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Valorization of Sugarcane Bagasse through Microwave-Alkaline Pretreatment for Agricultural Waste Management and Biofuel Production

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Abstract

The environment is essential to human life and survival because it provides everything necessary for existence, development, and well-being. When materials such as agricultural waste are not properly utilized or managed, they become major sources of environmental degradation and health hazards. This study establishes the optimal pretreatment conditions for sugarcane bagasse using microwave-alkaline pretreatment (MAP) to enhance the recovery of cellulose and hemicellulose which are invaluable polysaccharides needed to produce bioethanol. Box–Behnken design (BBD) of Design Expert software version 13 was used to generate the model for the study using microwave power (70–700 W), irradiation time (1–5 min), and NaOH concentration (1%–3% w/v) as process parameters. The adequacy of the model was ascertained by statistical analysis. The pretreated biomass was characterized using XRD, FTIR, and HRSEM. The results indicated that the optimal MAP conditions were 3 % NaOH concentration, power of 96W, and treatment time of 5 mins. At these optimal conditions, 63% of the lignin in sugarcane bagasse was removed, while 74% of the cellulose and 22% of the hemicellulose were retained. There were physicochemical and morphological changes in the pretreated sugarcane bagasse biomass which validate the efficacy of the pretreatment method. Therefore, the present study has established the valorization of waste biomass to produce valuable products using microwave-alkali pretreatment. Overall, the process offers a sustainable pathway for managing agricultural residues while contributing to cleaner environments and renewable energy development. Further studies are recommended to evaluate the scalability of the optimized pretreatment conditions and to assess their economic feasibility in industrial applications.

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Keywords: microwave-alkali, biomass, cellulose, hemicellulose, environment

1.0. Introduction

The need to maintain the global energy cycle has drawn the attention of researchers to inefficient waste management and how these wastes can be converted to sustainable energy. The environment has been an invaluable jacket which provides all the necessary support of life both for animals and plants as well as its abiotic components. It is, however, unfortunate that the continuous unguided use of natural resources as well as the poorly managed agricultural wastes are constituting a menace to the environment, leading to emission of greenhouse gases and health trauma. Reports from Hiranobe *et al*. 2024, show that sugarcane bagasse is notable among the mostly generated and underutilized waste. An estimated amount of 270-280 kg of bagasse can be generated from a ton of sugarcane (Lachos-Perez *et al*., 2016). The quantities of sugarcane bagasse being generated annually in Nigeria and other tropical countries are in millions of tons; the majority of these wastes are either burnt or left to decay, thereby releasing dangerous gases to the environment (Omonije, 2023).

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The main components of the fibrous sugarcane bagasse are lignin (17 -32%), cellulose (32-45 %) as well as hemicellulose (20 -32%) (Alokika *et al*., 2021). These major components have qualified it as a material that can be used to produce invaluable chemicals and energy generating substance such as bioethanol. The major challenge in the production of bioethanol using sugarcane bagasse is the very stubborn nature of lignin which restricts the enzymatic and fermentation processes (Ntunka *et al*., 2025). This has necessitated the need for a pretreatment process that will not only remove the lignin but also preserve the fermentable sugar for upstream processes. Although, a lot of pretreatment strategies, such as physical, chemical or biological methods, have been employed, most of these strategies are accompanied with limitations such as high cost, environmental pollution and use of outrageous quantities of energy (Joshi and Manjare 2024).

One of the recently emerged pretreatment strategies is the use of microwave in conjunction with other methods, especially chemical methods. For instance, the use of microwave with alkali which is also called Microwave-alkali pretreatment (MAP) has been reported as a method involving minimal chemical usage which selectively removes lignin. The selective removal of lignin will then grant access for enzyme to effectively hydrolyse cellulose and hemicellulose (Yuan *et al*., 2021). In comparing MAP to other well-known pretreatment strategies, MAP has some advantages such as optimal energy use, timesaving as well as the use of reduced chemicals which made it one of the best methods for biomass valorization.

