*Archives of Current Research International*

***Volume 24, Issue 6, Page 78-84, 2024; Article no.ACRI.119725***
***ISSN: 2454-7077***

# Compositional Analysis of the Lignocellulosic Biomass from Agricultural Waste (Rice Husk)

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***Authors' contributions***

*This work was carried out in collaboration among all authors. Authors SM and PKO conceived and design the study. The analysis and interpretation of results were performed by author SM. Authors SM and SS prepared the manuscript. All authors reviewed the results and approved the final version of the manuscript.*

***Article Information***

DOI: https://doi.org/10.9734/acri/2024/v24i6766

**Open Peer Review History:**
This journal follows the Advanced Open Peer Review policy. Identity of the Reviewers, Editor(s) and additional Reviewers, peer review comments, different versions of the manuscript, comments of the editors, etc are available here: https://www.sdiarticle5.com/review-history/119725

***Short Research Article***

***Received: 04/05/2024***
***Accepted: 06/07/2024***
***Published: 08/07/2024***

## ABSTRACT

Rice husk, a byproduct of rice milling, represents a significant agricultural waste biomass with untapped potential for various industrial applications. This study provides a comprehensive compositional analysis of rice husk to explore its utility as a valuable resource. The chemical composition of rice husk such as initial moisture content, hemicellulose, cellulose, lignin, ash and extractives were analyzed. The result reported in mean value i.e 9.884 ±0.56 (%) moisture content, 8.077±0.27 (%) extractives, 17.210 ± 0.69 (%) lignin, 21.073 ± 0.62 (%) hemicellulose and 38.640 ± 0.94 (%) cellulose, 15.0 ±0.87 (%) ash. This study concludes that rice husk is an abundant source of cellulose and holds significant potential for cellulose isolation.

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***Cite as:** Malik, Sheeba, Pramod Kumar Omre, and Shreejaya Sivadas. 2024. "Compositional Analysis of the Lignocellulosic Biomass from Agricultural Waste (Rice Husk)". Archives of Current Research International 24 (6):78-84. https://doi.org/10.9734/acri/2024/v24i6766.*

*Keywords: Agricultural waste; rice husk; biomass; cellulose.*

## 1. INTRODUCTION

Agricultural waste refers to the by-products and residues generated during the cultivation and processing of raw agricultural materials. Rice husk, a lignocellulosic biomass is a prevalent agricultural by-product in many rice-producing regions worldwide. For every 100 kg of paddy grain processed into rice, approximately 20 kg of rice husk waste is produced [1]. In recent decades, lignocellulosic biomass has come to be seen as a valuable resource rather than waste. Its abundant availability and potential to be converted into valuable products, such as bio energy, chemicals and fertilizers, highlight its significance. This abundant resource can be utilized for energy production through direct combustion, biochar, and biogas generation. In construction, rice husk ash improves the properties of concrete and its insulating capabilities make it valuable for building materials. Agriculturally, rice husk improves soil quality and serves as effective organic fertilizer [2,3,4,5]. Rice husk derived silica has various Industrial applications such as construction for electronics, and filtration media [6]. Additionally, it can be incorporated into animal feed, bioplastics, and eco-friendly composites, promoting sustainability and reducing waste [7,8]. In general, agricultural and industrial wastes contain numerous valuable elements that can be extracted and utilized in the production of various materials. Among these elements, cellulose can be derived from such wastes [9].

The Rice husk is the material, that has high lignocellulose content. Typically, most of the agricultural lignocellulosic biomass is composed of about 10–25% lignin, 20–30% hemicellulose, and 40–50% cellulose. Cellulose is a primary structural component of plant cell walls, providing mechanical strength [10]. Hemicellulose, composed of repeated polymers of pentoses and hexoses, complements cellulose in the cell wall structure. Lignin, which consists of three aromatic alcohols (coniferyl alcohol, sinapyl alcohol, and p-coumaryl alcohol) synthesized through a biosynthetic process, forms a protective barrier around cellulose and hemicellulose, enhancing their stability and integrity [11]. Characterizing raw materials, whether for their composition, structure, or other significant properties, is a fundamental initial step in most experiments and industrial conversion processes. These properties determine the range of products that can be derived from a specific lignocellulosic feedstock in a cost-efficient manner [12]. The composition of lignocellulose generally depends on its source and varies due to the genetic differences among various sources [13]. Various contemporary methods for compositional analysis of lignocellulosic biomass are available such as weende method, klason lignin method and gravimetric method [12,14]. Out of which, NREL methods are commonly used for characterization of lignocellulosic biomass for engineering applications. In present work, the chemical composition analysis of rice husk is done to determine its potential applications.

