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Research Article

Extraction and characterization of lignocellulosic agricultural biomass pretreatment method for biofuel

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ABSTRACT

Renewable resources have immense potential, as a cost-effective and sustainable energy alternative. Lignin is the most abundant organic material after cellulose and aromatic biopolymer. A total of four distinct raw materials, including lignocellulosic biomass from almond shells, walnut biomass shells, barks of babul tree, and bark of neem tree, were utilized to extract lignin. Following their pretreatment, proximate, ultimate analyses, EDS, and SEM techniques were used to describe the biomass and extracted lignin. Substrates like walnut shell, and babul tree bark were projected to have the highest concentration of lignin recovery when compared to other biomasses, while neem tree bark and almond shell had the lowest carbon content compared to neem tree bark. By laboratory experiment, we observed that walnut shell yield is 13 %, which is compared with BTB extracted lignin yield 11 % obtained. This report discusses the general biofuel usage scenario in Pakistan.The financial benefits of switching from accustomed fuels to biofuels. Thus, yield extraction with this technique can benefit society financially and reduce the need to dispose of biomass in the future.

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INTRODUCTION

Growing awareness of how susceptible and unsustainable the current production methods for energy production and resource depletion led to an increase in concern and understanding. It is recognized that the spread of renewable-based production systems and the diversity of resources have greatly aided the transition to a more resilient and robust energy production system constructed by the principles of the bioeconomy [1]. To meet the rising global energy demand, renewable energy's relevance in the generation sector is also crucial. Additionally, there is little

