Comprehensive Characterization of Lignocellulosic Biomass and their effective Delignification for Sustainable Bioenergy

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Abstract

The study provides a thorough examination of the biofuel potential of three unique lignocellulosic crop residues: rice straw (Oryza sativa), corn stalk (Zea mays) and sugarcane bagasse (Saccharum officinarum) of Odisha. In the investigation, we explored the compositional, thermal and structural characteristics of these biomass sources, shedding light on their suitability for sustainable bioenergy production. Proximate analysis indicated variances in critical factors in the range of 5.9-14.8% (moisture content), 1.8-19.4% (ash content), 60-72.4% (volatile matter) and 9.6-14.7% (fixed carbon) which contribute to the various energy generating capacities of these An in-depth investigation of cellulose, materials. hemicellulose and lignin concentration revealed the promise of sugarcane bagasse as a cellulose-rich option for bioethanol synthesis.

Thermochemical profiling using thermogravimetric and FTIR analysis revealed information about thermal stability and chemical changes, with pretreatment having an important role in increasing biomass accessibility and crystallinity. The significance of pretreatment-induced crystallinity for effective enzymatic *hydrolysis* and *fermentable* sugar generation was highlighted by X-ray diffraction (XRD) . Overall, this study advances our understanding of the intricate relationships between biomass composition, structure and bioenergy potential, offering valuable insights for the development of sustainable biofuel production strategies.

Keywords: Agricultural crop residues, proximate analysis, ultimate analysis, chemical pretreatment, bioethanol, crystallinity.

Introduction

The escalating global energy demand propelled by factors such as exponential population growth, industrialization and urbanization, has cast an urgent spotlight on sustainable energy solutions. While the reliance on fossil fuels has long been a linchpin of energy supply, the inherent limitations of these finite resources, coupled with the dire ecological repercussions of their utilization including pervasive climate change and environmental degradation, underscore the pressing need for innovative alternatives. Strikingly, a mere fraction merely 5% of today's energy consumption is derived from renewable sources underscoring the imperative to expedite the quest for transformative energy pathways⁹.

In this pursuit, biomass emerges as a compelling contender with the potential to redefine our energy landscape. Its innate ubiquity and cost-effectiveness render it an attractive candidate among the available renewable options¹⁴. The staggering global annual yield of biomass surpassing 220 billion tons from diverse origins⁷ ushers in a new era of energy exploration and innovation. Moreover, the valorization of agricultural residues as biofuel precursors offers an intricate tapestry of benefits, not only mitigating dependence on dwindling non-renewable resources but also nurturing a symbiotic circular economy, enriching both the environment and agrarian communities.

In this context lignocellulosic biomass found to be promising renewable energy source since its rich in cellulose. Lignocellulosic biomass comprising lignin (15-20%), hemicellulose (25-35%) and celluloses (40-50%)²⁸. The recalcitrance nature of biomass due to lignin hinders¹⁰ its' use in bioethanol production. Hence, it is necessary to characterise the biomass to find their suitability for the production of biofuel large scale³⁰.

Proximate analysis reveals the amount of moisture, quantity of ash, volatile substance, fixed carbon and the total amount of solids^{1,26} offering a preliminary glimpse into biomass's physical constitution. Conversely, ultimate analysis, delving into elemental constituents carbon, hydrogen, nitrogen, oxygen and sulphur offers an elemental perspective of biomass chemistry. The cellulose, hemicelluloses and lignin trinity, however, contain the key components of biofuel potential, demanding a transformational pretreatment regime to break down the resistant lignocellulosic matrix into hydrolyzable substrates¹⁹.

Our study demonstrates the comprehensive characterization and the chemical pretreatment of locally accessible biomass substrates namely, rice straw, corn stalks and sugarcane bagasse suggesting their large scale use for sustainable bioenergy production which further improves the economy of the farmers and reduces the dependency on fossil fuel²⁸.

