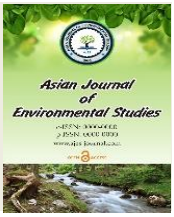
Asian Journal of Environmental Studies 1 (1) 08-13, (2023)

Available on: https://www.ajes-journal.com
Asian Journal of Environmental Studies (AJES)
e-ISSN: xxxx-xxxx, p-ISSN: xxxx-xxxx



Valorization of lignin extraction from pretreatment methods for exploitation of lignocellulosic bio resources to biofuel

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ARTICLE INFO

Article History:
Received: 19-10-2023
Revised: 14-12-2023
Accepted: 15-12-2023
Online: 31-12-2023

Editor: Dr. Zahid Naeem Qaisrani

Keywords:
1 Lignin Extraction
2 Pretreatment
3 Lignin Characterization
4 Biomass
5 Bio-fuel Production

ABSTRACT

Lignocellulose is considered as the most prevalent material that can be transformed into biofuels and other biological products. It is essential for the development of connective tissues because it confers stiffness, strength and resistance to external stimuli like infections. Lignin has a variety of economic applications, and its extraction might lead to the development of several novel applications. Therefore, it is of the most significant to develop efficient and long-lasting solution for lignin extraction from biomass. For this reason, one of the objectives of the current study is to examine several ways of lignin extraction from biomass such as “Rice Husk” & “Wheat Husk”. Following pretreatment, lignin was characterized by proximate and ultimate analysis, FTIR, and TGA. In comparison to other biomasses, wheat husk had the highest concentration of lignin, whereas rice husk had the lowest amount of Sulphur. Wheat husk for 4 hours yields 12% lignin and a minimum yield of 11%. A group of intricate organic polymers known as lignin serves as important structural components in the support tissues of vascular plants. The economics of producing biofuel from lignin could be greatly improved by its conversion into value-added products.

1. INTRODUCTION

environment's security and the viability of the economy. Renewable biofuels are viewed as a viable alternative and the ideal fuel additive for petrol (Jatoi, Abbasi, et al., 2023). A group of intricate organic polymers known as lignin serves as a crucial structural component of the tissues that support vascular plants and some algae. Lignin is the second-largest polymer on earth after cellulose and is commonly present in plants (Siddique, Soomro, Aziz, et al., 2021). The economics of producing biodiesel could be greatly enhanced by the conversion of lignin into value-added goods. Lignin has a variety of economic applications, and its extraction might lead to development of several novel applications. Therefore, it is of the most significant to develop efficient and long-lasting solution for lignin extraction from biomass (Siddique, Soomro, & Aziz, 2021). For this reason, one of the objectives of the current study is to examine several ways of lignin extraction from biomass, Like “Rice Husk” & “Wheat Husk”. Biomass to biofuel conversion has received more attention and is regarded as an original strategy. In order to produce biofuel, a variety of raw materials has been used as feedstock, depending on the cost-effectiveness, availability, and location of biomass (Kiełbasa et al., 2022). Lignocellulosic biomass has drawn the interest of numerous researchers from all over the world among the various raw resources. The potential use of lignocellulosic biomass for the creation of biofuel is discovered in the current review (Siddique et al., 2022). The rate of biomass hydrolysis and accessibility have been significantly increased by the application of various pretreatment techniques. This analysis focuses on current developments in pretreatment techniques for increased biofuel output. The mechanism of the biomass-processing pathway, optimization, and modelling studies have all been described in detail (Siddique, Soomro, Ahmad, et al., 2021). Lignin has many economic applications, and its extraction might lead to the development of several novel applications. Therefore, it is very important to develop a prominent and everlasting solution for lignin extraction from biomass (Ulakpa et al., 2023). For this reason, one of the objectives of the current research is to examine several ways of lignin extraction from biomass (Jatoi, Ahmed, et al., 2023). Since lignin is produced as a waste product in many industries, however, it has many useful applications. Hence, it should be necessary to save lignin from being wasted and to use it in production of useful products (Kumar et al., 2023). Biomass to biofuel conversion has received more attention and is regarded as an original strategy (Zahid et al., 2016). In order to produce biofuel, a variety of raw materials have been used as feedstock, depending on the cost-effectiveness, availability, and location of biomass (Sheng et al., 2021). This study also updated modern technology regarding the pretreatments that make it possible to produce very useful nanomaterials. Thus, an achievable strategy is put up for the efficient and environmentally friendly joint production of nanomaterials and biofuels to fully utilize lignocellulose (Mushtaq et al., 2017).