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doubt that the market for renewable energy has been dominated by [2]. Proper biomass consumption can be used in conjunction with optimal chemical processes to develop high-value commodity items. It is critical to establish plant financial potential as a viable solution to a salinity issue. The biomass materials that are utilized frequently to produce liquid biofuel include (NTB), (BTB), (WNS), and (AS). Additionally, we used various criteria to remove the lignin from this biomass [3]. The majority of lignocellulose biomass is made up of plant cell walls, which are responsible for the woody tissue of plants and offer strength and durability. The majority of the cell wall is composed of lignin, cellulose, and hemicellulose and is arranged into a complex structure. The typical dry weight percentages of cellulose, are 20-35 percent hemicellulose, and 5-30 percent, depending on the biomass type, source, and even harvesting time. The concentration of hemicellulose in crop leftovers is higher [4]. The word "biorefinery" refers to a process that integrates numerous conversion processes, including as biochemical, thermochemical, and energy conversion processes, to produce chemicals, biofuels, and other high-value industrial products from LCB via microbial growth. Full-scale LCB bioconversion to bioenergy/biofuels (biodiesel, bioethanol, and biobutanol) and biobased commodities (chemicals, feed, and food) offers a number of significant energy input and output product challenges[5]. Numerous polymers, including cellulose, hemicellulose, and lignin, as well as extra-polar and non-polar components, combine to create the LCB heteropolysaccharide complex. However, investigations on the impact of biomass size distribution on the end products of fast pyrolysis have been completed. Due to the fibrous nature of biomass and the possibility for fine particles to produce a significant amount of biofuel, it has been discovered that biomass size reduction is an energy-intensive and challenging operation. Because of their limited output, coarse biomass particles are misunderstood [6]. Biofuels are currently giving long-term energy and economic strength all around the planet. Almond and walnut shells are expected to have a bigger part in energy production than wheat straw and corn. Biomass is readily available and inexpensive, ensuring optimum energy conversion. Ethanol is produced from lignocellulose sources as well. To improve selectivity and yield, this biochemical procedure necessitates preprocessing [7]. As a result, this biomass material was used in an experiment to generate ethanol using a disc-refiner. To disrupt the crystalline structure of biomass and enhance its susceptibility to enzyme hydrolysis, pretreatment targets the hydrogen bonds and cross-linked hydrophobic contacts between cellulose, hemicellulose, and lignin. It is possible to enhance accessibility, pore size, and surface area while lowering the degree of polymerization (DP) and crystallinity of cellulose with the correct pretreatment [8]. The major pretreatment techniques for LCB fractionation might be physical, chemical, physicochemical, and biological [9]. The bulk of these processes have several disadvantages, such as being costly, not being "green," creating inhibitors or toxic substances, and so on. As a result, current research is focused on developing cost-effective and environmentally friendly pretreatment strategies to produce viable LCB fuel technology [10]. There is no simple model available that includes PSD with size verification. Measures that can be used to investigate coal-fired power stations' air particulate-based pollution emissions. It is beneficial to measure the particle size distribution in a contaminated area [11]. To achieve bulk flow, densification, and homogeneity of feed flow, biomass must be processed in a knife mill. Following that, size reduction is critical to producing bioenergy with an energy-intensive unit operation. Meanwhile, densification has been implemented to reduce transportation costs [12]. Biorefineries are critical to the creation of a knowledge-driven and environmentally conscious circular bioeconomy. The lignocellulosic material used as a feedstock plays an important role in the production of bioproducts as well as the energy necessary to power them[13]. The concept of a circular bioeconomy places a strong emphasis on recycling all waste while emitting no new garbage.The circular economy strives to replace the end-of-life phase through resource recovery, the use of renewable energy, the removal of harmful substances, and the zero discharge concept by adjusting the design of all relevant systems and business models [14]. Conventional biorefineries use high degrees of process integration and low waste output to transform biomass into a range of products for diverse industrial sectors[15].However, it isn't specified explicitly that study papers specify total specific energy. Industrially, lignin comes in a range of grades with various isolation techniques. Kraft lignin, sulfite lignin, soda lignin, and organosolv lignin are some of the current industrial sources of lignin[16]. The type of lignin produced and its properties are determined by the pre-treatment process that is carried out on lignocellulose before lignin is isolated. By enhancing porosity, improving surface accessibility, and lowering overall recalcitrance, pre-treatment makes lignocellulose more suitable for subsequent processing[17]. Pre-treatment, processing, physicochemical, biological, and combinational techniques are all possible. The pre-treatment procedures are then followed by solvent and by-product recovery, which results in the creation of biomass derivatives including paper, bioethanol, high-quality lignin, etc[18]. The majority of the lignin types that have been highlighted are a direct result of well-known pulping techniques. Unfortunately, the methods for these forms of lignin typically include caustic solvents that are harmful to the environment, such as sodium hydroxide, sodium sulfide, different sulfites, soda-anthraquinone, etc[19]. Furthermore, the majority of methods produce lignin derivatives that are undesirable for specific material applications that demand higher purity. Ionic liquids, which are thought of as green solvents that can preserve the natural molecular structure of lignin during the processing of lignocellulosic biomass, are being investigated as an alternative in more recent studies[20]. Ionic liquids are typically regarded as environmentally favorable because of their great thermal stability, minimal volatility, and usefulness. The effectiveness of ionic liquid processing of biomass was examined, along with the impacts of biomass loading quantity and processing time[21], with a focus on lignin recovery. The success of the processing was determined by how much lignin was removed from the biomass. To determine how successfully the mechano-chemical process worked, the rheological behaviour of combinations of biomass and the ionic liquid was also examined. The extracted lignin samples were also carefully documented and evaluated against commercially available lignin products[22]. Based on the necessity to provide not only clean energy but also environmentally sound processes, this article's contribution reflects the presentation of trends and opinions on the environmental consequences of various chemical pretreatment technologies [23]. The depiction of the subject's trends and viewpoints is this article's contribution. Based on the necessity to provide not only clean energy but also environmentally sound processes, this article's contribution reflects the presentation of trends and opinions on the environmental consequences of various chemical pretreatment technologies[24]. Bagasse can be converted into non-energy products utilizing a variety of methods, including those that use physical, chemical, mechanical, and biological extraction [25]. The ideal treatment strategy must be selected based on the treatment's final goal. Using physical and chemical pretreatment procedures, it is possible to separate the components of biomass[26]. To make raw materials available for later treatment, such as chemical and/or biological treatment, they must first undergo physical pretreatment, which involves manually reducing their size. The hydrolysis process provides the foundation for the primary techniques used to separate biomass components[27]. This procedure, also known as saccharification, breaks the hydrogen bonds in the lignocellulosic biomass components by removing the surrounding hemicellulose and/or lignin and altering the cellulose microfibril structure. The soluble monomeric and/or oligomeric sugars that result are subsequently collected[28]. The use of lignin-based biomass as an energy storage medium and current trends in lignin extraction from lignocellulosic biomass are addressed. The many extraction methods for lignin, the impact of the extraction conditions, and the operational tools used to achieve lignin separation are discussed[29]. We looked at lignin-based biomass thermochemical modification for energy storage applications. It was thoroughly explained how activation circumstances affected the lignin's surface functional groups, texture, and ability to store energy. In particular, a case is made to intensify research into using biomasses based on lignin[30]. The drive to use lignin-based biomasses more often will provide insights on the best ways to produce high-density energy storage materials for quicker commercialization from a techno-economic, energy, and safety perspective[31]. The rubberwood lignin that was extracted as part of the experiment was described. The final objective of this work was to develop ethanol or propanol, which were considerably safer and easier to recover, to replace hazardous acidic or alkaline solvents[32].

