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Chapter

Biomass Pretreatment and Characterization: A Review

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Abstract

Biomass has the potential to replace conventional fuels in a number of applications, particularly in biofuel production. It is an abundantly available renewable material with great potential as a feedstock for bioconversion processes for the production of energy, fuels and a variety of chemicals. Due to its biogenic origin, the carbon dioxide released from its combustion process does not impact atmospheric carbon dioxide. Despite these merits, a major problem hindering its widespread use has always been its recalcitrant nature, in terms of its inherent characteristics, which are unfavorable to its use in bioconversion and bio refinery processes. This makes it necessary for biomass to be pretreated before use in any conversion process for maximum product recovery. However, a major issue with regards to biomass pretreatment is the lack of rapid, high throughput and reliable tools for assessing and tracing biopolymer components of biomass relevant to the energy production potential of the biomass. This chapter therefore presents an overview of the pretreatment and characterization of biomass relevant to energy, fuels and chemicals production. The information provided will bequeath readers with the basic knowledge necessary for finding an auspicious solution to pretreatment problems and the production of energy from pretreated biomass.

Keywords: biomass, bioconversion, bio refinery, energy systems, energy production

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1. Introduction

The definition of biomass may vary due to a number of factors such as the heterogeneity of biomass, its application and origin [1]. However, any organic material directly or indirectly derived from the process of photosynthesis is considered biomass. Generally, biomass is a biological material that is present in various forms such as *wood*, *sugarcane bagasse*, *rice husk*, *rice straw*, *corn cob*, *paper waste*, *municipal solid waste*, *animal manure*, *sewage*, *algae*, *etc*. Traditionally, the use of biomass resources is becoming increasingly vital because of their substantial annual volumes and great economic potential. The main application of biomass is as a source of energy, and it has been reported that global biomass land and aquatic reserves stand at around 1.8 trillion tons for biomass, and about 4 billion tons for aquatic reserves, respectively [1]. In spite of these huge reserves, biomass remains under exploited, accounting for only about 14% of global energy with ca. 56 million TJ/year [1]. This is because the application of biomass as a source of energy has not shown a homogeneous distribution across the globe. In some developed countries,

energy production from biomass averages about 11% of the total energy produced; while in developing countries this can be up to 50% [2, 3]. For example, Europe generates about 3.5% of its energy from biomass, which is ca. 40 Mtoe/year; while countries like Austria, Finland and Sweden have about 13, 18 and 17% of their total energy produced from biomass resources; the United States on the other hand generates 3% of its energy from biomass [4]. In general, there is a huge potential for the exploitation of biomass as a source of energy because some countries in central and northern Europe have actually established large plants for heat and power production from biomass. However, there are two main routes by which biomass can be converted to energy and bio products. These are biochemical and thermochemical conversion routes. While the former involves breaking down biomass into gaseous and/or liquid fuels (such as biogas and bioethanol) through the use of bacteria, microorganisms and enzymes; the latter involves the use of heat to disrupt the complex chemical structure of biomass (particularly lignocellulosic biomass such as wood) into various products that includes heat, power, fuels, bio oil, biochar and chemicals [5, 6].

It is evident from the above that the application of biomass as a source of energy continues to attract global attention even in the midst of its gross underutilization. The low quality of biomass, often defined in terms of its inherent characteristics (high moisture content, low energy density, low bulk density, irregular size and shape) has been one of the major reasons for its under exploitation [7]. In other words, biomass in its natural form is difficult to use for the purpose of energy production hence pretreatment is often required to overcome its recalcitrant nature and make the biomass amenable for conversion through either of the conversion routes previously mentioned for maximum product recovery; the pretreatment leads to physical, chemical and structural changes to the biomass plays a vital role in the commercial viability of the energy production process of biomass [7–9]. Thus, bioconversion and bio refinery interests define the type of pretreatment measures for biomass [7, 10]. However, there are different categories of pretreatment viz; physical, chemical and biological pretreatments. These three classes of pretreatment are described in greater detail in Section 3.