The important operational parameters of MAP which includes microwave power, irradiation time, and concentration of alkali greatly determines its efficiency. The random use of these parameters may lead to energy wastage, loss of carbohydrates and retainment of excessive lignin. Therefore, optimizing MAP process parameters to maximally retain cellulose and hemicellulose while minimising the lignin content is a major step in the pretreatment of lignocellulosic agrowastes. In this study, MAP process parameters were optimized to effectively remove lignin while retaining cellulose and hemicellulose. This offers a dual benefit of reducing accumulation of agricultural waste as well as production of biofuel for a greener environment

2.0. Materials and Methods

2.1. Materials

Sugarcane bagasse biomass was gathered from farms and agro-industrial facilities in Bosso, Minna, Niger State, Nigeria. Sodium hydroxide (NaOH) and other chemicals used throughout the study were of analytical grade. Clean running tap water was utilized for all washing while distilled water was used in the dilution steps.

2.2. Preparation of Biomass sample

To get rid of any pollutants that stuck to the sugarcane bagasse samples, they were washed with running tap water and afterwards air-dried at sun temperature (35 ± 2) ° C until they reached a constant weight. After drying, the biomass was ground into small particles with a mechanical grinder, weighed, and kept in airtight containers at room temperature until it was needed again (Sluiter et al., 2012).

2.3. *Microwave-Alkaline Pretreatment (MAP)*

Microwave-Alkaline Pretreatment was conducted in accordance with the experimental parameters established by the Box–Behnken factorial design (BBD), utilizing Design Expert software version 11 (Egwim et al., 2015; Singh and Trivedi, 2014). Three independent variables were examined which includes: Concentration of NaOH (A): 1–3%; Power for the microwave (B): 70–700 W; and Time for treatment (C): 1–5 min. Before being microwaved, each biomass sample (10% w/v) was soaked in NaOH solution for 10 minutes. The BBD produced 17 experimental runs to test how these variables affected three responses: the percentage of lignin removed (R_1), the cellulose retained (R_2), and the hemicellulose retained (R_3). After the pretreatment, in order to attain a neutral pH for the pretreated biomass, the mixture was filtered, and the solid residue was rinsed many times with clean tap water. The residue was dried in an oven at 60°C until it reached a constant weight (Singh et al., 2020), and then it was kept for more tests. Table 1 shows the 17 experimental runs that the software generated. After being pretreated, the biomass leftovers were subjected to structural and compositional analysis.

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Table 1: Design Expert Software's Variable Factors and Experimental Runs for Optimizing Microwave–Alkaline Pretreatment of Sugarcane Bagasse

		Factor 1	Factor 2	Factor 3	Response 1	Response 2	Response 3
Std	Run	A: Conc. NaOH (%)	B: Power (Watts)	C: Time (Min)	Delignification (%)	Cellulose remaining (%)	Hemicelluloseremaining (%)
1	1	2	70	5			
1	2	3	70	3			
2	3	2	385	3			
1	4	2	700	5			
7	5	2	700	1			
1	6	1	385	5			
2	7	3	385	1			
1	8	2	385	3			
0	9	2	385	3			
7	10	2	385	3			
6	11	1	385	1			
1	12	1	70	3			
4	13	1	700	3			
1	14	3	385	5			
5	15	2	70	1			
1	16	2	385	3			
3	17	3	700	3			
5							
1							
3							
8							
9							
1							
6							
4							

2.4. Analysis of Composition

In Analysing the composition of pretreated biomass involve two major steps which are

2.4.1. **Total Lignin Quantification**

The National Renewable Energy Laboratory (NREL) approach given by Sluiter et al. (2012) was used to figure out the total lignin content. Briefly, 1.5 g of dry pretreatment biomass was mixed with 15 mL of 72% (w/v) H_2SO_4 and agitated for an hour. Then, 420 mL of distilled water was added to the mixture, and it was autoclaved for 1 hour at 121°C. The sample was filtered and a UV-visible spectrophotometer was used to measure the absorbance of the filtrate at 320 nm. The concentration of acid-soluble lignin (ASL) was found from this approach using Equation (1):

$$ASL = \frac{Abs \times Dilution}{\varepsilon \times W} \times 100 \quad (1)$$

where Abs = absorbance at 320 nm, ε = absorptivity of lignin (30 L/g·cm), and W = weight of the sample (mg).