MATERIALS AND METHODS

During the research investigation, two types of materials were obtained. The almonds and walnuts were taken from Loralai near Ziarat, Baluchistan, Pakistan, where they were purchased at a dry fruit shop. The samples' shells were removed using a hummer mill at the MUET chemical engineering department in Jamshoro, Pakistan.

Extraction of Lignin from Agricultural Biomass

A beaker with 1000 mL of distilled water already had 500 grams of biomass. In this procedure, NaOH was utilized. The sample underwent pretreatment and was kept at 110°C oven for three hours. Using filter paper, the pretreated solution was transferred to a beaker. Distilled water was used to clean the delignified solid. A magnetic stirrer

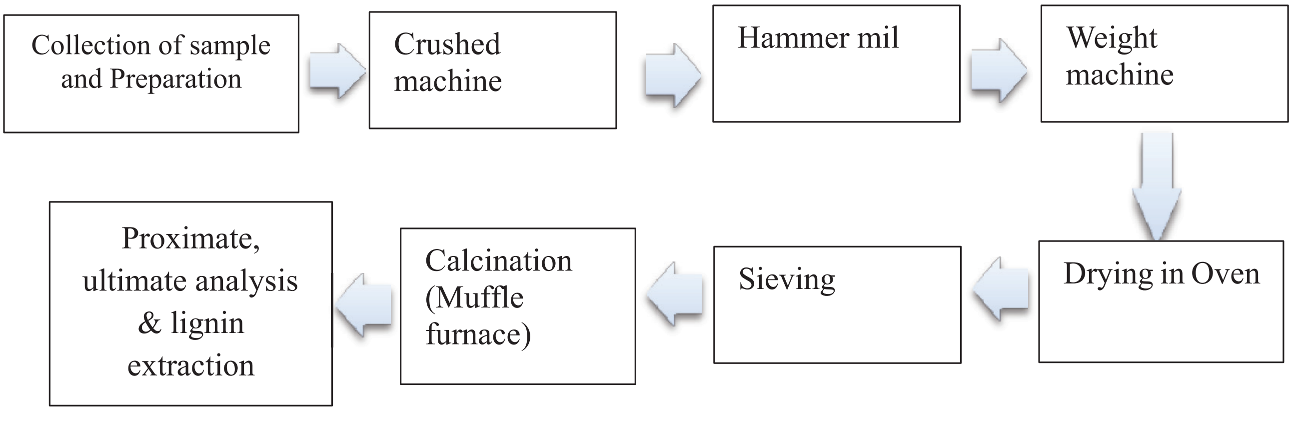


Figure 1. Description of a sieving-based particle size distribution approach.

was kept in the beaker for agitation while a magnetic hot plate heating stirrer was utilized for the solid-liquid ratio (1:20). Utilizing filter paper, the pretreated biomass lignin was extracted. To bring the pH level to 1, sulphuric acid was added dropwise with a pipette before precipitating the lignin. After separating the lignin residue, distilled water was utilized to dilute the mixture to the necessary neutral pH of seven. In the extraction procedure, lignin is to be separated. The lignin sample was then kept in an oven set at 105°C to dry out. The sample was dried before the extracted lignin was stored in plastic bags for analysis.

PROXIMATE ANALYSIS

Moisture Content

The dry oven test method (ASTM D2867-09) has been used to determine the different percentages of moisture content.

The sample was placed in an oven-dryer container and was weighted. The sample had been set up at a temperature that ranged from 130 to 145°C The material was taken out of the drying oven once drying was complete and allowed to cool to room temperature before undergoing a second weigh-in. As a result, using the difference, the moisture content of the sample might be calculated.

Analysis of Volatile Matter

Activated carbon's volatile matter content is determined using the Standard Test Procedure (ASTM D5832-98). In a weighed, covered crucible, one gram of the substance is heated for seven minutes at 950 °C. The difference is used to estimate how much volatile material is present after the sample has cooled to room temperature in a desiccator.