Another critical step to the optimization of biomass conversion and bio refinery processes is related to the characterization of biomass to determine its suitability for the aforementioned conversion processes. This means that the effectiveness and impact of pretreatment on biomass can be determined through the use of a variety of high-quality analytical techniques able to provide information on quintessential biomass characteristics that can be used to maximize product recovery. Depending on the end application of biomass, some of the useful characterization techniques include *atomic force microscopy (AFM)*, *Fourier transform infrared spectroscopy (FT-IR)*, *scanning electron microscopy (SEM)*, *transmission electron microscopy (TEM)*, *X-ray diffraction (XRD)*, *solid state nuclear magnetic resonance (SSNMR)*, to name a few. An overview of these analytical tools is presented in subsequent sections. For a full understanding of the characteristics of biomass, its value and information for the design and operation of the energy conversion systems using the biomass as feedstock, it is vital to undertake biomass characterization before and after pretreatment. Due to the complex nature of biomass, the study of the mechanisms involved in its conversion process to energy and bio products is quite challenging. The lack of rapid, high throughput and reliable tools for assessing and tracing biomass components relevant not just to energy production but also to other value added products remains a major bottleneck in studying the impact of biomass pretreatment and process parameters. This chapter therefore presents a critical review of biomass pretreatment and characterization and discusses the applications of state-of-the-art analytical techniques commonly used to understand the features

of both pretreated and non-pretreated biomass relevant to the production of fuels and chemicals. It is believed that improving the fundamental knowledge of biomass pretreatment and characterization will lead to significant advances in the field of sustainable energy and chemicals production from biomass.

2. Overview of biomass composition

The composition of biomass is largely diverse and dependent upon its origin and species. Besides plant biomass, which are commonly referred to as lignocellulosic biomass (LCB) due to their three major cross-linked polysaccharide constituents (cellulose, hemicellulose and lignin), there are other biomass materials whose primary components include *lipids*, *proteins*, *starch*, *inorganics and minerals*. These types of biomass materials are regarded as non-lignocellulosic biomass (NLCB) and include *sewage sludge*, *animal manure*, *algae*, etc. The major constituents of both the LCB and NLCB are organic in nature and determine the characteristics of the entire biomass [7]. In comparison to LCB however, NLCB pose a greater threat to the environment because of higher content of heavy metals and heteroatoms such as nitrogen (N) and phosphorus (P); [11–13]. The heavy metals can pollute water systems, accumulate in food chains and cause serious health issues [14, 15]. Even though the N and P composition of NLCB can serve as a source of nutrients for plants, excessive amounts of these elements can lead to eutrophication of a water body, a condition linked to the deterioration of water quality via excessive growth of algae and other aquatic plants, resulting in oxygen depletion of the water body, leading to the death of aquatic animals [12].

While the structural unit of NLCB is such that various atoms are arranged in an orderly manner, LCB is characterized by complex internal structure with main components that equally displays structural multiplicities. **Figure 1** shows the internal structure of lignocellulosic biomass and how its three primary components are distributed.

The internal structure of lignocellulosic biomass reveals a crystalline fibrous structure of cellulose, which forms the core of the complex structure of plant biomass. Positioned between the micro- and macrofibrils of the cellulose matrix is hemicellulose; while lignin plays a structural role that encapsulates both cellulose and hemicellulose.

The composition of biomass varies significantly depending on the source of the biomass. In addition to the three primary components of LCB (**Figure 1**), there are other minor components such as *extractives*, *proteins*, *water* and *inorganic components*

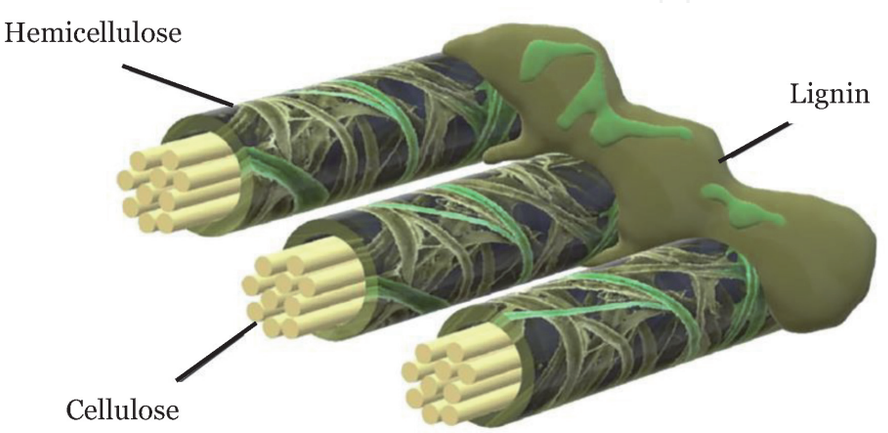


Figure 1.
A structural representation of lignocellulosic biomass. Reproduced with permission from [1].

