evaluated: absence of reduction in biomass particle size; no alteration in HCL structural integrity; minimal production of inhibitory byproducts; low energy expenditure; and utilisation of a cost-effective pretreatment catalyst to recover valuable lignin byproducts.

Table 3: Advantages and disadvantages of LCB pretreatment procedures.

Method	**Different kinds of pretreatment techniques**	**Advantages**	**Disadvantages**	**Ref.**
Physical	Mechanical milling/grinding	Affordable for softwoods; no harmful inhibitors; enhanced surface area and flow properties of CL materials; lower response time.	Considerable energy and power; expensive equipment.	[19]
	Ultrasound	Shorter processing times; less chemical consumption; minimal hydrolysis time.	Energy-intensive; if extended, it may negatively impact hydrolysis.	[20]
	PEF	Ambient circumstances; low energy usage; no mechanical components.	Not appropriate for hardwoods.	[21]
	Extrusion	Reduced generation of breakdown products and toxic inhibitors.	Insufficient cooling capabilities; reduced residence time.	[22]
	Microwave	Low energy usage; shorter reaction times; few harmful inhibitors.	Low penetration in bulk goods.	[23]
Chemical	Acid hydrolysis	High solubility; short reaction and permanence period; high glucose output; for feedstock with low lignin concentration.	Very toxic, dangerous and corrosive; costly; possible harmful inhibitors.	[24]

	Alkaline hydrolysis	Lignin removal; partial HCL hydrolyzation; increases surface area reduces polymerization and crystallinity levels.	Low digestion; extensive residency time; high chemical expense for recovery; reduced effectiveness for lignin-rich biomass.	[25]
	Ionic liquids	Reduced crystallinity; easier phenol recovery and reuse; less harmful inhibitors.	Expensive chemicals; toxicity; irreversible CL inactivation.	[26]
	Organosolv	High-quality phenol for creating value-added goods; simple collection of solvents through distillation; less harmful inhibitors.	Expensive; needs close supervision.	[27]
	Oxidative delignification	Increased CL accessibility for enzymatic hydrolysis.	Reduced CL and HCL may inhibit microbes' development.	[28]
	Ozonolysis	Effective removal of lignin.	Expensive, due to high ozone Ct.	[29]
Physicochemical	Steam explosion	Less environmental impact by reduced pesticide usage; energy efficiency; no recycling costs.	Longer residence time; toxic inhibitors; insufficient lignin removal; dependent on biomass temperature, size and MC.	[30]
	CO_2 explosion	Minimal environmental effect; no harmful inhibitors; inexpensive; non-flammable; high conversion output.	Unchanged lignin or HCL; high CO^2 pressure; costly to process.	[31]
	LHW	No catalysts or chemicals; no harmful inhibitors; inexpensive.	Increased energy use; high water consumption; low-concentration products.	[32]
Biological	Bacteria, fungi	Lower energy usage; environmentally beneficial; no chemicals or catalysts; no harmful inhibitors; profitable and sustainable.	Slow; HCL partial hydrolysis; health hazards; lignin, CL and HCL microbial destruction; affected by physical factors.	[33]

Physical pretreatment techniques

Prior to enzymatic hydrolysis, LCB undergoes physical pretreatment to reduce polymerisation and particle size, hence enhancing surface area and accessibility, which makes the operation more efficient and simple. Physical pretreatment techniques for LCB disintegration include mechanical grinding, ultrasound, microwave treatment, pyrolysis, PEF and electromagnetic radiation. Although these methods are expensive, due to their substantial energy demands, and the fact that they are affected by temperature, time of residence and pressure levels, they produce few harmful compounds [26]. Additionally, when coupled with chemical pretreatment, these procedures exhibit a synergistic effect, and enhance yield [35].

Mechanical technique

Milling, grinding, cutting, and extrusion are mechanical techniques employed to prepare LCB, and the first two are the most often used ones. Moreover, the method is contingent upon the moisture content of the residue. Mechanical pretreatment enhances productivity, particle density, metabolic accessibility and overall conversion of LCB to biofuels, while minimising harmful byproducts. This approach enhances average density, porosity within covering region and flow properties of cellulosic carbohydrates [36]. However, milling procedure has demonstrated ability to reduce CL crystallisation, hence enhancing digestion of lignin biomaterial. Other milling techniques such as two-roll milling also enhance enzymatic hydrolysis in lignin-based materials

Variation in particle size and crystallinity is influenced by type of biomass, milling technology and processing duration. Rod milling of wheat straw has yielded significant size reduction, and has reduced crystallinity relative to alternative milling methods, with an optimal duration of 60 min [37].

Mechanical pulverisation of agricultural materials requires different power levels depending on plant quality and particles size. Comminution necessitates a substantial quantity of energy. To enhance biomass grinding equipment, it is essential to comprehend mechanical properties of biomass [38].

