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Physico-Chemical Characterization of Alkali-Treated Ethiopian Arabica Coffee Husk Fiber for Composite Materials Production

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Abstract: Chemical treatment is a significant factor in improving the natural fiber quality for composite materials production. In this study, the alkaline treatment of Ethiopian Arabica coffee husk by sodium hydroxide (NaOH) was performed to improve the fiber quality. A total of 10% (*w*/*w*) NaOH has been applied for the alkaline treatment. Comprehensive physicochemical characterizations, such as proximate analysis, cellulosic composition, porosity, and structural analysis of treated and untreated coffee husk, have been conducted and compared. The experimental results showed that lignin and hemicellulose were reduced by 72% and 52%, respectively, thus improving the overall fiber quality. Therefore, this study indicated alkaline treatment of Ethiopian coffee husk is effective for fiber quality enhancement. It can be applied as a potential feedstock for fiber production in the composite production sector.

Keywords: Ethiopian coffee husk; composite material; NaOH treatment; fiber analysis

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1. Introduction

Natural fibers have received much attention in recent years as an option reinforcement in polymer composites because of their advantages over glass and carbon fibers in research and industrial applications. These fibers are less expensive, have a lower environmental impact, have a lower density, use less energy, and have a broader range of applications [1–3]. Plant, animal, and mineral-based fibers are the three types of natural fiber sources. Plant fibers contain more cellulose than animal fibers, but animal fibers contain more protein. In general, plant fibers are stronger and more rigid than animal and mineral fibers [4]. Plant chemical composition varies by geographic location, age, soil, and climate conditions, as well as within different portions of the same plant [1]. Dry coffee refining produces about 1 kg of CH per 2 kg of coffee beans [2]. This huge amount of coffee husk generated from coffee processing industries results environmental pollution. Environmental pollution is solved by changing the disposal system of coffee husk, as well as changing it into value-add products, such as its use as a reinforcement material in composite materials.

Coffee is the world's second most valuable commodity, after petroleum, consumed by roughly one-third of the world's population [2,3]. It enables us to overcome this environmental issue. Complements the value additions of these waste materials [4]. The coffee husk contains lignin and hemicellulose. Lignin is an amorphous polymer with a complex system of go-linking [4,5]. It is well-known to pass numerous useful agencies and high amounts of material of guaicyl, syringyl, and p-hydroxyphenyl chemical units [4,5].

Extracting lignocellulosic biomass, along with coffee husk, is both economically and environmentally appealing for practical application. To remove lignin, several systems have been suggested, including alkali treatment, acid remedy, natural solvent rehabilitation,