Type of lignocellulosic biomass	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Hardwood	40–55	24–40	18–25
Softwood	45–50	25–35	25–35
Grasses	25–40	35–50	10–30
Leaves	15–20	80–85	–
Sugarcane bagasse	40–45	30–35	20–30
Wheat straw	33–40	20–25	15–20
Sweet sorghum bagasse	45	27	21

Table 1.
The composition of the organic fractions of various lignocellulosic biomass materials (dry basis) [17–20].

such as *silicon (Si)*, *sodium (Na)*, *potassium (K)*, *calcium (Ca)*, *magnesium (Mg)* and *aluminum (Al)*; these minor constituents do not markedly contribute to the formation of the total structure of the biomass [16]. The organic components of various LCB materials are summarized in **Table 1**.

The composition of NLCB, on the other hand, vary from material to material and contains more multifarious elements (such as those previously mentioned) that are embedded in its structural unit; in contrast to LCB, the different compositions of NLCB leads to different thermochemical conversion behaviors when these materials (NLCB) are used as feedstock in the mentioned bioenergy conversion routes [21].

3. Biomass pretreatment

Pretreatment is a necessary process step for both biochemical and thermochemical conversion of biomass and involves structural alteration aimed at overcoming the recalcitrant nature of biomass. It is required to improve biomass characteristics in order to enhance the energy utilization efficiency of the biomass [7, 22]. In pretreatment processes requiring heat, the degradation ability of LCB is controlled by its polymeric and aromatic constituents (cellulose, hemicellulose and lignin), while the heteroatoms and inorganic elemental components of NLCB act as catalysts to facilitate decomposition, leading to the formation of a product with a carbon framework and structural changes that increases the performance of the pretreated material in bioconversion processes [23–25]. The most important barriers facing current pretreatment technologies are high costs and how to obtain a pretreated product with minimal degradation of vital components. These issues are yet to be convincingly tackled by past and present research and development hence extensive studies aimed at the development of technologies that will further exploit the physical, chemical and biological pretreatment approaches are required. The pretreatment methods must be specifically tailored toward biomass origin and its application in bioconversion and bio refinery processes. **Figure 2** shows a schematic layout of the classes and types of pretreatment processes required for the two main conversion routes (biochemical and thermochemical) for biomass.

The following subsections present a further description of the main classes of pretreatment.

3.1 Physical pretreatment

Physical pretreatment of biomass is intended to reduce particle size by mechanical comminution in order to increase surface area and pore size. For LCB materials,

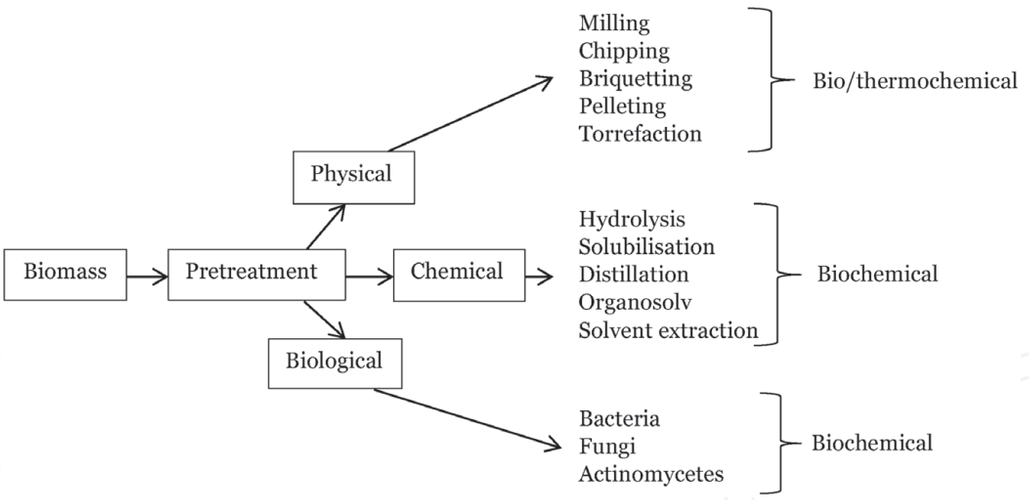


Figure 2.
A layout showing different pretreatment methods for biomass and their corresponding energy conversion routes.