ASH Content

Using the muffle furnace test method (ASTM D 2866-94), ash content is assessed. A muffle furnace is used to hold the sample of 0.1g of activated carbon until it reaches a constant weight at a constant temperature of 650 ^{0}C.

The fixed carbon is then determined utilizing the formula that follows. After moisture, volatiles, and ash content are taken into account, fixed carbon is the quantity of carbon that remains from the overall percentage.

Ultimate Analysis of Biomass

The SC 832 LECO was used as the model, and the final analysis was run on a raw sample weighing 350 mg. To acquire the results on a dry basis, the biomass sample was dried at 110°C for 1 hour after sieving to a particle size of 150 microns. A more detailed study reveals the levels of elemental carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O) in the unprocessed BTB and NTB samples. Of all the necessary components, the babul tree contains the highest C and the lowest S.

Characterization of lignin using eds

The electron beam excites the atomic surfaces, causing the atoms to emit certain X-ray wavelengths that disclose the atomic structure of the elements. This technique is frequently used for computing the elemental compositions of small samples, such as those under a few cubic micrometers (ECM). An energy-dispersive detector, a solid-state device that can discriminate between various X-ray energies, analyses the X-ray emissions. The atomic composition of the specimen's surface is then revealed by the subsequent assignment of the correct elements.

EDS equipment can be used to detect the presence of lignin. EDS, also referred to as energy-dispersive X-ray spectroscopy, is a method for analyzing a material's surface composition. This method was used to identify the components of the lignin sample taken from biomass. The walnut shell, almond shell, neem tree bark, and babul tree bark were the four samples that were tested.

SEM Analysis of biomass lignin SEM is an imaging technique that is used to examine the surface morphology of a variety of materials, including lignin, a sophisticated organic polymer included in biomass cell walls. SEM analysis is a useful tool in the study of lignin.

RESULTS AND DISCUSSION

In current research, both preliminary and final analyses of the feedstock were performed. Tables 1 and 2 display tabular results of the experiments. The proximate analysis uses specialized techniques to determine the amounts of ash, volatile matter, fixed carbon, and moisture. The amount of biomass that burns as inorganic waste (ash), volatile matter, and solid-state (fixed carbon) can be calculated by carefully studying the biomass. In this lab, bark from babul and neem trees is acceptable as biomass. The results of the final

Table 1. Proximate analysis of biomass sample

Biomass sample	Moisture content percent	% of Volatile Matter	% of Ash Content	% of Fixed carbon
WNS	3.2	77.89	1.89	16.12
AS	5.72	75.81	1.71	16.79
BTB	4.11	76.08	1.98	19.00
NTB	5.22	69.81	3.97	16.15

Table 2. Final examination of raw biomass samples that have been pre-treated (presented on a dry basis)

Feedstock	Carbon(%)	Hydrogen(%)	Nitrogen(%)	Sulfur(%)	Oxygen(%)
WNS	48.1	6.2	0.76	0.35	43
AS	46.20	6.54	0.88	0.27	45
NTB	52.80	4.6	0.64	0.17	41
BTB	54.70	4.3	0.19	0.14	38

Table 3. The lignin yield from the barks of Babul and Neem trees at 100 °C during various periods

Material type	Temperature (°C)	Time (hr.)	Liquid to solvent concentration percent	Biomass / liquor ratio	Speed of stirrer	(% of Yield wt.)
BTB	100	2	2%	1:20	Constant	11
		3				9
		4				7
NTB		2				10
		3				8
		4				7