and new technology [4,6–8]. The goal of lightweight construction is to keep or possibly increase product functionality. Reducing automotive mass by 100 kg saves 0.71 fuel each 100 km, both directly and indirectly. Fiber-reinforced thermoplastics-based technologies can be intergraded into the manufacturing process for light weight composite structures. The main motivators for lightweight materials applications are weight savings and potential cost savings. According to Fan, J. and Njuguna, J. [9], it means that the weight and possible cost savings experience significant weight reduction with improved performance, which means less fuel consumption and CO_2 emission. In addition to mass reduction, composite materials have consistent potential advantages, in terms of noise and vibration reduction, as well as energy absorption capability. Lightweight composites can scale back fuel consumption and CO_2 emissions to the atmosphere [9]. Coffee husk has high density, eco-friendly material, and cost-efficient resources and would possibly replace manufactured fiber in industries. Coffee husk is very important for the production of products when value-added to it but it is not utilizing as the availabilities of resources [10]. Polyphenolic compounds allied to hemicellulose or lignin subsets in lignocellulosic agro-waste have effective characteristics for dominant microorganisms or aerobic mechanisms [11]. Different polyphenols, such as phytonutrients, glycosides, flavones, or synthetic groups, are supported by their structure or synthetic groups [6]. These types of lignocellulosic wastes are accustomed extract polyphenols that are largely evaluated on their inhibitor activity in the main victimization of organic solvents, such as fermentation methyl alcohol or alcoholic beverage [7,12]. Aside from these benefits, CH waste conjointly does have some drawbacks, such as cathartic dissolved organic elements found in coffee husks, such as polymer, tannin, and phytic acid, as well as polysaccharide into the exhaust stream, which increases the natural gas requirement, chemical gas request, and organic matter content and reduces gas output within the water habitats [13]. Straightforward utilization of coffee husk seems to be wasteful, due to low capability and potency [14]. Physio-chemical treatments are commonly used to improve the mechanical properties of coffee husks [15]. Physical pretreatment strategies involve chemical treating, heating, grinding, autoclaving, drying, boiling, and extractions [16]. The physical modification of CH eliminates contaminants, organic, and inorganic substances from the coffee husk's skin [17]. Among varied chemical treatment options, hydroxide preparation has been extensively used for the surface treatment of agricultural residues to boost its properties and potency. Alkali such as NaOH decompose the attachment among elements of lingo-cellulose by hemicellulose chemical reaction but also polymer deploy-memorization. Organic fats, waxes, as well as other low-mass compounds, are removed from the CH after treatment with NaOH [18,19]. NaOH also improves the natural material's physical, mechanical, and chemical characteristics, such as the responsiveness, durability, and natural capacity [20]. The ionization of the hydroxyl groups in the medium is triggered by sodium hydroxide (NaOH) solution, which chemically modifies the structure by breaking hydrogen bonds [21,22].

The effectiveness of this procedure is determined by elements such as reaction time, temperature, and the concentration of the alkaline solution. High concentrations of NaOH solution, on the other hand, might accelerate fiber delignification, thus weakening and degrading the microstructure of the fiber [22–24]. As the summary of the research in the field reveals, several scholars have studied and examined the impact of chemical treatment on various natural fibers. Plant fiber qualities, for example, are naturally influenced by the temperature and fertility of the land in which the plant grows. Research on the fiber of coffee envelope in the Oromia region of Ethiopia, on which this study focuses, has yet to be published. As a result, the purpose of this research is to characterize the fiber of coffee husk from Oromia, South-West Ethiopia, by investigating the impacts of chemical treatment on physio-chemical composition characteristics, such as by using a tool for composite materials. This study determines which one provides the most cellulose molecule exposure, allowing CHF to fully realize its potential as a polymeric bio-composite reinforcing filler for use in high-density polyethylene matrices. These composites can be used in household products, telephone closing boxes, and automotive application for plastic wood.

2. Materials and Methods

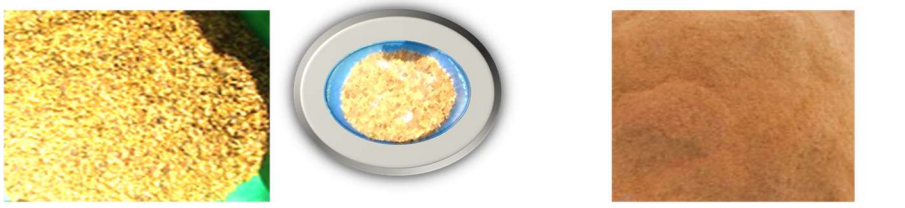
2.1. Materials

For this investigation, coffee husk fiber was gathered from the Oromia region of Ethiopia, around Ilu ababor and Buno Bedele, South-West Ethiopia. Ethanol, toluene, alcohol, nitric acid, alkali (NaOH), sulfuric acid (H_2SO4), HCl, and acetone were obtained from the Merck Group Chemical Company (Darmstadt, Germany). Evaluations of the physical and chemical characteristics of fibers and tools for laboratory work, such as autoclave, measuring cylinder, desiccator, electronic balance, soxhlet, oven, water bath, and furnace, were used to determine the characteristics of the fiber. The chemical composition of the fiber was also determined using Fourier transform infrared spectroscopy (FTIR) and optical microscopy (OM).