physical pretreatment decreases cellulose crystallinity and degree of polymerization. It is a vital step prior to both the biochemical and thermochemical conversion of biomass [7, 26]. However, there is limited information about the mode of action of physical pretreatment processes, particularly with respect to how the chemical composition of biomass is modified or how its structure is affected. The application of biomass determines the type of physical pretreatment method to be applied. For instance, biochemical conversion of LCB will require size reduction through milling in order to improve enzymatic digestibility of lignocellulosic components. For thermochemical conversion applications of biomass, milling is required for densification, pelletization and even torrefaction prior to thermochemical conversion. In both conversion pathways (biochemical and thermochemical), prior size reduction is necessary in order to eliminate mass and heat transfer limitations. Chipping is also another physical pretreatment technique commonly employed when the biomass is to be used as feedstock in thermochemical conversion processes. This is because most thermochemical conversion systems are size specific hence require feedstock size in the range 50 mm by 50 mm in diameter [7].

As previously stated, densification, pelletization and torrefaction are all considered physical pretreatment methods for biomass intended as feedstock for thermochemical conversion processes [7]. These pretreatment techniques use heat to initiate changes that leads to improved biomass characteristics. A major drawback of the physical pretreatment technique however is its lack of ability to get rid of the lignin content of LCB materials, which renders the cellulose content of the material inaccessible. Other shortcomings include high energy consumption and the prohibitive cost of scale-up for commercial purposes. Studies [26, 27] have shown that the process of lignin removal from LCB materials could be one of the major reasons for the high energy demand of physical pretreatment techniques hence the overall energy efficiency of a bio refinery process may be ultimately affected by this pretreatment method [26].

3.2 Chemical pretreatment

Chemical pretreatment of biomass involves the use of organic or inorganic compounds to bring about disruption of biomass structure through interaction with intra- and inter polymer bonds of primary organic components. Biomass, particularly LCB materials are resistant to chemical breakdown hence biomass is regarded as being recalcitrant in nature. A host of factors are responsible for the recalcitrance

nature of biomass including the structural complexity and heterogeneity of biomass, the crystalline nature of its cellulose content, and the extent of lignification [7, 28]. Throughout the chemical pretreatment process, the structural recalcitrance of LCB is disrupted, resulting in the reduction of cellulose crystallinity and depolymerization as well as the degradation of cellulose and the breakdown of lignin [29, 30]. For biochemical conversion of biomass, particularly LCB, chemical pretreatment is commonly undertaken in order to isolate the respective biopolymeric constituents of the material. **Figure 3** shows the effect of chemical pretreatment on LCB.

Examples of compounds that have been used for the chemical pretreatment of biomass and which had significant effect on its structure include acids, alkali, organic solvents, and ionic liquids [32, 33].

3.3 Biological pretreatment

Biological pretreatment of biomass is mostly linked to the action of enzyme-producing fungi that are able to degrade, depolymerize and cleave the cellulose, hemicellulose and the lignin contents of biomass. This pretreatment method has several advantages over other pretreatment methods including its limited or no generation of toxic substances, high yield of needed products, low energy requirements and specificity of substrate and process reaction [34, 35]. However, its major disadvantages include the fact that the process is too slow and needs careful control of conditions of fungi growth as well as the large space required to carry out the process [36]. According to Agbor et al., 2011 [37], the residence time required for biological pretreatment processes is in the range 10 and 14 days. In addition, the organic components of biomass are consumed by the microorganisms' hence biological pretreatment processes faces techno-economic challenges and are considered commercially less attractive when compared to other pretreatment methods [38–40]. The types of fungi commonly used in biological pretreatment of biomass include *brown-*, *white-* and *soft-rot fungi*, *actinomycetes* and *bacteria*. These fungi are particularly known for their removal of hemicellulose and lignin as well as for their mild effect on cellulose. Nevertheless, *white-* and *brown-rot fungi* have a variety of ways to access and degrade LCB materials such as wood, and their very strong metabolism has been applied in industrial operations. For instance, *white-* and