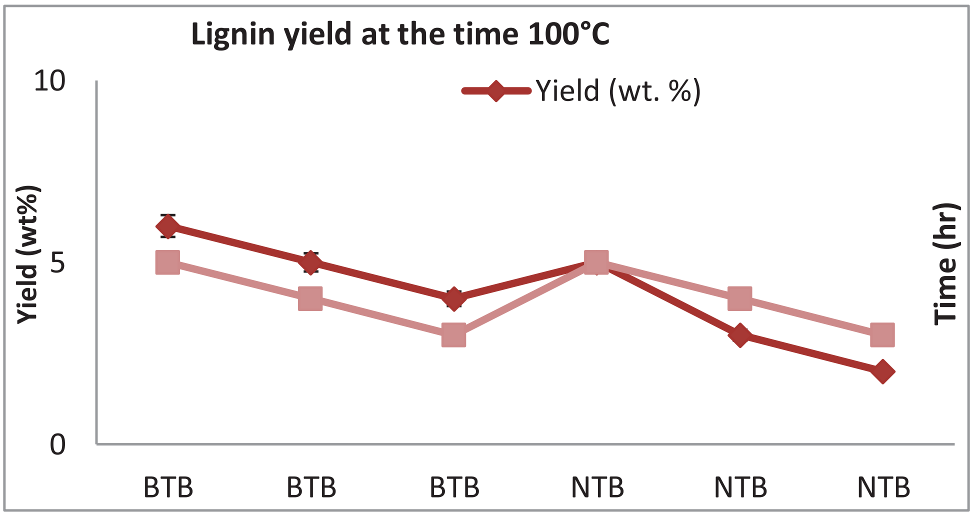


Figure 2. Extraction of lignin and its yield.

Table 4. Yield of lignin for WNS, AS at temperature 130 °C

Material type	Temperature (°C)	Time (in hr.)	Liquid to Solvent concentration percent	Biomass /Liquor ratio	Speed stirrer	(% Yield wt.)
WNS	130	2	2%	1:20	Constant	13
		3				9
		4				7
AS		2				12
		3				10
		4				8

analysis of the methods used to calculate the percentages of C, H, N, S, and O are shown in Table 2.

When compared with NTB, BTB contains more C. The C content raises the heating value. While nitrogen will raise the quantity of NOx, sulfur will increase the amount of SOx in the atmosphere. Here, laboratory testing was done on the four biomass samples, BTB and NTB,WNS,AS. Biomasses were handled by the alkali technique. Based on these results, Table 1 shows that lignin extraction was successful in identifying the relative concentrations of moisture, volatile matter, ash, and fixed carbon in each biomass. A good yield is 11% BTB yield for 2 hours at 100°C, as shown in Table 3. NTB, on the other hand, calls for a minimum content yield of 14%.