2.0 MATERIALS AND METHODS

2.1 MATERIALS

Materials, which are being, selected (Wheat Husk & Rice Husk) for the research are claimed to be easily available in Pakistan. In biomasses mentioned before the composition of lignin is 18.6% in “Wheat Husk” and 25% in “Rice Husk”. No difficulties and hard milestones will be generated during the collection of such biomasses. They are widely available in every city of Pakistan. They can be easy to store and easy to handle. Industries will not get into any kind of trouble while examining such biomasses.

Figure 1 Wheat Husk (Sample)

Figure 2 Rice Husk (Sample)

2.2 METHODS

There are various methods for the extraction of lignin from different biomasses, some of them are more effective and some of them are less effective, some have good results, and some have not so good results so far. The efficient method can be selected based on the basic requirements from the researchers and availability of suitable equipment. The suitable method can also be selected on the basis of physical and chemical properties of reactants which are being used in certain experimentation. Selection of prominent methodology is the primary step of researchers because all the further steps are dependent on this step. The results and effectiveness can be positioned on the basis of purity or lignin extracted by using suitable methods and techniques. Some of the common methods by which lignin can be extracted from different biomasses for the production of useful products are listed below. These methods are used by many researchers around the world to reach their objectives and to gain premium quality results, the methods are.

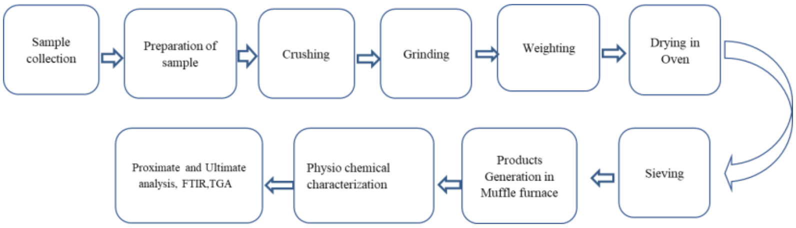

Figure 1 Research Methodology for experimental setup

2.2.1 LIGNIN EXTRACTION FROM BIOMASS

The purpose of this research was to extract lignin from various biomasses. A solution of two weight percent (W/V) NaOH was used. The temperature of the extraction was a key factor for greater lignin yield recovery, so for each experiment, 20 grams of NaOH was taken and combined with 1000 ml of distilled water and 50 grams of biomass. The mixtures were then dried for 2, 3, and 4 hours, respectively, in a drying oven at 100 degrees Celsius in order to calculate the yield of both biomasses at different times. After blending the alcohol solution with a mechanical stirrer, the mixture was kept in a dry oven with Rice Husk and Wheat Husk. (ratio of 1:15). Then, everything was put to the beaker, and the mixture was filtered using filter paper to take out the water and extract the lignin from the combined liquid on the filter paper, recovering the pretreatment biomass. Both tests were run sequentially after being washed with distilled water to remove excess alkali. We then drained the water, collected, and dried the residue using filter paper. The aqueous filtrate was acidified with concentrated sulfuric acid using a pipette and a pH meter, first with sulfuric acid and then by adding acid to bring the pH down to 1. Following an hour of boiling, the mixture was cooled, and the solid residue rinsed with distilled water. When pH 7 was obtained, undesired materials were washed with distilled water and impurities were eliminated before drying. This precipitate was separated by filtering with filter paper and then washing with distilled water to remove the lignin residue. The lignin samples were dried at 110°C in a drying oven for further processing. In this investigation, sodium hydroxide (NaOH) and sulfuric acid (H2SO4) were the substances used. The extracted lignin was then quantified, filtered, and placed in sealed containers for further analysis. Table 1 shows the yield percentage at 1000C of both biomasses when reacted with 2% sodium hydroxide (NaOH) under biomass liquor ratio of 1:15 shown respectively.