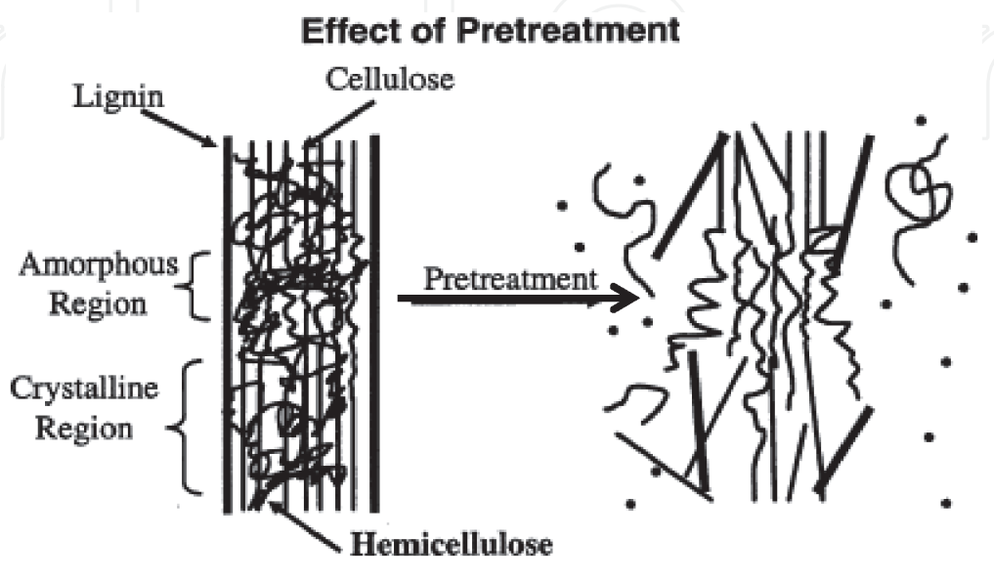


Figure 3.
A schematic representation of the impact of pretreatment on the surface and internal structure of lignocellulosic biomass. Adapted from [31].

Pretreatment methods	Advantages	Disadvantages	Reference
Physical pretreatment	• Simple and easy operation • Very large volumes of biomass can be handled • Increases surface area and pore size • Increases bulk and energy densities • Reduces cellulose crystallinity • Does not involve the use of chemicals	• Power consumption quite high • Require additional pretreatment steps	[8, 25, 43, 44]
Chemical pretreatment	• Increases accessibility to cellulose • Alters the structure of lignin • Hydrolyzes hemicellulose into various sugar fractions	• High cost of chemicals • Corrosion related issues with equipment • Liable to form inhibitory substances • Requires long residence time (time consuming)	[45–47]
Biological pretreatment	• Simple equipment • Efficiently degrades both cellulose and hemicellulose • Suitable for both high and low biomass moisture contents • Low energy requirements	• Slightly degrades lignin • Very slow rate of hydrolysis • The process requires a large space • Require long residence times	[36, 37, 48]

Table 2.
A summary of the advantages and disadvantages of the classes of pretreatment.

brown-rot fungi are known to have a brightening effect on kraft pulp obtained from hardwoods, with cost reductions in bleaching chemicals and potentially decreasing the environmental impact on paper mill operations [41].

In spite of the fact that many pretreatment methods have been investigated, while others are still in their developmental stages, it is quite onerous to assess and compare pretreatment technologies. This is because most pretreatment technologies involve upstream and downstream costs associated with processing, capital investment as well as complicated chemical recycling and waste treatment systems [42].

Table 2 presents a summary of the advantages and disadvantages of the different classes of biomass pretreatment.

4. Biomass characterization

Heterogeneity remains an inherent characteristic of biomass. The feasibility and viability of products recovery from biomass depends upon its properties. The two main conversion pathways earlier mentioned are basically used to recover products of value from biomass. The choice of the conversion route also depends on the features of biomass hence characterization is essential to better understand quintessential physicochemical properties of biomass that will determine how suitable the material is for conversion; these properties are keys to the efficient utilization of biomass in bioconversion processes [1, 7]. However, the characteristics of biomass are largely swayed by its primary organic components (cellulose, hemicellulose and lignin), which vary depending on biomass source, species, climatic conditions, etc. Depending on the end use of biomass, characterization of biomass is commonly determined and reported in terms of proximate and ultimate analysis using a variety of analytical tools some of which are described in subsequent sections of this review. This provides vital information for evaluating various application potential of biomass, particularly its energy production potential, which also takes into

Type of biomass	Proximate analysis (wt.%)			Ultimate analysis (wt.%)					
LCB	MC	VM	FC	A	C	H	O	S	N
Wood & woody biomass	5–63	30–80	6–26	1–8	49–57	5–10	32–45	<1–1	<1–1
Herbaceous biomass	4–48	41–77	9–35	1–19	42–58	3–9	34–49	<1–1	<1–3
NLCB									
Animal & human waste biomass	3–9	43–62	12–13	23–34	57–61	7–8	21–25	1–2	6–12
Aquatic biomass	8–14	42–53	22–33	11–38	27–43	4–6	34–46	1–3	1–3

Abbreviations: Moisture content (MC), Volatile matter (VM), Fixed carbon (FC), Ash (A), Carbon (C), Hydrogen (H), Oxygen (O), Sulfur (S), Nitrogen (N).