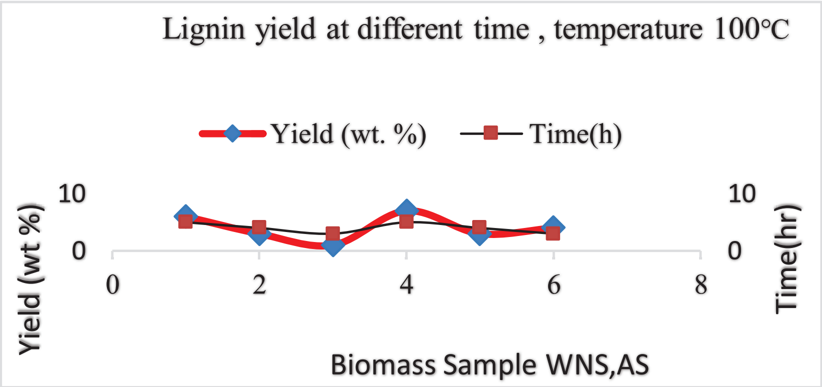
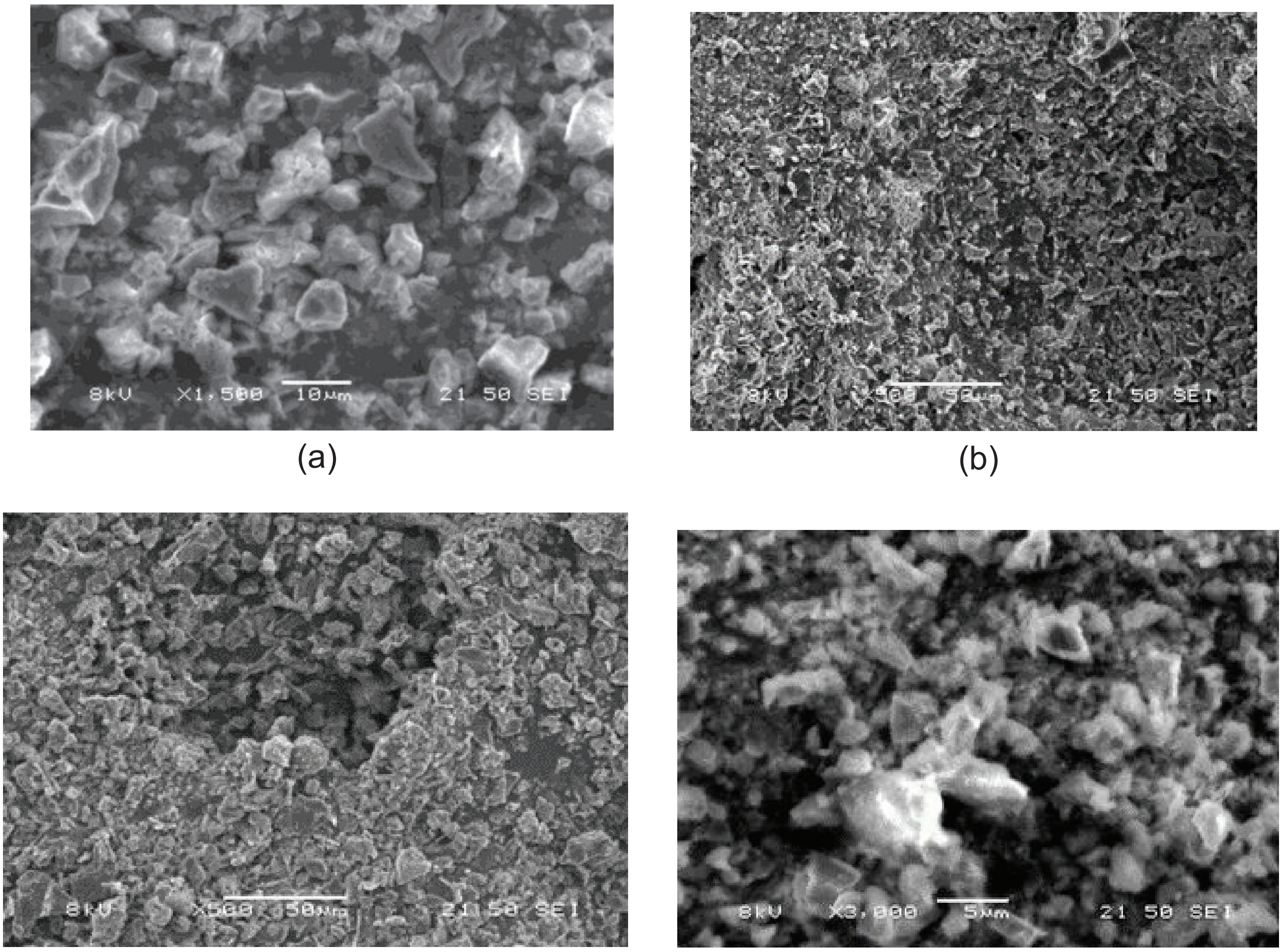


Figure 3. Extraction of lignin and yield.



(a) (b)

(c) (d)

Figure 4. SEM Analysis of lignin (a) Bark of Neem Tree and (b) Bark of Babul Tree (c)Walnut shell (d) Almond shell.

Figures 2 and 3. show the extraction of lignin from various biomass-to-liquid ratios together with the yield of lignin and the duration of the process.

Using SEM, Figure 4 depicts lignin in several substrates. The SEM produces a high-resolution image of a lignin sample surface using an electron beam. To analyze the surface morphology of the materials, SEM and an electron microscope were employed. For acylating agent erosion and side chain length both simultaneously contributed to the surface layer.

The thickness of some elements present in the NTB and BTB, WNS, and AS, such as cell walls, fibers, and structures, may have been measured in this instance by the SEM examination. The 5 m represents the features' average.

However, as the EDS graphical representation of Figures 5, 6, 7 and 8 demonstrates, the bands at 2937 and 2843 cm^{-1} which are mostly caused by CH stretching in aromatic methoxyl groups such as, in methyl and methylene groups on side chains. The entire lignin bandwidth, which spans from 3410 to 3460 cm^{-1}, is caused by the hydroxyl groups found in phenolic and aliphatic structures. In this area, the lignins of hemp, jute, and flax form distinct bands with maxima at 2917 and 2847 cm^{-1}. These peaks are from aliphatic methylene groups that have been stretched with CH.