## 2. MATERIALS AND METHODS

The Rice husk was collected from Madan milling, Kichha, Uttarakhand. The reagents and chemicals viz. ethanol, sulphuric acid and nitric acid were purchased from Sigma-Aldrich.

### 2.1 Initial Chemical Composition of Rice Husk

The chemical composition of lignocellulosic biomass can be divided into five components: cellulose, hemicellulose, lignin, ash, and extractives [15]. Consequently, the chemical composition of rice husk was initially assessed by determining its moisture content.

#### 2.1.1 Determination of initial moisture content

The moisture content of rice husk was determined using the Official Method of Analysis,1999 [16]. A 3-gram sample of rice husk was placed in a pre-weighed Petri dish and spread evenly with a spatula. The Petri dish containing the sample was then placed in a hot air oven at 105°C for 3 hours. After this period, the sample was removed and transferred to a desiccator to cool before being weighed. The sample was subsequently returned to the oven, and readings were taken at hourly intervals until a constant weight was achieved. The moisture content (w.b %) was calculated using the following equation:

$$M.C\ (\%) = \frac{W_1 - W_2}{M} \times 100 \quad (1)$$

Where,

$W_1$ = Weight of sample with petri dish before drying, g

$W_2$ = Weight of sample with petri dish after drying, g
M = initial weight of the sample taken, g

### 2.1.2 Determination of ash content

The ash content of rice straw was determined according to the standard laboratory analytical procedure of NREL [17]. A 3-gram sample of rice husk was placed in a dried and pre-weighed crucible. The crucible containing the sample was then heated over a flame until no more smoke was produced. After allowing the sample to cool, the crucible was placed in a muffle furnace at 575°C for 24 hours until a constant weight was achieved. Once cooled in a desiccator, the ash content was determined by the difference in weight, as calculated using equation (2):

$$AC\ (\%) = \frac{W_2 - W_1}{ODW} \times 100 \quad (2)$$

where,

AC = Ash content (%) in rice husk
$W_1$ = Weight of empty crucible, g
$W_2$ = Weight of crucible with sample after process completion, g
ODW = Oven dry weight of the initial sample taken, g

### 2.1.3 Determination of extractives

The determination of extractives was performed using ethanol extraction according to the National Renewable Energy Laboratory (NREL) procedure [18] as well as the method described by [19]. A 3-gram sample of rice husk was placed in a thimble and heated in a heating mantle. The extraction process continued for 24 hours. Upon completion, the sample underwent vacuum filtration followed by washing with 100 ml of ethanol. The sample was then air-dried and weighed. The extractives were calculated using the following equation:

$$E\ (\%) = \frac{W_2 - W_1}{ODW} \quad (3)$$

Where,

E = Extractives (%)
$W_1$= Weight of initial sample, g
$W_2$= Weight of sample after extraction, g
ODW = Oven dry weight of initial sample taken, g

### 2.1.4 Determination of lignin content

The total lignin content of rice husk was evaluated using a Laboratory Analytical Procedure (LAP) from the National Renewable Energy Laboratory (NREL) [20] as reported by Wang et al. 2018 [21]. Total lignin is the sum of acid-soluble and acid-insoluble lignin. For the determination of acid-soluble lignin, 0.3 grams of rice husk was placed in a 250 ml Erlenmeyer flask, and 3 ml of 72% sulfuric acid was added and mixed well with a Teflon rod. The sample was soaked in a water bath at 30°C for 2 hours. Subsequently, the sulfuric acid concentration was diluted to 4% by adding 84 ml of deionized water, and the mixture was autoclaved for 1 hour at 121°C. After the autoclaving cycle finished, the sample was removed, cooled to room temperature, and vacuum filtered. The filtrate was then diluted with deionized water to achieve an absorbance range of 0.7-1.0 Au, measured using a UV-Vis spectrophotometer at 205 nm. The acid-soluble lignin was calculated using the following equation.