Table 3.
The properties of various lignocellulosic and non-lignocellulosic biomass materials [49].

account heating value when the biomass is used as feedstock in thermochemical conversion processes such as gasification [25]. **Table 3** presents the most important characteristics of various lignocellulosic and non-lignocellulosic biomass materials.

The proximate analysis estimates the physical properties of biomass with direct influence on the combustion performance of biomass, while ultimate analysis provides a qualitative and quantitative estimation of chemical properties in terms of the weight fractions of elemental components (such as C, H and O) and determines the heating value of biomass, a vital property of biomass materials intended as feedstock for thermochemical conversion processes; the amounts of other elements such as N, S and Si can help determine the environmental impact of using biomass as a fuel [25, 50, 51]. The proximate and ultimate analyses are discussed further in the following section.

4.1 Analytical techniques for biomass characterization

As earlier alluded, the determination of biomass characteristics often requires the use of a wide variety of state-of-the-art analytical techniques able to provide not just compositional information, but also pretreatment process validation. However, the type of analytical technique to be used is defined by the application choice of the biomass so that analyses results are interpreted in relation to the specific application. A few of the analytical techniques commonly used in characterization studies involving biomass materials are discussed in the following subsections.

4.1.1 Fourier transform infra-red spectroscopic analyzer

The Fourier transform infra-red spectroscopic (FTIR) is a sensitive technique for the qualitative and quantitative analysis of organic materials such as biomass. It identifies chemical bonds by generating a range of infrared (IR) retention in the form of spectra that represents sample profile. This analytical tool is particularly useful for tracing and tracking changes in biomass molecular structure caused by pretreatment and can distinguish between functional groups [51]. It is a useful technique for the characterization of biomass materials intended for a whole range of applications including biochemical and thermochemical conversion applications. For example, the FTIR can be used to investigate the possibilities of the removal of hemicellulose and lignin from a chemically pretreated biomass in a pulping process. It can also be used to understand the most reactive components of biomass

materials and how these components affect the thermochemical conversion process of biomass.

The FTIR technique relies on the fact that most organic materials absorb light within the IR region of the electromagnetic spectrum. The frequency of absorption of light is measured in wave numbers that is typically in the range 4000–600 cm^{-1}.

4.1.2 X-ray diffraction analyzer

To the best of the author's knowledge, the X-ray diffraction (XRD) is the only analytical instrument able to reveal comprehensive structural information of materials. Structural information that can be obtained includes *chemical composition*, *deformation*, *crystal structure*, *crystal size* and *orientation* as well as *layer thickness*. This instrument can be used to analyze a wide range of materials including nanomaterials. The XRD is equally a valuable analytical tool for studies involving biomass characterization for various applications. In XRD analysis, the extent of crystallinity is calculated based on an equation: (Eq. (1)) [52, 53]:

$$CrI\ (\%) = \left(\frac{I_{002} - I_{am}}{I_{002}}\right) \times 100\% \tag{1}$$

where CrI is the crystallinity index, while I_{002} represents the overall peak intensity and I_{am} the baseline intensity.

XRD is especially useful for the determination of the efficiency of hydrolysis for chemically pretreated biomass materials intended for the production of various chemical compounds such as sugar molecules and oligosaccharides, which are products of a fermentation process for the production of bioethanol.

4.1.3 Solid state nuclear magnetic resonance spectroscopy

For the analysis of biomass materials considered for the purpose of biofuels, biochar or chemicals production, the solid state nuclear magnetic resonance spectroscopy (SSNMR) is the ideal technique. This technique allows detailed structural elucidation of major constituents of biomass, particularly lignocellulosic biomass such as wood. It complements the XRD technique since the degree of cellulose crystallinity can also be determined. Nonetheless, the XRD is based on the proposition that X-ray scattering can be divided into two structural components that are amorphous and crystalline [54], while SSNMR is characterized by orientation-dependent interactions that are observed in a very broad spectrum that provides detailed information on material chemistry, structure and dynamics in the solid state. The production of biofuels and chemicals from lignocellulosic biomass require an unfettered access to cellulose and hemicellulose, thus SSNMR can be used to comprehend bioconversion of biomass as a function of process conditions [55].