Figure 2 Extracted Lignin from Rice Husk

Figure 3 Extracted Lignin from Wheat Husk

3 RESULTS AND DISCUSSIONS

The main aim of this research was to focus on the efficient method of extraction of lignin from different biomasses by using such reactants, which are readily available across the country. Hence, we used “Rice husk” and “Wheat husk”, and the results were quite satisfying. According to our studies “Chemical and thermal Pretreatment” is considered to be the most efficient way of extraction of lignin from other biomasses as compared to any other pretreatment method. It can be done at laboratory level. Equipment involved in this pretreatment method can be managed easily. It is less costly as compared to other pretreatment methods. It gives results that are more prominent. The observations and results which we have taken in our research are mentioned in (Fig 1, 2, 3, 4, 5, 6, 7,8,9) and (Table 1, 2, 3) respectively. Hence, the final product, i.e. lignin catalyst will be used to produce biodiesel etc.

3.1 THERMOGRAVIMETRIC ANALYSIS (TGA)

TGA (Thermogravimetric Analysis) is widely used to study the thermal properties and behavior of lignin, a complex and heterogeneous polymer derived from plant biomass. The main purposes of performing TGA analysis for lignin include:

3.1.1 THERMAL STABILITY ASSESSMENT

TGA helps determine the temperature range at which lignin undergoes decomposition or degradation. By measuring the weight loss of lignin as a function of temperature, TGA can identify the onset temperature and extent of thermal degradation. This information is crucial for understanding the thermal stability of lignin and its potential applications in various industries.

3.1.2 PROCESS OPTIMIZATION AND QUALITY CONTROL

TGA can assist in optimizing lignin extraction, purification, and processing methods. By monitoring the weight loss or residue formation during thermal treatment, it is possible to assess the effects of processing parameters on lignin properties. TGA can also be used for quality control purposes to ensure consistent lignin characteristics and identify any variations or impurities in lignin samples. TGA allows for the comparison of thermal properties among different types of lignin obtained from various sources rice husk and wheat husk or through different extraction methods. It can help evaluate the influence of factors such as lignin source, isolation technique, and purification on the thermal stability and decomposition behavior of lignin samples (Watkins et al., 2015). Overall, TGA analysis plays a crucial role in understanding the thermal properties, stability, and behavior of lignin. It provides valuable information for lignin characterization, process optimization, and the development of lignin-based materials in various industries, including bioenergy, biopolymers, composites, and pharmaceuticals.

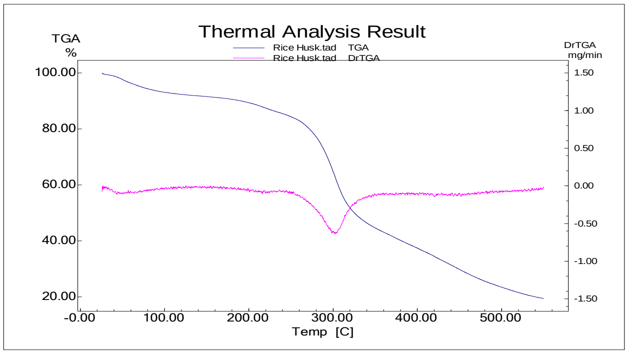

Figure 4 Thermal Analysis of WH

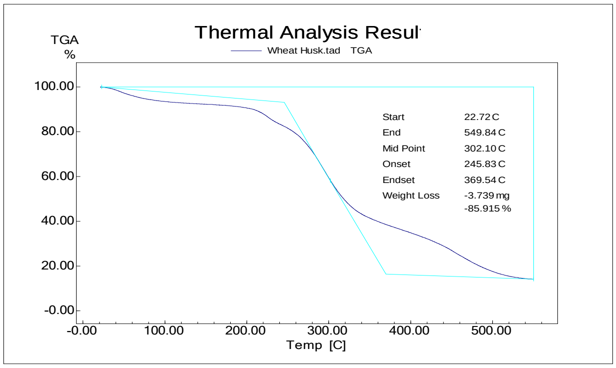

Figure 5 Thermal Analysis of RH

Table 1 Lignin yield at 100 °C for both biomass at different time intervals

Sample	Temp (°C)	Time (h)	Liquid Solvent conc. (2% NaOH) ratio	Biomass Liquor ratio	Stirrer speed	Yield (wt.%)
RH	100	2	2%	1:15	Const.	8
RH		3				9
RH		4				11
WH		2				9
WH		3				10
WH		4				12