Material and Methods

Collection of Biomass and Preparation of Samples: The research focused on three common agricultural crops: rice

straw (*Oryza sativa*), corn stalk (*Zea mays*) and sugarcane bagasse (*Saccharum officinarum*) cultivated in the Rayagada district, Odisha. These crops yield substantial quantities of lignocellulosic wastes, often either incinerated or left to decompose in the fields. For our investigation, we selected these crop residues to ascertain their potential for biofuel production. The crop residues were meticulously collected from the fields and subsequently transported to the laboratory for analysis. Upon collection, the biomasses underwent sun drying under ambient conditions for 48 hours followed by pulverization into fine powder form (particle size < 1.0 mm) using a pulverizer. The powdered samples were then stored in a hermetically sealed container to ensure their preservation for future use.

Proximate Analysis: The physico-chemical composition of the three crop residues was elucidated through proximate analysis of organic matter. 1 g of biomass sample was put in a dry crucible and baked at $105 \pm 30^{\circ}$ C for 3 hours to evaluate the moisture content. The moisture content was determined using the following formula:

 $\frac{(\text{Initial weight of the sample - Weight of the sample after drying)}{\text{Initial weight of the sample}} \\ \times 100 = Moisture \ content \ (\%)$

The total amount of solids of the biomass was then calculated as:

Total solids (%) = 100 - Moisture content

1 g of solid crop wastes was put in a furnace and fired in a Muffle oven at 575°C for 3 hours to determine ash content. The ash content was estimated using the equation by subtracting the weight before and after heating:

 $= \frac{(\text{weight of the container with ash} - \text{weight of the container})}{\text{weight of the sample taken}}$

$$\times 100$$

By putting 1 g of material in a sealed beaker within a Muffle oven at $925\pm100^{\circ}$ C for 7 minutes, volatile matter, indicative of non-water gases in biomass, was determined. The volatile matter content was estimated as follows:

Volatile matter (%) = (Weight loss – Moisture (%))

Here,

$$Weight loss (\%) = \frac{Weight before heating - Weight after heating)}{Weight before heating} \times 100$$

The fixed amount of carbon was calculated using the following equation:

Moisture (%) + Volatile matter (%) + Ash (%) = Fixed amount of carbon (%)

Ultimate Analysis: The ultimate examination reveals the elemental makeup of agricultural crop residues. The samples were analysed using a CHNSO detector (Flash clever V CHNS/O). At 980°C, 5 mg of material was flash combusted with a continuous flow of helium and oxygen gases. The percentages of elements carbon (C), hydrogen (H), nitrogen (N) and oxygen (O) in the biomass were determined using a GC-thermal conductivity detector ²⁴.

Compositional Analysis: The biomass's cellulose and hemicellulose content were determined using published techniques ²⁹. 1 g of oven-dried crop waste was combined with 100 mL of cold neutral detergent solution, 30 g of sodium lauryl sulphate and 10 millilitres of 2-ethoxy ethanol. The pH was set at 7.0. Neutral detergent fiber (NDF) was determined using the formula:

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NDF (%)
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= (weight of the container + NDF) – weight of the container weight of the sample taken

× 100

For acid detergent fiber (ADF) determination, 1.0 gram of the biomass sample was refluxed with 100 mL of cold acid detergent solution. Hemicellulose content was calculated as:

Hemicellulose (%) + ADF (%) = NDF (%)

Cellulose and lignin content were calculated using the equations²²:

Cellulose (%) = $(X - YW) \times 100$

 $Lignin (\%) = (Y - ZW) \times 100$

where X = ADF + crucible weight, Y = crucible weight + lignin, Z = crucible weight + ash and W = weight of the sample.

Chemical Pre-treatment of Lignocellulosic Biomass: Chemical pre-treatment, a popular approach for delignifying lignocellulosic biomass, comprises of either alkaline or acidic treatment, or a mix of the two. In alkali treatment, different bases like ammonium, calcium, potassium and sodium hydroxide are used. For many years, sodium hydroxide is commonly used and it has been shown to disrupt the lignin structure of biomass, increasing enzyme accessibility to cellulose and hemicellulose. In the present study, 5g of each LB sample was treated with 100 mL of various NaOH concentrations (1%, 2%, 3%, 4% and 5%). The samples were autoclaved at 15 psi for 15 minutes. The treated biomass was rinsed with distilled water until it reached a neutral pH and then dried overnight at 60°C. Similarly, in acid treatment, the dried biomass samples were treated with varying HSO₄ concentrations followed by autoclaving, washing and drying¹⁷.