Initial particle size, feed rate, moisture content and machine settings significantly affect energy used for LCB size reduction. Milling method generates methane and ethanol, while avoiding production of detrimental inhibitors like furfural. Nonetheless, milling is not a financially feasible option, due to substantial energy requirements and increasing costs.

Pyrolysis

Pyrolysis is a thermal breakdown endothermic process that requires minimal energy, and it can be classified as slow, moderate, rapid or swift, according to temperature and residence time [19]. This method breaks down LCB at temperatures exceeding 300 °C, resulting in the formation of residual charcoal and gaseous byproducts. Dilute acid hydrolysis of residual char has resulted in about 80% degradation of CL into reducing sugars [39]. Employing specialised catalysts like sodium carbonate or zinc chloride enhances the method's efficacy in oxygen-rich settings.

Extrusion

Extrusion is an innovative approach for LCB pretreatment. During extraction, biomass suffers blending, heating and shearing, which leads to chemical and physical modifications, as it traverses the extruder at temperatures higher than 3000 °C [34].

The interplay between barrel temperature and screw speed alters LCB structure, resulting in fibre shortening and defibrillation, which increases carbohydrate accessibility for enzymatic hydrolysis. Enhancing reactor conditions can improve the technique's efficiency.

Pulsed electric fields (PEF)

PEF pretreatment method involves applying electrical power to vegetal materials or biomass situated between two electrodes. Application of a high-intensity electric field to a cell membrane induces rapid electromagnetic breakdown and structural modifications. The electric field enhances the permeability of solute per litre, and may cause mechanical damage to plant tissue [21]. Pretreatment with PEF during biomass-to-fuel conversion can expose CL in plant fibres. Plant cells have been shown to rupture significantly when subjected to strong electric field intensities from 5 to 20 kV per cm. PEF pretreatment induces irreversible disruptions in cell membrane, facilitating CL degradation via its constituent sugars by enzymes and acids. Chemical modification of plant material, including LCB hydrolysis, requires incorporation of an appropriate agent to decompose and assimilate the cell wall [22]. PEF pretreatment provides several advantages, such as ambient processing, low energy consumption (duee to 100 μs pulse duration), absence of mechanical components, and no requirement for specialised equipment design. Enzymatic digestion of switchgrass was significantly improved by PEF pretreatment [23].

Chemical pretreatment techniques

Acid and alkaline hydrolysis

Chemical methods employ compounds to enhance efficiency, such as acids, alkalis, ozone and organic solvents, which must be eliminated prior to enzyme hydrolysis, incurring in significant costs and releasing detrimental inhibitors.

LCB is desiccated prior to immersion in an acidic solution, at a certain temperature, for a designated duration. The treated substance is further filtered, isolating the liquid phase from the solid substrate. Acid hydrolysis entails the addition of concentrated or diluted acids (0.2-2.5 w/w) to a substrate, followed by continuous agitation at temperatures ranging from 130 to 201 °C. Acids including HCl, H_2SO_4 and HNO_3 can effectively destroy CL [24].

Dilute acid hydrolysis occurs under two conditions: at temperatures higher than 160 °C, with continuous flow, for modest solid loads (5-10%), and at temperatures lower than 160 °C, using a batch process for substantial material loads (10-40%). Highly acidic degradation employs potent acids, like HCl and H_2SO_4, without involvement of enzyme hydrolysis. Pretreatment using potent mineral acids may be poisonous and corrosive, requiring implementation of an anti-corrosion reactor. Cost of recovering extremely concentrated acids is high. Hydrolysis using mild acids has been effective for LCB pretreatment. It has been determined that dilute H_2SO_4 enhances both reaction rate and CL hydrolysis. This technique produces highly soluble lignin in HCl, yielding increased glucose synthesis without requiring enzymatic hydrolysis. Production of a significant amount of hydroxymethylfurfural, a detrimental inhibitor, diminishes the method effectiveness.

Alkaline hydrolysis procedure involves immersing LCB in solutions containing Ca, K, NH_4OH and Na, at a specified temperature and duration. Amplification of LCB material enlarges its internal dimensions, leading to significant CL decrystallization, inadequate HCL solvation, and modifications in lignin organisation [27]. Alkaline process is milder than alternative pretreatment techniques, and it has produced encouraging outcomes with maize burning, bagasse and softwood as primary materials. The efficacy of this approach is significantly dependent on lignin content of biomass. Main drawbacks of this method are extended residence period and neutralisation of previously processed sludge.

Physicochemical pretreatment techniques

Physicochemical pretreatment methods, which include ammonium fibre explosion, carbon dioxide explosion, hydration explosion and liquid hot water (LHW), are prevalent and cost-effective.

Hydrothermal treatment of biomass degrades and dissolves HCL, removing lignin and enhancing CL availability during hydrolysis. Hydrothermal processes are categorised based on temperature: hydrothermal carbonisation (HTC), LHW, pressurised hot water extraction (PHWE) and liquefaction with hydrothermal energy [29].