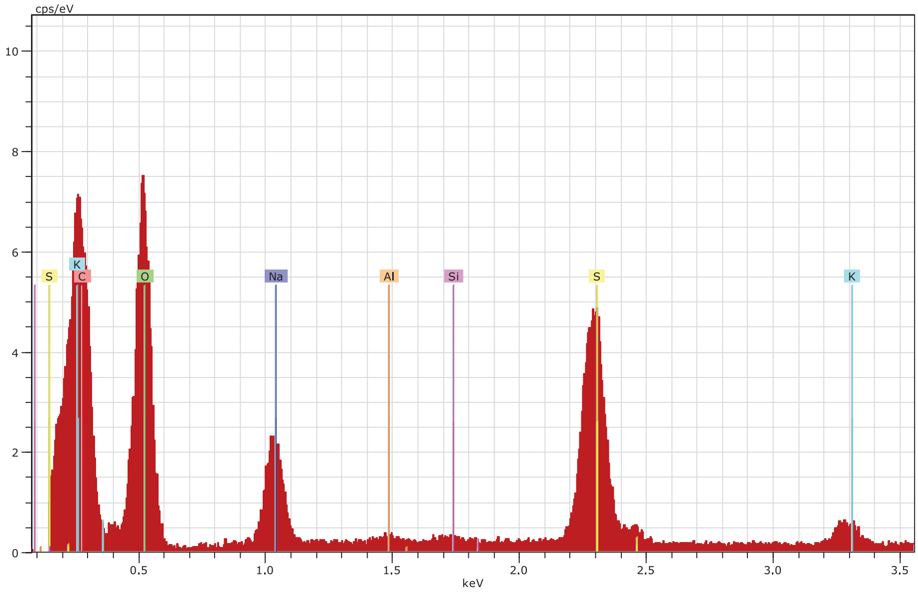


Figure 5. Walnut shell Lignin Biomass.

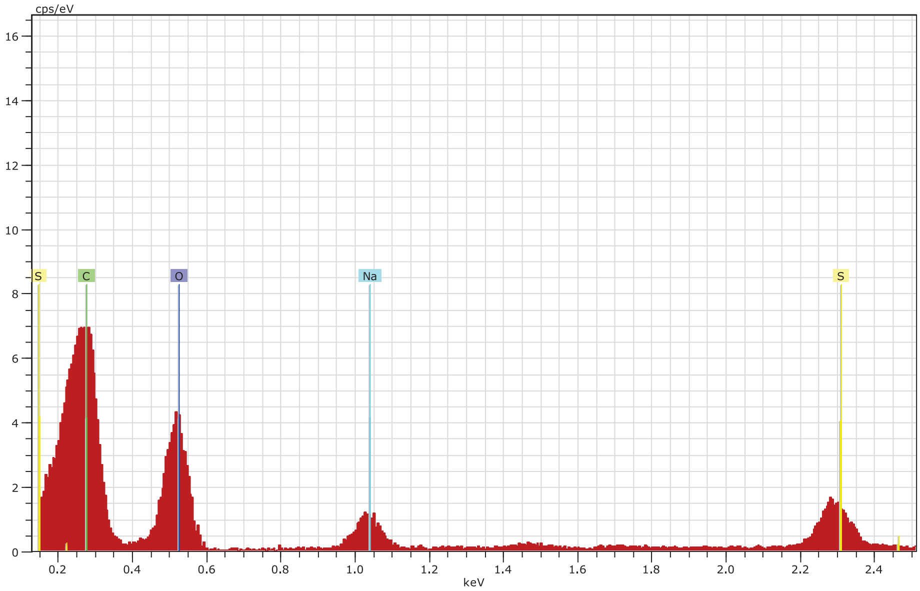


Figure 6. Almond shell Lignin Biomass.