$$ASL\ (\%) = \frac{UV_{Abs} \times V_f \times D}{\epsilon \times ODW \times Pathlength} \quad (4)$$

Where,

ASL = Acid insoluble lignin (% w of dry mass)
$UV_{Abs}$ = Absorbance for the sample at appropriate wavelength (205 nm)
$V_f$ = Volume of filtrate (86.6 ml)
D= dilution factor (9)
ε = Absorptivity of biomass at specific wavelength (110 L/g-cm)
ODW= Oven dry weight of sample, g
Path length = Path length of UV-Vis cell in cm (1 cm)

The residue remaining after the hydrolysis process for acid-soluble lignin was washed with hot water until the filtrate became pH neutral. The residue was then placed in a dried and pre-weighed crucible, which was subsequently kept in a muffle furnace at 575°C for 4 hours. After 4 hours, the crucible was removed, placed in a desiccator to cool, and then weighed. The acid-insoluble lignin was then calculated using the following method.

$$AIL\ (\%) = \frac{W_{dry} - W_a}{ODW} \times 100 \quad (5)$$

Where,

AIL= Acid insoluble lignin (%)
$W_{dry}$ = Weight of dry material in crucible remained after acid soluble determination, g
$W_a$ = Weight of ash after sample taken out from muffle furnace, g

ODW= Oven dry weight of initial sample taken, g

The total lignin content of rice husk was calculated by adding acid soluble and acid insoluble lignin;

$$\text{Total lignin } (\%) = \text{ASL}(\%) + AIL\ (\%) \qquad (6)$$

### 2.1.5 Determination of cellulose and hemicellulose

The cellulose and hemicellulose content of rice husk was evaluated using the Technical Association of Pulp and Paper Industry (TAPPI) method as described by De Oliveira et al. [22] and Zheng et al. [23]. A 2-gram sample of rice husk powder was weighed and transferred to a 250 mL conical flask. Then, 25 mL of a nitric acid/ethanol solution (1:4 by volume) was added to the flask. The mixture was refluxed for 1 hour, and this process was repeated several times until the sample turned white. The powder was then repeatedly washed with distilled water and filtered until the pH became neutral. The resulting residue was dried at 105°C. The α-cellulose content (C, %) was then calculated using the following method.

$$C = \frac{RH}{RH_1(1-W)} \times 100 \qquad (7)$$

Where,

RH= Weight of remained residue, g
$RH_1$= Weight of rice husk sample, g
W= Moisture content of rice husk in %
The hemicellulose of rice husk was calculated by the following equation;

$$HC\ (\%) = (100 - A - E - TL - C) \times 100] \qquad (8)$$

Where,

HC = Hemicellulose (%)
A= Ash content of rice husk (%)
E = Extractives (%)
TL = Total lignin (%)
C = Cellulose (%)

## 3. RESULTS AND DISCUSSION

The chemical composition of the lignocellulosic fibers are the most important variables that determine the overall properties of the fibers [24]. Initial composition of rice husk was determined by the NREL standard methods. The initial composition (initial moisture content, ash, extractives, lignin content, hemicellulose and cellulose) of rice husk were calculated in triplicate and mean value is reported in Table 1. The obtained results were in accordance with previous studies. The differences in composition are due to climate and geographical conditions, weather conditions and difference in varieties and type of soil etc.

### 3.1 Moisture Content

Moisture content indicates the amount of water present in biomass, expressed as a percentage of the material's weight. It significantly affects not only the harvesting and preparation stages but also the transport, storage, processing, and quality of the final products [25]. The initial moisture content of rice husk was 9.884 %.