The residue was dried in an oven at 105°C for 4 hours, then ashed at 300°C for 1 hour, and then left in a desiccator to cool before being weighed to find the acid-insoluble lignin (AIL) as stated in Equation (2):

$$\text{AIL} = \frac{Weight\ of\ sample\ at\ 105°C - \text{weight of sample at } 300°C}{Initial\ weight\ of\ sample} \times 100 \quad (2)$$

The total lignin was found by adding ASL and AIL together (Equation 3):

$$\text{Total lignin} = \text{ASL} + \text{AIL} \quad (3)$$

Total cellulose and hemicellulose quantification

The Chesson–Datta gravimetric method was used to find out how much cellulose and hemicellulose were in untreated

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and pretreated samples (Maryana et al., 2014). One gram of biomass was refluxed with 150 mL of distilled water at 100°C for one hour and then filtered. The residue was rinsed with 300 mL of hot water and dried in the oven at 100°C until it weighed the same (b).

Then, 150 mL of 0.5 M H_2SO_4 was added to the dry residue and heated for an hour. The residue was cleaned, dried at 60 °C, and then weighed (c). To the residue was added 10 mL of 72% H_2SO_4, it was left to soak for 4 hours, then diluted with 150 mL of 0.5 M H_2SO_4 and then refluxed for another hour. The residue was cleaned until it was neutral, dried at 105°C for 6 hours, and then weighed (d). Then, using Equations (4) and (5), the total amount of cellulose and hemicellulose were calculated as indicated in Equation 4 and 5

$$Hemicellulose\ content = \frac{b-c}{a} \times 100 \quad (4)$$

$$Cellulose\ content = \frac{c-d}{a} \times 100 \quad (5)$$

The amount of lignin removed was estimated by comparing the lignin levels in untreated and pretreated biomass samples. Design Expert version 13 was used to make response surface plots and analysis of variance (ANOVA).

2.5. *Characterization of Pretreated Biomass Samples*

X-ray Diffraction (XRD), High-Resolution Scanning Electron Microscopy (HRSEM), and Fourier Transform Infrared Spectroscopy (FTIR) were used to examine the changes which has occurred in the structure of the biomass after pretreatment.

2.5.1 X-ray Diffraction (XRD) Analysis

X-ray Diffraction (XRD) patterns detect crystallinity of the cellulose. The 2θ range of 0° to 90° was used to acquire diffraction data. Using the Scherrer equation (Equation 6), average size of the crystallites (D) was calculated:

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (6)$$

where K = 0.94, λ = 0.1541 nm (X-ray wavelength), β = full width at half maximum (FWHM) in radians, and θ = Bragg's diffraction angle. Using the Segal approach (Jamaldheen et al., 2018), the crystallinity index (CrI) was estimated by equation 7

$$CrI = \frac{I^{002}Crystalline - Iamorphous}{I^{002}Crystalline} \times 100 \quad (7)$$

I^{002} and I_{am} are the intensities of crystalline (22.5°) and amorphous (18.7°) areas, respectively.

2.5.2 High-Resolution Scanning Electron Microscopy (HRSEM)

The surface morphology of both untreated and pretreated sample was examined using High-Resolution Scanning Electron Microscopy (HRSEM). Images were taken at different levels of magnification to see how microwave–alkaline treatments changed the organization of the fibers, the amount of space between them, and the roughness of their surfaces.

2.5.3. FTIR Spectroscopy (Fourier Transform Infrared)

Changes in functional groups as a result of Microwave-Alkaline pretreatment was observed using FTIR spectroscopy (Thermo Nicolet Avatar 370, with KBr beam splitter and DTGS detector). Spectra were recorded between 4000 and 400 cm^{-1} with a resolution of 4 cm^{-1} to find the peaks that are unique to the functional groups of lignin, cellulose, and hemicellulose.

2.6. Statistics Analysis

All experiments were performed in duplicate. The data were presented as mean ± standard error (SE). Microsoft Excel version 2021 and IBM SPSS Statistics version 21 (IBM Corp., Chicago, USA) were used to do the statistical analysis. A one-way analysis of variance (ANOVA) and Duncan's Multiple Range Test (DMRT) were used to look at the differences between the means. We accepted significance at $p < 0.05$.