The chemical barriers resulting from lignin renders the hydrolysis process of biomass into fermentable sugars quite complicated. Therefore, advances in spectroscopic techniques, especially spectroscopic methods, have enabled researchers to elucidate the structural characteristics of biomass in relation to specific applications.

4.1.4 Thermo-gravimetric analysis analyzer

When there is a need to investigate the combustion behavior of biomass materials, the thermo-gravimetric analysis (TGA) is particularly useful and valuable for determining thermal parameters relevant to the thermochemical conversion of biomass. Proximate analysis data can be obtained from TGA. In this technique, the

sample is combusted at desired heating rates in a chemically inactive atmosphere of nitrogen or argon such that the mass of the sample is monitored as temperature increases. The change in mass of the sample is usually plotted as a function of time or temperature. The TGA is a high temperature analytical instrument that adequately mimics the conditions existing in a typical thermal energy production system [56]. For studies involving the need to determine the kinetics of thermal decomposition of biomass, TGA is equally very helpful as it provides qualitative information that can be used to understand process conditions and design parameters of thermochemical conversion systems [57]. This requires that TGA be conducted at different heating rates and its derivative (DTG) used to simplify the reading of the characteristic peaks obtained from the thermogram of change in mass versus temperature.

4.1.5 Differential scanning calorimetry

The differential scanning calorimetry (DSC) is a thermoanalytical tool used to directly assess the heat energy uptake that occurs in a sample within a controlled increase or decrease in temperature. The instrument monitors phase transitions that lead to heat flow between crucibles since the process involves the heating of two crucibles (one which contains the sample to be analyzed, and the other serving as a reference without a sample). In this analysis, heat flow is measured as a function of temperature so that combustion profiles that will help determine the series of stages that characterizes the thermal performance of a material can be evaluated. In some instances, the DSC can be used as a complementary analytical tool to the TGA, particularly when monitoring softening or glass transition temperature range [8]. The DSC is very valuable for the analysis of biomass materials intended as feedstock for thermochemical conversion processes and the data from DSC can help improve the understanding of the effect of activation energy on the rate of biomass conversion in the aforementioned thermal energy systems [58]. The reactivity of biomass materials can also be determined using DSC and the volatiles from the analysis can be identified using gas analyzers.

4.1.6 CHNS analyzer

The CHNS analyzer is the carbon (C), hydrogen (H), nitrogen (N) and sulfur (S) analyzer used to measure the weight percentages of these elements in a given material. In CHNS analysis, the weight fraction of oxygen is usually determined by difference with respect to a generally accepted equation (Eq. (2)).

$$O = 100 - (C + H + N + S) \tag{2}$$

The CHNS analyzer is an elemental analyzer whose principle of operation is based on combustion that allows the quantitative determination of the above elements without the need for time consuming sample preparation steps or the use of toxic chemicals. Elemental composition is one of the most important features for biomass utilization [25, 59]. In addition to facilitating the determination of the environmental compatibility of using biomass as a fuel in thermal energy systems, CHNS analysis can be used to obtain information about calorific value and establish the combustion performance of biomass, if the weight proportions of primary elemental components such as C, H and O are known. One of the simplest ways to calculate the calorific value of biomass without the need for analytical tools is from an equation developed by Sheng and Azevedo, 2005 [60]:

$$CV(MJ/kg) = -1.3675 + 0.3137C + 0.7009H + 0.0318O \quad (3)$$

where CV is the calorific value of biomass.

Calorific value is an important property of biomass for design calculations or numerical simulation of thermochemical conversion systems using biomass as feedstock [25, 60].

4.1.7 Pyrolysis-gas chromatography/mass spectrometry (Py-GC/MS)

The Py-GC/MS is a technique used to identify non-volatile compounds. It involves high temperature heating of a sample to decomposition into smaller molecules that are separated by gas chromatography and identified by mass spectrometry. This technique is particularly suited for the analysis of biomass materials intended as feedstock in pyrolysis or hydrothermal liquefaction (HTL) processes for the production of charcoals and bio-oils as the mechanisms involved in these two thermochemical processes (pyrolysis and hydrothermal liquefaction) can be conveniently investigated. For example, pyrolysis of biomass is a relatively complex process that involves both simultaneous and successive chemical reactions which occurs when the biomass is heated in an unreactive environment. Due to the compositional and structural variability of biomass, major constituents degrade under non-identical mechanisms at different temperature ranges and at different rates. Therefore, to explore the complexity of this process, cutting-edge analytical tools such as the Py-GC/MS are required.