Analysis of Pretreated Biomass: To assess the delignification of biomass samples, the treated samples were

analysed for free cellulose through anthrone test. 0.2 g sample was combined with 10 mL of 67% (v/v) sulfuric acid and incubated for an hour. The resulting solution was diluted and a portion was mixed with anthrone reagent. UV-Vis spectrophotometer (Systronics-119) was used to measure the optical density of the solution at 630 nm.

XRD and FTIR Analysis of Lignocellulosic Biomass: The crystalline index in terms of absorption peaks was determined using X-ray diffraction on the biomass sample. The XRD analysis revealed that cellulose exists in a variety of crystalline forms. X-ray diffraction research was carried out using a Cu-ka radiation source at an angle of 20 from 10°C to 40°C. Fourier transmission infrared microscopy (FTIR) was used to characterise functional groups in biomass before and after pretreatment with alkali (NaOH) and acid (H₂SO₄). FTIR analysis captured pre- and post-treatment differences in chromatograms throughout a wavelength range of 400-4000 cm⁻¹.

Results and Discussion

Identification and Selection of Biomass Sources: The study began by methodically collecting three unique lignocellulosic crop leftovers from the study region in search of sustainable and ecologically favourable energy sources. Herbarium specimens were rigorously processed and sent to the Department of Botany at Berhampur University for identification and scientific nomenclature. This verification procedure assured the accuracy and dependability of the crop species chosen. Duplicates of each herbarium specimen were also carefully conserved within the laboratory, providing as a repository for future reference.

Biofuel Potential of Lignocellulosic Biomass: The concept of biofuel generation from agricultural wastes, which is generally viewed as trash in impoverished and undeveloped countries, offers extraordinary promise as a sustainable energy option. Crop wastes rich in cellulosic and hemicellulosic elements appear as effective reservoirs for biofuel generation among the plethora of biomass resources. Our research focused on three regionally abundant crops with high lignocellulosic biomass content: rice straw (*Oryza sativa*), corn stalk (*Zea mays*) and sugarcane bagasse (*Saccharum officinarum*). **Proximate Analysis- Insights into Biomass Composition:** Proximate analysis which uncovers the elemental composition of biomass, is a cornerstone in determining its viability for biofuel generation³². In our study, we performed proximate analysis of lignocellulosic biomass such as rice straw, sugarcane bagasse and corn straw. The results are shown in table 1.

The moisture content, a pivotal determinant of combustion yield, ranged from 5.9% (rice straw) to 14.8% (corn stalk), exerting a pronounced influence on energy generation. Increased moisture content also increased the creation of unidentified gas products, indicating greater conversion of organic liquids; in general, thermochemical systems require feedstock with less than 15% moisture ⁴. Notably, rice straw exhibited the highest ash content 19.40% and sugarcane bagasse has the least ash content 1.8%. The biomass with more than 22% ash content reduces its calorific value ⁷. Though rice has nearly 20% ash content but its least moisture content (5.9%) offers its large scale use in biofuel production.

The volatile matter content, reflecting the combustible fraction, peaked at 72.4% in corn stalk, least in rice straw 60%. The presence of more volatile materials results in a larger liquid yield¹⁶. Higher levels of volatile matter suggested that fuel combustion would be simple whereas rice straw has maximum fixed carbon 14.7% and least in corn stalk 9.6%. When compared to other biomass, the amount of fixed carbon was found to be in a good range (13.73-15.55%) ¹⁷. Our findings support the notion that the chemical composition of lignocellulosic biomass boosts up bioenergy potential ¹⁸. The amount of minerals and inorganic elements in biomass is a critical factor influencing combustion efficiency.

Comparatively, wood, crop residues and rice husk bear ash contents of 0.5%, 5-10% and 30-40% respectively, underscoring diverse inorganic profiles ²⁷. Remarkably, corn stalk's volatile matter content surpassed other biomass constituents, delineating its energy rich character. Cellulose's importance in our research reflects its importance in biofuel synthesis. The variable mix of cellulose, hemicelluloses and lignin-critical polysaccharides determines the landscape for fermentation-based bioethanol generation.