2.2. Alkali Treatments of Coffee Husk Fiber

The coffee plant and its local husk preparation systems are illustrated in Figure 1A–D. Arabica coffee (coffee Arabica) is a shrub that grows to a height of about 12 feet in its natural state, but not more than 4.5 m when grown. After the fruit ripened, it was collected, dried, and coffee processing produced the rind and the husk made, as demonstrated in Figure 1A–E. The coffee husk has a thickness of 3–5 mm and length of 0.20 m. For this study, the matured coffee bean was collected and dried by sunlight for 48 h, as shown in Figure 1A–C. The dried coffee bean was put in the machine to remove the husk from coffee bean; then, coffee husk was generated, as shown in Figure 1D,E, but also washed by clean water up to a dirty of husk removed oven-dried for 24 h. For 2 h, the fiber was immersed in a 10% NaOH solution. To remove impurities from the fiber's surface, the husks were washed frequently with normal water and then with clean water before drying at room temperature for 24 h. The coffee husk fiber has been treated with 10% NaOH alkali, as indicated in Figure 1F. Lastly, the treated coffee husk was ground with a milling machine of 0.18 mm to obtain the powder, as shown in Figure 1G below.

A. Coffee plant **B. Coffee bean** **C. Dry Coffee bean** **D. Coffee husk**



E. Untreated coffee husk **F. Treated coffee husk** **G. Coffee husk powder**

Figure 1. Coffee husk fiber preparation process.

2.3. Physical Properties of Coffee Husk Fiber

2.3.1. Densities (ρ_F)

The sample dried at 105 °C for 24 h, then ready 3 g sample was weighted.

With 0.001 g preciseness balance and immersed in water for 2 h, at the finish of immersion amount, removed the sample from the beaker, and wiped off the surface water with a clean and dry towel before weighing them once more. This measurement was made with a pycnometer for solids and distilled water as the immersion liquid. The temperature

in the room was 21 °C, with the pycnometer reading 60% and desiccator reading 4%. The distilled water prepared it in room temperature in the university lab, which is 25 to 35 °C for this experiment and we measured the density of the distilled water; the density was 0.998 g/cm^3, and the density of CH fibers was calculated as follows. Samples were measured at (25 ± −0.3) °C, using Mettler Teledo [25]. The Equation (1) formula provides the density of fibers.

$$\rho F = \frac{(m_2 - m_1)}{(m_3 - m_1) - (m_4 - m_2)} \times \rho w \quad (1)$$

where ρF is of fiber's density (g/cm^3), m_1 is the mass of the suspension wire in the air, m_2 is the mass of the suspension wire in the liquid, m_3 is the mass of the suspension wire plus sample mass in air, m_4 is the mass of the suspension wire plus the sample mass in the liquid (gram), and ρw is the distilled water standard density.

2.3.2. Coffee Husk Fiber Moisture Absorption

Moisture content can affect the physical characteristics of composites, as well as the matrix-fiber interface [25]. To determine the wetness content of coffee husk fiber, weight 3 g of coffee husk was dried at (105 ± −2.0) °C for 24 h, according to ASTM D1762-84 (2007), until a relentless weight was measured. The moisture content was found on a share basis (TAPPI T 262 om-02) by the Equation (2) [25].

$$\%\ M_C = \frac{(W_{fb} - W_{fa)}}{W_{fb}} \times 100\% \quad (2)$$

where Mc is the moisture content of fiber, W_{fb} is the weight of fiber (g) before putting in an oven, and W_{fa} is the weight of fiber (g) after putting in an oven. Coffee husk water absorption testing at room temperature was advised. The test was performed, following the ASTM D570 standard [20], by removing samples from the oven at regular intervals and immediately weighing them with a digital electric scale to determine the amount of water absorbed. All samples were dried in an oven, until they reached a constant weight. BD-NE10 biological optical microscopy is used for tests at 10× to 100× this means that a size of 0.2 µm objects can be detected. The resolution of optical microscope is limited to 0.2 µm, and the practical magnification limit is 1000×.