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3.0 Results and Discussion

3.1. Cellulose, Hemicellulose and Lignin Content of Dried Unpre-treated Agrowaste

Typical of any lignocellulosic biomass, the major component of sugarcane bagasse are cellulose, hemicellulose, and lignin. The result of the compositional analysis in the present study as shown in Table 1 shows that there are 48% of cellulose, 24% of hemicellulose, and 21.5% of lignin in the original sugarcane bagasse used for this study before pretreatment, although this composition is slightly different from the composition (45%, 26%, and 21% for cellulose, hemicellulose and lignin) reported for sugarcane bagasse by Boneberg et al. (2016). However, Xu et al. 2016; Rezania et al. 2020 showed in their studies that discrepancies in biomass composition are affected by factors like plant genetics, ambient circumstances, processing methods, and analytical methodologies

3.2. Effect of Microwave-alkaline Pre-treatment on the Weight and Percentage of Lignin in Sugarcane Bagasse

It was noticed that after pretreatment with microwave-alkaline pretreatment (MAP), there was a considerable decrease in the weight of the remnant biomass, as shown in Table 2, which was an indication of lignin removal. The least of the solid mass after pretreatment (1.96 g) was noticed at 3% NaOH, 700 W, and 5 minutes (Run 14), leading to a mere 4.9% lignin retention. This corresponds with the results of Wang et al. (2020), who noted that when the concentration of alkali is increased and the reaction time is prolonged, there is increase in the quantity of lignin removed and at the same time a reduction in the quantity of biomass recovery is noticed. The same trend was observed by Kim et al. (2016), who concluded that the removal of lignin enhances cellulose accessibility by disrupting cross-linkages and diminishing crystallinity, therefore improving enzyme interaction and hydrolysis efficiency.

3.3. Process Variables and Responses for Microwave-alkaline Pre-treatment of Sugarcane Bagasse

The experimental results (Table 3) indicated that lignin removal, cellulose retention, and hemicellulose retention fluctuated with processing settings. The optimum delignification of sugarcane bagasse was attained with 3% NaOH, 73 W, and a duration of 5 minutes, resulting in 66% lignin removal, 74% cellulose retention, and 22% hemicellulose retention. These circumstances resulted in a high holocellulose content (96%) and minimal solid recovery, demonstrating effective lignin removal without much carbohydrate degradation. Relative to prior investigations, these factors provide benefits regarding energy efficiency, reagent utilization, and treatment duration. Rezende et al. (2018) documented a 40% augmentation in cellulose content following acid-alkali pretreatment, while our study attained a 36% rise under gentler, shorter, and more environmentally sustainable settings. Likewise, the technique surpassed the NaOH–autoclave method employed by Wang et al. (2019) and Hamouda et al. (2015), which necessitated 7.15% NaOH and exceeded seven hours of reaction time.

Table 1: Cellulose, Hemicellulose and Lignin Content of Dried Unpre-treated Agrowaste

Agrowaste	Cellulose Content (%)	Hemicellulose Content (%)	Lignin Content (%)
Sugarcane bagasse	48	24	21.5

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Table 2: Effect of Microwave-alkaline Pre-treatment on the Weight and Percentage of Lignin in Sugarcane Bagasse

Weight (g)				
Run	Treatment conditions	Before delignification	Weight (g) after delignification	% total Lignin retained
1	Unpre-treated	5.00	5.00	21.50
	2%/70 W/5 min	5.00	3.40	8.88
2	1%/385 W/5 min	5.00	3.15	7.07
3	1%/700 W/3 min	5.00	3.23	6.84
4	3%/70 W/3 min	5.00	3.24	9.74
5	2%/385 W/3 min	5.00	2.00	8.56
6	2%/385 W/3 min	5.00	2.85	9.70
7	2%/70 W/1 min	5.00	4.16	12.64
8	3%/700 W/3 min	5.00	2.91	6.21
9	2%/700 W/1 min	5.00	3.51	9.01
10	1%/70 W/3 min	5.00	3.85	11.35
11	2%/70 W/5 min	5.00	3.49	8.99
12	2%/385 W/1 min	5.00	3.81	10.69
13	2%/385 W/3 min	5.00	3.53	9.44
14	2%/700 W/5 min	5.00	1.96	4.90
15	3%/385 W/5 min	5.00	2.82	6.04
16	1%/385 W/1 min	5.00	3.81	11.31

Table 3: Process Variables and Responses for Microwave-alkaline Pre-treatment of Sugarcane Bagasse