4.1.8 Scanning electron microscope

The scanning electron microscope (SEM) is a type of electron microscope that produces the image of a sample by scanning the surface of the sample with a focused beam of electrons that interact with the sample to produce a variety of signals used to obtain information about surface composition and topography. The macroscopic nature of biomass requires that some form of pretreatment, such as size reduction, be performed in order to reveal properties of interest for any microscopic and nanoscopic analyses. Thus, by employing imaging techniques such as the SEM, it is possible to study the physical and chemical underpinnings of the prodigy of biomass recalcitrance to breakdown. The SEM can be used to investigate the morphological properties of biomass relevant to the specific application of the biomass. The information obtained can then be used to hone biomass pretreatment methods that will enhance biomass susceptibility to biochemical or thermochemical conversion. It is however worthy to mention that the moisture content of biomass can be very problematic to some microscopic techniques (such as the SEM) since analyses using these techniques are usually performed on dry samples. As such, samples with reduced moisture content are often required before analysis to avoid the introduction of structural artifacts that may interfere with the SEM images of the sample.

5. Future prospects

The large-scale substitution of fossil fuel with biomass resources is a topical issue not just for the production of energy but also for the production of chemicals, bio products and materials. Moreover, due to the large availability of biomass throughout the world, the production of the high value-added products from biomass can be achieved under any geographical conditions and the feasibility and viability of

the production of the value-added products depends on biomass characteristics and the pretreatment method employed. However, biomass complexity and the high capital and operation costs associated with biomass pretreatment as well as the mechanisms involved in the conversion process of biomass are some of the challenges associated with the use of biomass for the production of energy, chemicals and fuels. Therefore, efforts should be geared toward the design of more easy-to-use and cost effective technologies at all levels so as to encourage the widespread application of biomass and attract investment in this field. In addition, not much is known about the optimal biomass pretreatment conditions because they are seldom reported. Consequently, for the efficient and feasible utilization of biomass in bioconversion processes, information about the optimum conditions of pretreatment is vital and efforts made to report such information. It has been reported [61] that researchers and policy makers are in need of useful information that may lead to the much needed improvements in this field of research. So, efforts made to report optimal pretreatment conditions for biomass will create further awareness on the advantages of the exploitation of biomass resources for the production of renewable energy and other bio products.

On the other hand, characterization of biomass also faces significant challenges ranging from the nature of the biomass and the multiphase bioconversion processes using the biomass as feedstock as well as the lack of experimental validation of the cutting-edge analytical techniques used for biomass characterization. Efforts made to address these barriers through continued research will equally lead to optimization of bioconversion and bio refinery processes. Needless to say that, since a single analytical technique cannot provide all of the needed information simultaneously with optimal resolution and high sensitivity, complementary techniques are often required to achieve full understanding of the physical and chemical underpinnings of the prodigy of biomass recalcitrance as it undergoes bioconversion processing. This may ease the challenges associated with experimental validation. Nevertheless, it is vital to mention that each type of characterization technique has its own merits and demerits under a particular set of circumstances and that the shortcomings of one technique may be compensated for by the merits of the other.

6. Conclusions

Pretreatment and characterization of biomass are key steps for the efficient utilization of biomass materials in bioconversion processes. A determination of the best pretreatment method and parameters requires an evaluation of its effects on biomass using cutting-edge analytical tools able to provide information that will facilitate better understanding of the origins of biomass recalcitrance and the mechanism and impact of pretreatment relevant to the optimization of different bioconversion pathways.

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Conflict of interest

The authors declare no conflict of interest.

Notes/Thanks/Other declarations

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Abbreviations

LCB	biomass
NLCB	Non lignocellulosic biomass
FTIR	Fourier transform infra-red spectroscopic
IR	infrared
XRD	X-ray diffraction
SSNMR	solid state nuclear magnetic resonance spectroscopy
TGA	thermo-gravimetric analysis
DTG	derivative thermogravimetry
DSC	differential scanning calorimetry
SEM	scanning electron microscope

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