Table 1
Proximate analysis of rice straw, sugarcane bagasse, corn straw biomass. The values are average of
three individual samples \pm SEM

Biomass	Moisture (%)	Ash Content (%)	Volatile matter (%)	Fixed carbon (%)	VM/FC ratio
Rice straw	$5.9\ \pm 1.2$	19.40 ± 1.9	60 ± 1.6	14.7 ± 1.5	4.08
Sugarcane Bagasse	13.5 ± 1.7	1.8 ± 0.9	71.5 ± 0.8	13.2 ± 1.7	5.41
Corn stalk	14.8 ± 1.3	3.2 ± 0.7	72.4 ± 1.4	9.6 ± 0.9	7.54

Sugarcane bagasse was found to be a cellulose rich entity in our investigation, with 45.3% cellulose content and high hemicellulose 26.4% and lignin content 21.2% as indicated in table 2. This demonstrates that the ability of biofuel generation is dependent on carbohydrate reserves. When compared to published cellulose, hemicellulose and lignin levels in other crops, our findings highlight the uniqueness of each biomass³³. Notably, sugarcane bagasse contains 71.7% aggregated cellulose and hemicellulose, making it an appealing material for bioethanol synthesis.

Ultimate Analysis- Elemental Signatures and Fuel Efficiency: Figures 1, 2 and 3 show the elemental spectrum derived from the final analysis. Carbon, hydrogen, nitrogen, sulphur and oxygen form the lignocellulosic biomass's elemental bedrock. Significantly, sugarcane bagasse contains highest carbon content (43.37%), hydrogen (6.25%) and oxygen (32.61%) whereas lowest carbon content (35.76%), hydrogen (5.2%) and oxygen (27.1%) were observed in rice straw. The nitrogen content was more in corn stalk (1.97%) and very negligible quantity was found in sugarcane bagasse (0.65%).

Elemental data provides insights into agricultural residue fuel efficiency, emphasising their unique roles in bioenergy generation. From the studies, biomass with carbon content (35%-45%), hydrogen (5%-6.5%), oxygen (30%-42%), nitrogen (0.5%-2%) and also high percentage of volatile matter leads to more biofuel yield²³.



Figure 1: Ultimate analysis of Rice straw biomass for carbon, hydrogen, nitrogen, sulphur and oxygen.



Figure 2: Ultimate analysis of sugarcane bagasse for carbon, hydrogen, nitrogen, sulphur and oxygen

Analysis of Pretreated biomass: The biomass samples such as rice straw, sugarcane bagasse and corn stalk were subjected for chemical pretreatment with acid (H_2SO_4) and alkali (NaOH). The pretreated samples were analysed for presence of free sugar (cellulose). The data presented in the figure 4 and 5 indicates that 2% concentrations of both acid

and alkali showed a significant delignification of biomass. Moreover, the alkali pretreatment is preferable over acid because alkali is more efficient in breaking the ester bonds between lignin, hemicellulose and cellulose and it can also prevent hemicellulose polymer breakdown⁶.

 Table 2

 Chemical analysis of rice straw, sugarcane bagasse, corn stalk biomass. The values are average

 of three individual samples + SEM

of three motividual samples \pm SEW						
Biomass	Cellulose	Hemicellulose	Lignin			
Rice straw	38.2±0.8	18.6 ± 0.4	14.8±0.1			
Sugarcane Bagasse	45.3±0.6	26.4±0.7	21.2±0.5			
Corn stalk	39.6±1.1	28.8 ±0.2	16.5±0.4			



Figure 3: Ultimate analysis of corn stalk for carbon, hydrogen, nitrogen, sulphur and oxygen.



Figure 4: Percentage concentration of Acid pretreated biomass - rice straw, sugarcane bagasse, corn stalk.