*RH: Rice Husk, WH: Wheat Husk

3.2 PROXIMATE ANALYSIS

Proximate analysis is used to estimate Carbon (%), Hydrogen (%), Nitrogen (%), and Sulphur (%) within the lignin being tested. The results are shown in Table 2 and Figure 6.

Table 2 Proximate Analysis of Lignin Samples

Biomass	MC (%)	VM (%)	AC (%)	FC (%)
WH	2.08	75.79	1.84	16
RH	3.82	76..97	2.78	15.6

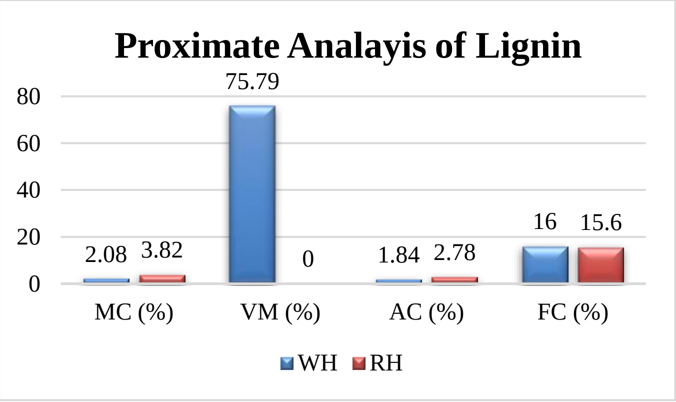

Figure 6 Proximate Analysis of Lignin

3.3 ULTIMATE ANALYSIS

The ultimate analysis of lignin refers to the determination of its elemental composition, typically including carbon, hydrogen, nitrogen, sulfur, and oxygen. The purpose of performing ultimate analysis on lignin includes:

Table 3Ultimate Analysis of Lignin

Biomass	C (%)	H (%)	N (%)	S (%)	O (%)
WH	43.2	5	3.48	0.36	39.4
RH	39.98	2.45	4.43	0.53	52.61

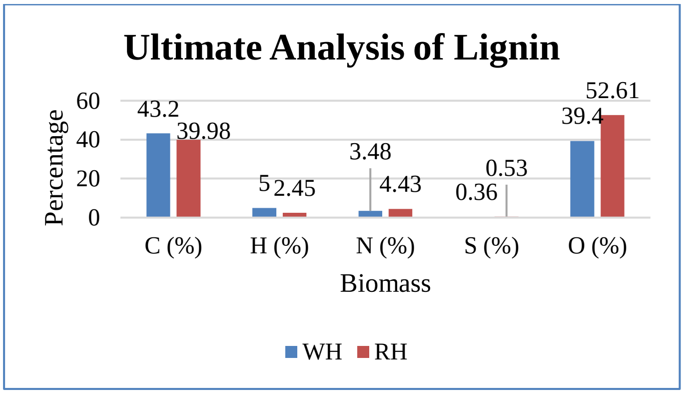

Figure 7 Ultimate Analysis of Lignin

done to study the physical properties of the final product. Weather the extracted product is lignin or not! Steps include in doing characterization are listed below.

3.4 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

The FTIR spectrum of lignin extracted from wheat husk & rice husk generally exhibits similar features to lignin from other sources. Here are some key peaks commonly observed:

- The broad peak around 3400-3200 cm-1 corresponds to the stretching vibrations of O-H bonds, indicating the presence of hydroxyl groups in lignin.
- The peak around 2920-2850 cm-1 represents the stretching vibrations of C-H bonds in the aliphatic (saturated) regions of the lignin structure.
- The peak around 1720-1600 cm-1 indicates the presence of carbonyl groups (C=O) and aromatic skeletal vibrations in the lignin structure.
- The peak around 1500-1420 cm-1 corresponds to the aromatic C=C stretching vibrations, indicating the presence of aromatic rings in lignin.
- The peak around 1260-1230 cm-1 represents the syringyl (S) and guaiacyl (G) ring breathing vibrations in lignin (Sowmya Dhanalakshmi & Madhu, 2021).