	Factor 1	Factor 2	Factor 3	Response 1	Response 2	Response 3
Run	A: NaOH Conc (%)	B: Power (Watts)	C: Time (Mins)	Lignin removed (%)	Cellulose retained (%)	Hemicellulose retained (%)
	2	70	5	58.7	60	22
2	1	385	5	67.1	61	21
3	1	700	3	68.2	70	18
4	3	70	3	54.7	75	20
5	2	385	3	60.2	65	19
6	2	385	3	54.9	64	19
7	2	70	1	41.2	63	21
8	3	700	3	71.1	57	15
9	2	700	1	58.1	62	14
10	1	70	3	47.2	52	22
11	2	385	3	58.2	63	20
12	3	385	1	50.3	63	15
13	2	385	3	56.1	63	19
14	2	700	5	77.2	70	19
15	3	385	5	71.9	69	21
16	1	385	1	47.4	63	21
17	2	385	3	60.2	65	19

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3.4. Three-Dimensional Response Surface Plots of Pretreated Sugarcane Bagasse

Response surface plots (Figures 1-3) depicted the interactive effects of the variables. Delignification of sugarcane bagasse increased with elevated NaOH concentration and extended treatment duration, while excessive power levels or alkali concentrations resulted in hemicellulose breakdown. The retention of cellulose exhibited a positive connection with NaOH content and reaction duration at moderate power levels (about 385 W). These findings highlight the necessity of equilibrating the parameters to attain optimal delignification while preserving carbohydrate content.

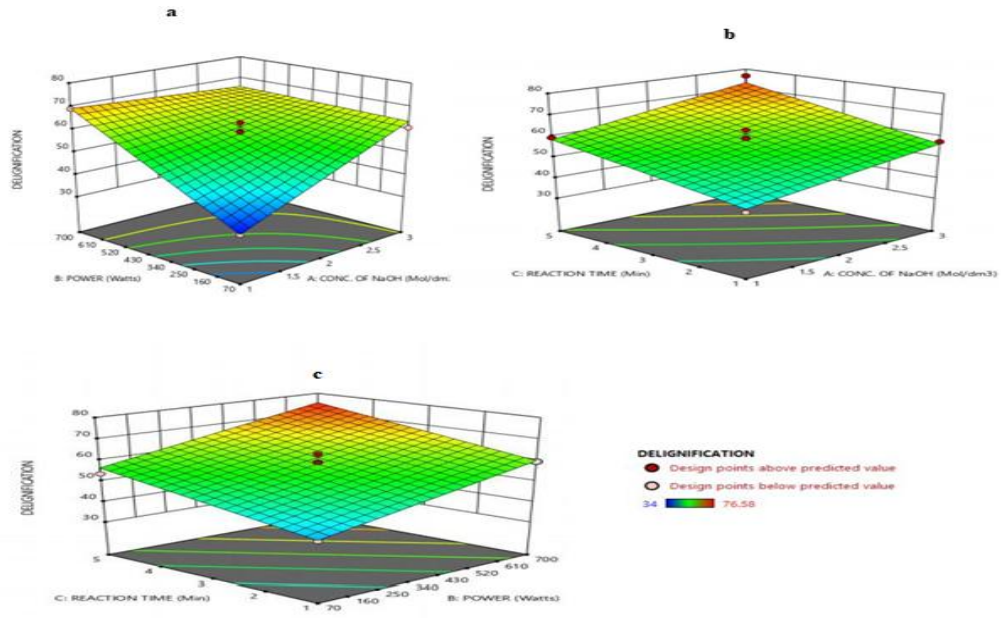


Figure 1: Three-Dimensional Response Surface Plots of Sugarcane Bagasse for:Percentage Delignification at (a) constant Time, (b) constant power (c) constant NaOH concentration