### 3.2 Ash

Biomass ash primarily consists of inorganic compounds rich in alkali and alkaline earth metals. It is a significant cause of abrasive wear in biomass processing equipment, particularly in size-reduction machinery where biomass and tools interact at high velocities. The ash content in natural fibers can also affect the properties of composites [26]. The ash content of rice husk was found to be 15 %. These results are in lined with previous studies as mentioned in Table 1.

**Table 1. Initial composition of rice husk and comparison with previous studies**

| S. No. | Constituents | Average (% weight of dry matter) | | | |
|---|---|---|---|---|---|
| | | Current study | Previous Studies | | |
| | | | Johar et al. [27] | Nikzad et al. [28] | Bisht et al. [29] |
| 1. | Moisture Content | 9.884 ±0.56 | N/A | N/A | N/A |
| 2. | Extractives | 8.077±0.27 | N/A | 11.9 | N/A |
| 3. | Total Lignin | 17.210 ± 0.69 | 23 | 19.22 | 20 |
| 4. | Hemicellulose | 21.073 ± 0.62 | 33 | 15.24 | 25 |
| 5. | Cellulose | 38.640 ± 0.94 | 35 | 37.55 | 35 |
| 6. | Ash | 15.0 ±0.87 | 25 | 16 | 17 |

### 3.3 Extractives

The extractives in lignocellulosic biomass primarily include waxes, fats, resins, tannins, sugars, starches, and pigments. Extractives can interfere with component analysis, particularly lignin. Therefore, lignocelluloses with high extractive contents should be extracted first to avoid obtaining unacceptable results. In present study, extractives in rice husk were 8.077%. Nikzad et al. [28] also reported 11.9% extractives in rice husk.

### 3.4 Total Lignin

Lignin contributes to hydrophobicity and structural rigidity by binding hemicelluloses to cellulose in the cell wall. Also, lignin negatively impacts the conversion of cellulose. This effect is influenced by several factors, including the total lignin content, lignin composition, and structure, particularly the hydroxyl group content [30]. The total lignin content in rice husk was 17.21%. According to literature, lignocellulosic biomass comprises approximately 35%–55% cellulose, 25%–40% hemicellulose, and 15%–25% lignin.

### 3.5 Hemicellulose

Hemicelluloses are considered the second most abundant renewable component of lignocellulosic biomass, after cellulose. The content and structure of hemicellulose, including the length and type of the main chain as well as the distribution and type of side chains, changes depending on the variety of lignocellulose [31]. The hemicellulose content in rice husk was 21.973 %. Bisht et al. [29] also reported 25 % hemicellulose content in rice husk.

### 3.6 Cellulose

Cellulose is one of the leading constituents of lignocellulosic biomass. The results obtained in this study, showed that cellulose content in rice husk is 38%. The results are in lined with previous studies [27]. The composition of the lignocellulose components varies considerably between different plants. Jung et al. [32] compared the cellulose, hemicellulose, and lignin content in the stems and leaves of Miscanthus, switchgrass, reed, and sorghum and reported that the woodier reed (Phragmites australis) contains significantly more lignin than Miscanthus, switchgrass, or sorghum. In general, stalks typically contain higher concentrations of lignin, cellulose, and hemicellulose compared to leaves [33].

## 4. CONCLUSION

In the present study, the compositional analysis of rice husk was conducted. The lignin, hemicellulose and cellulose content in rice husk was found to be 17.210 ± 0.69 %, 21.073 ± 0.62 % and 38.640 ± 0.94 % respectively. These findings indicate that rice husk is an excellent source of cellulose. The extraction of cellulose from rice husk will not only provide a sustainable alternative to traditional cellulose sources but will also contribute to waste reduction and environmental conservation. Besides this, lignocellulosic biomass or agricultural waste can be converted into various value- added products such as bioenergy, biofuels, biochemical and building material. Moreover, these can be used as reinforcement for packaging applications. Understanding the physicochemical properties of biomass is crucial before designing any thermochemical or biochemical processing method, as these properties strongly impact the conversion efficiency of the process.

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## ACKNOWLEDGEMENTS

The authors express their gratitude to TEQIP-III, World bank, implemented by the College of Technology, GBPUAT, Pantnagar, India, for providing the essential financial support for this work.

## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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