Thermogravimetric Analysis: TGA is a quick technique for determining the change in mass of a material with temperature. At extremely low heat and mass-transfer effects, this mass change offers accurate information on thermal stability, reactivity, reaction mechanism, breakdown content determination kinetics, and compositional analysis¹⁵. The first stage of decomposition takes place at temperatures ranging from 50 to 100 °C, owing mostly to water loss³. At temperatures ranging from 100 to 280°C, low molecular weight molecules and other volatile organic compounds (extractives) evaporate. At temperatures ranging from 100 to 200 °C, the biomasses are thermally stable.

Devolatilization commences between 250 and 300°C for the majority of samples. Weight loss at this stage is often lower in species with high lignin content. All biomasses lost most of the weight between 300 and 400°C, which might be attributed to sugar breakdown as shown in figure 6. The first

peak was attributed to hemicellulose breakdown (250-300°C) and the second to cellulose decomposition (300-400°C). Above 400°C, the biomasses lost weight steadily gradually owing to lignin breakdown³. Liberation of volatile compounds encompassing cellulose, hemicelluloses and lignin accentuates this phenomenon, paving the way for biofuel synthesis²⁵. TGA provides an approximation of the feedstock composition; such estimations can be used in screening activities.

FTIR Analysis: FTIR spectroscopy, which has been widely used to analyse the structural properties of materials before and after chemical treatments, can offer direct information on changes in functional groups and crystallinity. Figures 7, 8 and 9 represent the spectral diagrams of untreated and chemical treated (both alkali and acid) rice straw, sugarcane bagasse and corn stalk samples respectively.



Figure 5: Percentage concentration of alkali pre-treated biomass – rice straw, sugarcane bagasse, corn stalk.



Figure 6: Thermo-gravimetric analysis (TGA) analysis of crop residues (R: rice straw, B: sugarcane bagasse and C: corn stalk).

The spectral bands of untreated biomass samples were observed at the ranges of $3440.55 - 3447.46 \text{ cm}^{-1}$ (O-H stretching intramolecular hydrogen bonds for cellulose I), 2916.77- 2918.48 cm⁻¹ (C-H stretching for hemicellulose/ xylose), 1722.49 - 1733.97 cm⁻¹ (C-O stretching for the acetyl and ester linkages in lignin) and 1633.26 -1636.84 cm⁻¹ (associated with the aromatic rings present in lignin showing all the three biomass samples).

In the pretreated (both alkali and acid), biomass samples showed the peaks in the ranges of 3440.55- 3445.85 cm⁻¹ (O-H stretching intramolecular hydrogen bonds for cellulose I), 2855.32 - 2919.49 cm⁻¹ (C-H stretching for

hemicellulose/xylose) and 1508.57-1637.88 cm⁻¹ (aromatic rings).

However, the peak representing C-O stretching for the acetyl and ester linkages in lignin (1722.49 - 1733.97 cm⁻¹) is not observed in all the pretreated biomass samples, indicating the removal of lignin. Remarkably, pre-treated biomass samples exhibited a profusion of discernible downward peaks. Emblematic of distinct functional groups in the fingerprint region (1508.57-442.45 cm⁻¹) demonstrates the decreased crystallinity of the samples. The comparative analysis of alkali and acid treatment underscores alkali's superior suitability for fermentation, a finding of paramount significance in optimizing biofuel production²⁰.



Figure 7: FTIR analysis of crop residues viz. (a) Rice straw (b) alkali treated (c) acid treated



Figure 8: FTIR analysis of crop residues viz. (a) Sugarcane bagasse (b) alkali treated (c) acid treated

XRD Analysis- Crystallinity Index and Fermentability: XRD is a strong method for determining a material's crystallographic structure, phase composition and crystallite size. The crystallinity index of the samples was calculated using the amorphous subtraction method ²¹. Untreated rice straw exhibited a peak around $2\theta = 16.5^{\circ}$, 22.5° and 34.6° with a crystallinity index of 44% whereas acid treated rice straw exhibited a peak around $2\theta = 22.32^{\circ}$ and 34.9° with a crystallinity index of 58% and alkali treated rice straw exhibited a peak around $2\theta = 22.56^{\circ}$ and 34.90° with a crystallinity index of 60% as shown in figure 10. The characteristic assignments of the cellulose crystals are 110, 200 and 004 respectively.^{2,11,31}