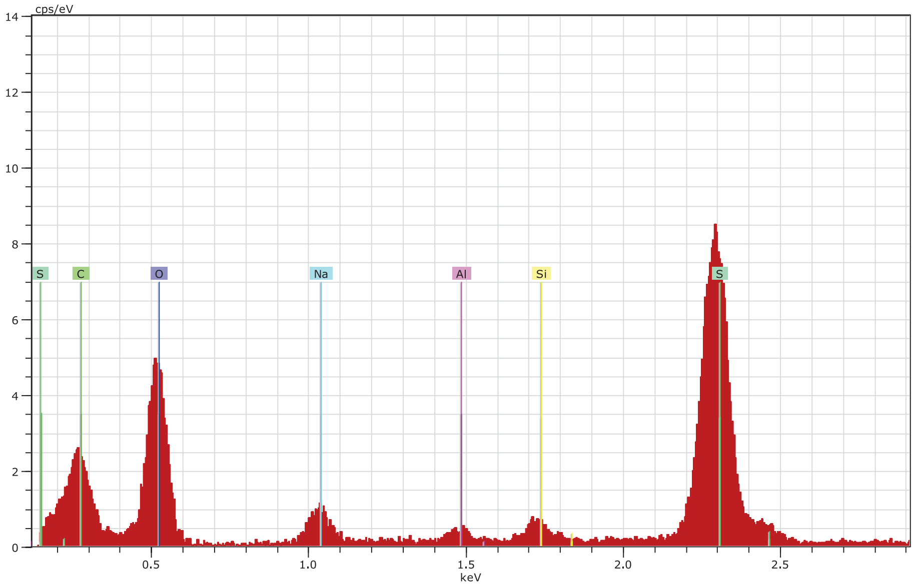


Figure 7. Neem tree bark Lignin Biomass.

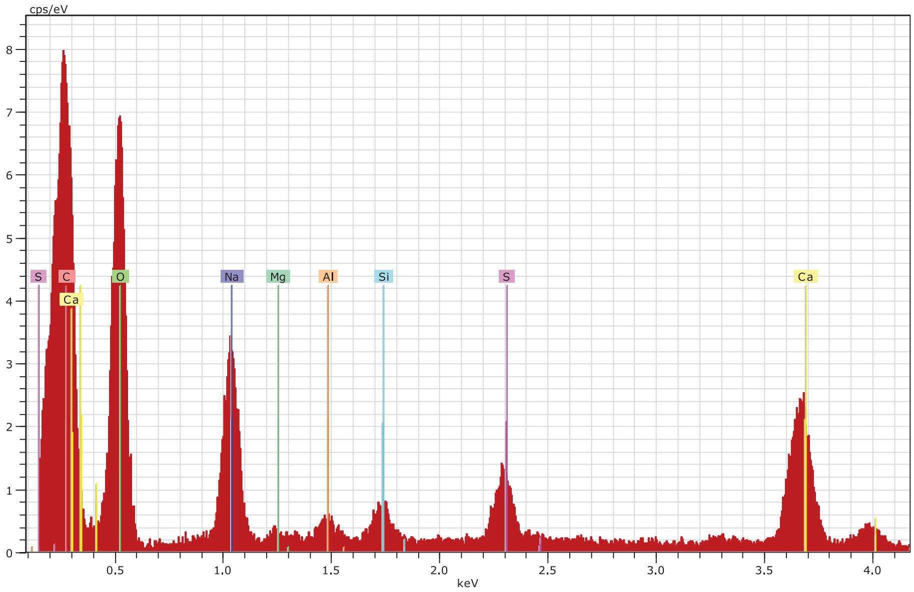


Figure 8. Babul tree bark Lignin Biomass.

This is emphasized here because these bands can be used because of the fatty acids included in lignin preparations. For example, Figure 7 shows a faint for the medium bands resulting from unconjugated carbonyl/carboxyl stretching between 1705 and 1720 cm^{-1}. The cause of a shoulder at 1680 cm^{-1} is most like conjugated carbonyl-carboxyl stretching. However,the band at 1705 cm^{-1} widens for sulfur-free softwood lignin, solvent-extracted from hardwood lignin, and oxidized lignins (flaxox), in contrast to Kraft lignin and softwood lignosulfonates. Impurities such as water, lignin, and proteins can provide weak absorptions at 1650 cm^{-1}. The weak absorptions at 1650 cm^{-1} cause stronger bands at 1705 and 1600 cm^{-1} to asymmetrically amplify.

CONCLUSION

The current work involves extracting lignin from some biomass sources, including walnut, almond, neem, and babul tree bark. Although the polymeric substance made from the lignin recovered from biomass by alkaline pretreatment was more useful for commercial and household usage. It was determined that changes in yield, lignin extraction temperature, and time had an impact on lignocellulose feedstock. However, WNS's excellent lignin yield was high at 13 % when the time was 2 hours as well and BTB had a lignin production at 2 hours of 11% yield. Additionally, lignin derived from biomass that has been extracted using babul tree bark as well as walnut shell is higher in yield, whereas lignin outputs from neem tree bark and almond shell are at a minimum. It was determined that alkali and acid were employed in this procedure so that the pretreatment process would examine the highest yield extraction. Further, proximate, ultimate analysis, SEM, and EDX characterized the lignin-extracted sample.

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AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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