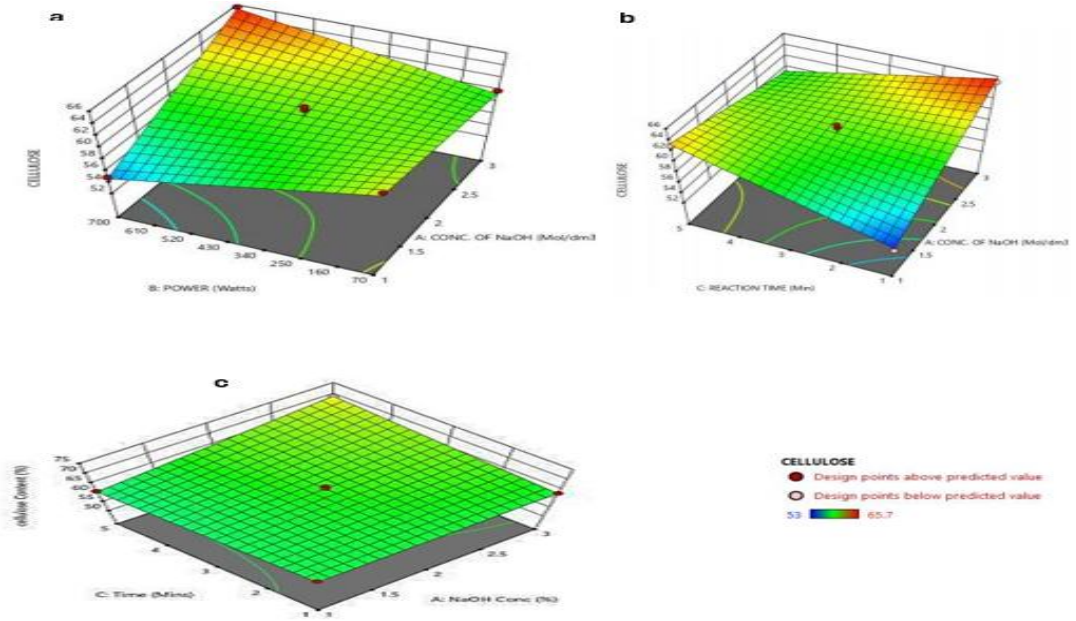


Figure 2: Three-Dimensional Response Surface Plots of Sugarcane Bagasse for:
Percentage Cellulose Removed at (a) constant Time, (b) constant power (c) constant NaOH concentration

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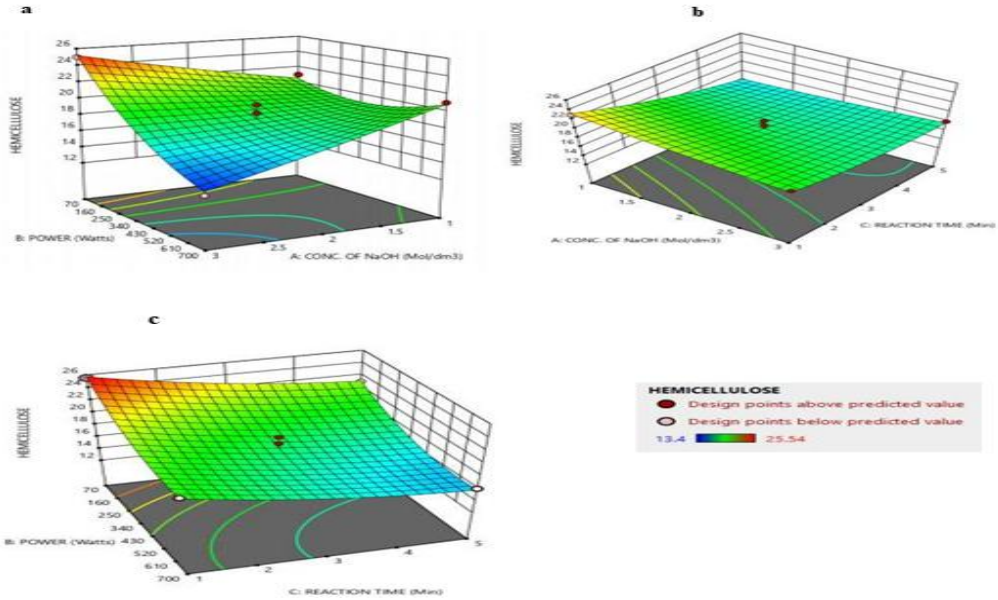


Figure 3: Three-Dimensional Response Surface Plots of Sugarcane Bagasse Biomass for: Percentage Hemicellulose Retained at (a) constant Time, (b) constant power (c) constant NaOH