Figure 9: FTIR analysis of crop residues viz. (a) Corn stalk (b) alkali treated (c) acid treated



Figure 10: XRD analysis of crop residues viz. (a) Rice straw (b) alkali treated (c) acid treated



Figure 11: XRD analysis of crop residues viz. (a) Sugarcane bagasse (b) alkali treated (c) acid treated



Figure 12: XRD analysis of crop residues *viz.* (a) Corn stalk (b) alkali treated (c) acid treated

X-ray diffraction (XRD) patterns of sugarcane bagasse untreated sample exhibited a peak around $2 = 22.02^{\circ}$, 30.5° and 34.72° with a crystallinity index of 48% which are supposed to represent the typical cellulose-I structure, similarly acid treated rice straw exhibited a peak around 2 = 22.66° , 28.50° and 35.67° with a crystallinity index of 62% and alkali treated sample exhibited a peak around $2\theta =$ 22.78° and 35.37° with crystallinity index of 65% shown in figure 11. The untreated corn stalk exhibited a peak around $2\theta = 22.09^{\circ}$ and 34.8° with a crystallinity index of 42% which is supposed to represent the typical cellulose-I structure whereas the alkali treated sample exhibits a peak around $2\theta = 16.39^{\circ}$, 22.77° , 27.14° , 27.15° and 34.91° with a crystallinity index of 64% as shown in figure 12.

Based on the above observation, acid or alkali pretreatment involves exposing the biomass to acidic or alkaline conditions to remove hemicellulose and lignin, thereby increasing the accessibility of cellulose. This process breaks down the less organised and amorphous portions of the biomass, resulting in an enhanced cellulose-rich substance¹². The increase in crystallinity observed in the XRD graph indicates that after pretreatment, the remaining cellulose becomes more ordered and structured. This is most likely owing to the elimination of amorphous components (hemicellulose and lignin) that hampered cellulose chain organisation⁵.

Higher crystallinity suggests that the cellulose is now more accessible to enzymes or other processes that may be involved in biomass conversion such as enzymatic hydrolysis for biofuel production. The increased structural order makes it easier for enzymes to bind and break down the cellulose into fermentable sugars¹³. In summation, our comprehensive investigation into the proximate and ultimate composition, thermal behavior and molecular architecture of lignocellulosic crop residues unveils their remarkable biofuel potential. The discerned composition and structural insights underscore the significance of judiciously selecting and pretreating biomass sources to harness optimal energy yield. By delineating the intricate interplay between biomass constituents and energy production, this study contributes to the evolving landscape of sustainable bioenergy generation.

Conclusion

In conclusion, this study systematically investigated the biofuel potential of three distinct lignocellulosic crop residues: rice straw (*Oryza sativa*), corn stalk (*Zea mays*) and sugarcane bagasse (*Saccharum officinarum*). The research encompassed a range of analytical techniques to assess the composition, thermal behavior and structural characteristics of these biomass sources.

Proximate analysis yielded significant insights into the fundamental characteristics of the biomass samples. Changes in variables including the amount of moisture, ash, volatile matter and fixed carbon were observed, each contributing to the unique energy generation potential of the materials. The research on cellulose and hemicellulose further demonstrated the variety of the biomass sources. Sugarcane bagasse, in particular, exhibited notable cellulose richness alongside significant hemicellulose content, indicative of its viability for bioethanol production.

Thermochemical profiling including thermogravimetric and FTIR analyses, unveiled valuable information about the thermal stability and chemical transformations within the biomass samples. The role of pretreatment in enhancing biomass accessibility and crystallinity was evident, underscoring its importance in facilitating enzymatic hydrolysis and subsequent biofuel production. X-ray diffraction (XRD) analysis provided valuable insights into the crystallinity and fermentability of the biomass. Pretreatment was found to contribute to increased crystallinity, enhancing the potential for efficient enzymatic breakdown and the generation of fermentable sugars. Collectively, this comprehensive study sheds light on the intricate relationships between biomass composition, structure and bioenergy potential. By elucidating these crucial factors, this research advances our understanding of sustainable bioenergy generation and provides valuable guidance for future endeavors in this burgeoning field.

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