It is important to note that the specific positions and intensities of these peaks can vary depending on the extraction method, processing conditions, and the specific characteristics of the biomass used.

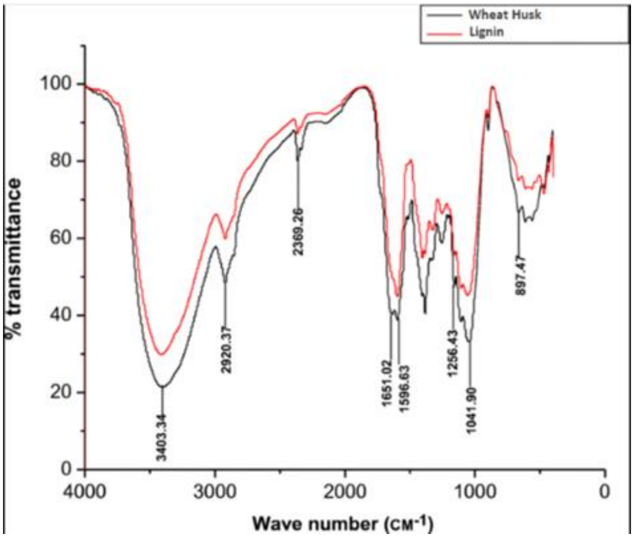

Figure 8 FTIR for WH (Before and after Pretreatment)

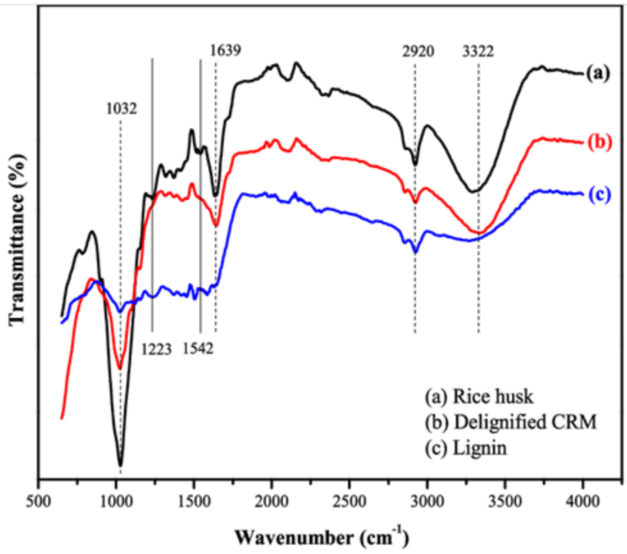

Figure 9 FTIR for RH (Before and after Pretreatment)

CONCLUSIONS

The current work provides information on lignin extraction from biomass resources made of wheat and rice husks. Additionally, extracted lignin rice and wheat husk from biomass has positive yields compared to wheat husk lignin. It was concluded that lignocellulose feedstock was influenced by a change in time, temperature for extraction of lignin yield. Rice husk for 4 hours achieve less yield of lignin 11 % as well as wheat husk achieve more yield of lignin, which is 12%. Finally, it was decided that acid, as well as alkali, were utilized for the process. The extracted material was then characterized by using FTIR and TGA. Thus, extracted lignin recovery from biomass, which is by alkaline pretreatment, was more beneficial in commercial and domestic utility as a polymeric material. It is biodegradable and biocompatible lignin used in medicine, composite material, and wood. The alkaline treatment was capable of removing lignin from the rice husk lignin, and wheat husk biomass according to the instrumental analysis finding. However, a small portion of the impurities of sugar is available in biomass-derived lignin. This can be removed by the use of acid treatment.

ACKNOWLEDGEMENTS

The authors acknowledge Coal resource and research laboratory, at MUET Jamshoro.

CONFLICT OF INETEREST

No competing interests are disclosed by the authors.

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