HTC is usually performed within temperatures in the range from 180 to 250 °C, transforming biomass into C molecules with distinct characteristics. This process encompasses hydrolysis, dehydration, polymerization, aromatisation and streamlined processes. At temperatures exceeding 280 °C, HTL yields crude, an oil-like material [32].

LHW method is carried out at temperatures ranging from 140 to 230 °C, which, in conjunction with PHWE, lead to partial destruction of lignin and HCL molecules, and to reduction in its structural integrity.

PHWE, conducted at temperatures in the range from 150 to180 °C, is employed to pretreat paper and decompose biomass, hence reducing CL concentration in products from forest biorefineries.

LHW is an effective technique for pretreatment of lignin-based materials. The method uses heated expanded water, at temperatures ranging from 160 to 260 °C, and high pressure, eliminating the need for chemicals or catalysts. There are three methods for LHW pretreatment, contingent upon biomass and orientation of water flow into the reactor: the first one involves co-current pretreatment, wherein biomass sludge is heated in water, at an elevated temperature, for a brief duration prior to its exposure to a cold environment [33]; the second one entails applying hot water to biomass under controlled conditions, referred to as countercurrent pretreatment; and the third one is flow-through pretreatment, wherein hot water circulates through LCB, to function as a stationary bed.

Efficacy of LHW pretreatment relies on maintaining a pH range from 4 to 7 during the process [34]. An alkaline or neutral pH may convert HCL into oligomers, hence restricting the formation of monosaccharides and other breakdown products. These processes offer the advantage of being executed at moderate temperatures, using less energy, and obviating the need for chemical agents. However, this approach has the drawback of producing low-concentration chemicals, due to water dilution [35].
Low-temperature extraction of water-soluble material components, such as pectin and tannin, requires maintaining equipment's temperature below boiling point of water (100 °C) [42].

Biological pretreatment techniques

Physicochemical and biological treatment methods are frequently employed in textile industry. Biological pretreatment is affected by chemical, physical and biological factors. It has become more preferred in recent years, due to its potential benefits over traditional physical, chemical or physicochemical pretreatment procedures, such as: it minimizes waste effluent, generates value-added products from LCB, requires lower energy consumption, has enhanced yield, is absent from detrimental inhibitors and promotes environmental sustainability [43, 44].
Biological pretreatment technique for preparing biomass employs bacterial and fungal combinations, being economically viable, environmentally sustainable and energy-efficient. Bacteria synthesise enzymes that decompose constituents of CL, HCL and lignin. Fungal group mostly consists of white and brown-rot fungi, microbes and basidiomycetes. Likewise, *Cellulomonas fimi* and *Thermoonospora fusca* have initially been utilised for CL production, while anaerobic bacteria such as *Bacteria thermocellulun* and *Bacteroids cellulosomes* exhibit significant CL activity, despite having low enzyme concentration. However, they have multiple drawbacks, including incomplete hydrolysis of glucose molecules, protracted processing times and microbial degradation of lignin and CL compounds, along with technical and economic limitations, which render it inappropriate for commercial use. At present, biomass processing requires environmentally sustainable methods. Research indicates an increasing interest in this environmentally friendly biological activity, highlighting the need for further investigation.

Conclusion

Biomass production constitutes a significant source of energy derived from renewable resources. The distinctive configuration of HCL and CL within lignin matrix renders it very resistant. To produce bioethanol and valuable products from LCB, lignin must be extracted through processing. Researchers around the world have developed successful pretreatment methods, encompassing chemical, physical, physiochemical and biological approaches. Chemical and physicochemical processes exhibit superior cost-effectiveness and efficiency in conversion. Every pretreatment procedure possesses distinct advantages and disadvantages contingent upon characteristics of employed biomass. Consequently, dependence on a singular pretreatment method may yield suboptimal outcomes. An effective and economical technology for disaggregating HCL and CL from lignin has not yet been developed. Understanding structure, composition and reaction mechanisms of biomass is essential for pretreatment methodologies. This may result in sustainable and economical technologies that lower energy consumption and enhance productivity. This research recommends employing LCB to maximise bioethanol production and generate supplementary value, since it is a cost-effective and environmentally sustainable resource.

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Authors' contributions

N. Karamat: improved manuscript, references and dois. **M. M. Memon**: supported in figures and tables. **A. A. Bhutto**: improved paper format. **M. Siddique**: rechecked the whole manuscript. **S. K. Suri**: removed mistakes in the manuscript, solved plagiarism. **M. S. Aamir**: helped with abstract and conclusion. **S. Parvaiz**: did proof reading of whole paper.

Abbreviations

AH: ammonium hydroxide
CL: cellulose
HCL: hemicellulose
HTC: hydrothermal carbonization
LCB: lignocellulosic biomass
LHW: liquid hot water
MC: moisture content
PEF: Pulsed Electric Field
PHWE: Pressurised Hot Water Extraction

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