3.5. Structural Analysis of Pretreated and untreated Sugarcane bagasse

X-ray diffraction (XRD) study as shown in Figure 4 demonstrated an enhancement in the crystallinity index (CrI) of sugarcane bagasse from 35.94% (untreated) to 43.31% (pretreated), signifying the efficient elimination of amorphous constituents such as lignin and hemicellulose. The improvement in crystallinity aligns with findings by Luz et al. (2020) and Ovalle-Serrano et al. (2018), who associated delignification with enhanced enzymatic hydrolysis efficiency. The minor alteration in diffraction peaks between untreated and pretreated samples (from 16.67°, 22.05°, and 35.2° to 16.25°, 22.22°, and 35.9°) indicates that there is no conversion of native cellulose I to alternative allomorphs, hence maintaining the cellulose crystalline structure while enhancing accessibility.

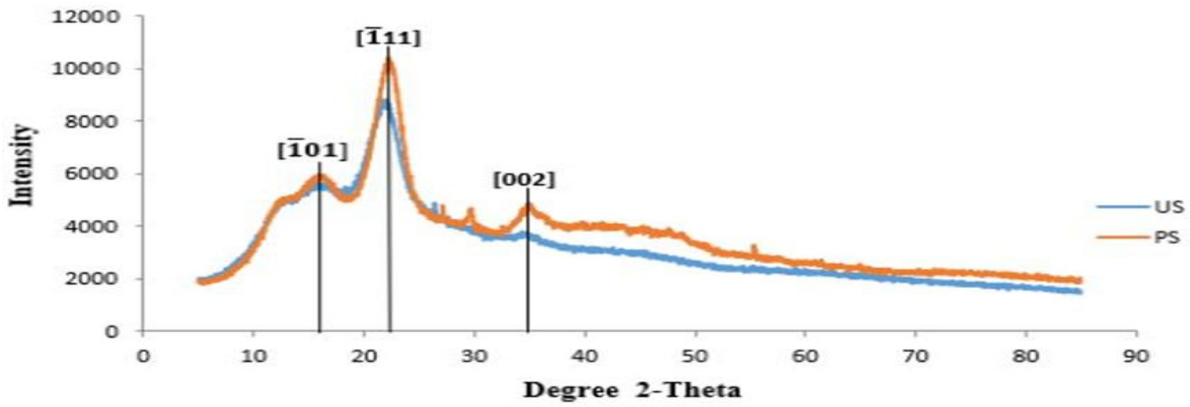


Figure 4: X-Ray Diffraction Pattern of the Untreated (US) and Pre-treated Sugarcane Bagasse (PS)

3.6. Chemical Analysis of Pretreated and untreated Sugarcane bagasse

The Fourier-transform infrared (FTIR) spectra as shown in Figure 5 further validated the elimination of lignin and hemicellulose. The extensive O–H stretching bands within the range of 2995–3400 cm^{-1} exhibited reduced intensity in treated samples, signifying breakdown of hydrogen bonds and cleavage of lignin. The reduction in absorbance at 3401 cm^{-1} and the intensified bands between 2208–2995 cm^{-1} (C–H stretching of aliphatic groups) indicate structural

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reorganization and enhanced crystallinity (Boukir et al., 2019; Luz et al., 2020). The distinctive peaks in 1765–1715 cm^{-1} (C=O) and 1646 cm^{-1} (O–H bending of absorbed water) further substantiate the breakdown of hemicellulose and delignification during pretreatment.

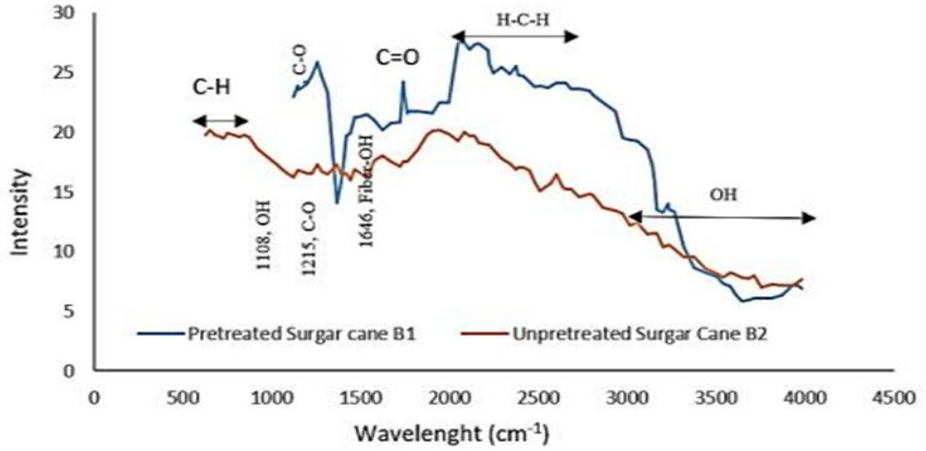


Figure 5: Infrared Spectra of the Untreated and Treated Sugarcane Bagasse

3.7. Morphological Analysis of Pretreated and Untreated Sugarcane Bagasse

Scanning Electron Microscopy (SEM) as shown in Plate 1 demonstrated a clear morphological alteration. Untreated sugarcane bagasse exhibited a dense, compact surface, but the processed sample demonstrated fibrillation, fissures, and enhanced porosity. The microstructural alterations result from NaOH-induced swelling, which compromises lignin–carbohydrate bonds and improves fiber accessibility (Sahare et al., 2012; Haldar et al., 2018). The increased surface area enhances enzyme access and hydrolysis, hence augmenting saccharification efficiency and, subsequently, potential ethanol output.

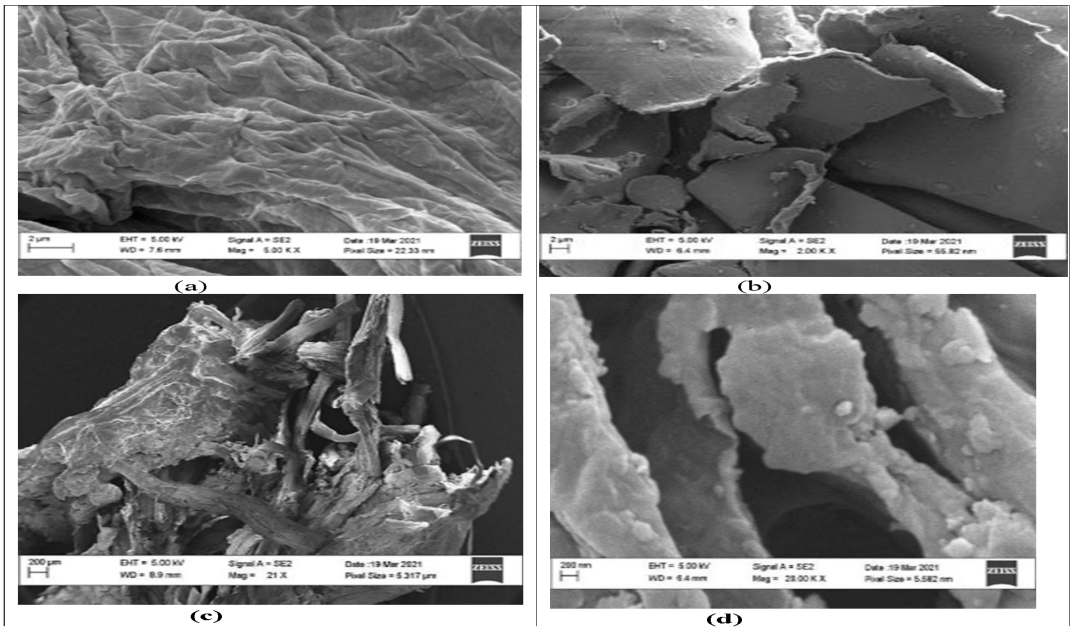


Plate 1: Scanning Electron Micrograph of Sugarcane Bagasse for(a)unpre-treated sample at magnification of 10 µm; (b) pre-treated sample at Magnification of 10 µm; (c) unpre-treated sample at Magnification of 200 µm; (d) pre-treated sample at Magnification of 200µm.

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4.0. Conclusion

This Microwave-assisted alkaline pretreatment efficiently disrupted the lignocellulosic structure of sugarcane bagasse, leading to substantial lignin removal and improved cellulose crystallinity. The improved parameters (3% NaOH, 73 W, 5 min) exhibited enhanced delignification efficacy with reduced energy and chemical consumption, rendering the process both industrially viable and environmentally sustainable. The documented physicochemical and morphological changes validate the efficacy of this pretreatment method for large-scale bioethanol production from agricultural leftovers.

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