2.3.3. Fiber Diameter Measurement

A single fiber sample is cut into small splices, placed on a microscopy slide, and projected microscopically onto a screen. Using optical microscopy, the diameter of the coffee husk was measured. The average diameter value of five randomly selected samples were reported at five locations along the length.

2.3.4. Determination of Volatile Matter (VM)

Volatile matter is the total loss in weight excluding moisture, where the coffee husk was heated out of contact with air under specified conditions. It mainly contains carbon dioxide, carbon monoxide, hydrogen, hydrogen sulfide (H_2S), and ammonia. According to ASTM D1762-84 (2007), considering 5 g of coffee husk fiber, then after the sample was heated at (105 ± −2.0) °C for 2 h, heating associate degree oven-dried. The volatile matter of the coffee husk was determined by heating an oven-dried sample at 950 °C for seven minutes in the absence of oxygen gas. To determine the loss weight of the husk, heat a known weight of moisture free coffee husk sample in a covered platinum crucible at (950 ± 2) °C final temperature. The volatile matter was computed as the difference between the initial weight heated and final weight heated of the sample, to the ratio of initial weight of the coffee husk sample using Equation (3).

$$VM\% = \frac{\text{Mass of sample at } 105 \pm 2\ ^\circ\text{C(g)} - \text{Mass of sample at } 950 \pm 2\ ^\circ\text{C(g)}}{\text{Mass of } t\text{(g)}} \quad (3)$$

2.3.5. Determination of Fixed Carbon

The fixed carbon can be calculated by two ways: (a) proximate analysis; (b) ultimate analysis. The proximate analysis is very important empirical analysis used to determine fixed carbon. The data varies with the procedures adopted provides practical utility of coffee husk. The proportion of the fixed carbon content (*FC*) of the fiber was calculated by subtracting the sum of volatile matter (*VM*), ash content (*AC*), and moisture content (*MC*) from 100%. *FC* was determined by Equation (4).

$$FC(\%) = 100\% - (VM\% + AC\% + MC\%) \quad (4)$$

2.3.6. Determination of Porosity of Coffee Husk Fiber

The dry sample weight and grain density can calculate grain volume. Using the density of quartz as the grain density can yield accurate results for various reasons. Once water is added, a wire mesh screen is placed on the top of the sample to prevent materials from becoming suspended. H_2O was gently supplemented over the sample till the H_2O level goes over the sample. The cylinder should specifically rock from the edges to urge eliminate any disturbances; then, the final word H_2O was recorded. Then, the number of supplemental H_2O to the cylinder and, therefore, the H_2O level record. The cylinder must be cleaned after each test. The porosity of the husk is then determined by Equation (5).

$$\rho(\%) = \left(\frac{v_i - v_f}{v_s}\right) \times 100\% \quad (5)$$

where ρ is the porosity of the sample, V_i is a combined volume of sample and water, V_f is the final total volume of the sample and adds water, and V_s is the volume of the sample.

2.4. Chemical Composition of Coffee Husk Fiber

The following tests were performed to assess the content of lignocellulosic elements in coffee husk fiber (CHF) after the treatments. Extractive content was determined according to NBR 14853 [26,27], using the following solvents: (i) a solution of toluene and ethanol (2:1) for the first 4 h of extraction; (ii) solely ethanol for the second 4 h of extraction; and (iii) 100 °C in water immersion for the third extraction. The lignin content was determined using NBR 7989 [26,27], with the sample free of extractives. Cellulose and hemicellulose concentrations were determined using Rewell's techniques [26]. The ash content of the samples was evaluated using the TAPPI standard (T211 om-02) [26,28].

Extraction of Wax, Cellulose, Hemicellulose, and Lignin

Its chemical composition influences fiber qualities, and fibers themselves can be thought of as fibrous composite materials. TAPPI standard T 264 method [25] was used to determine both NaOH treated and untreated coffee husk. Cellulose % was determined by Equation (6).

$$\text{Cellulose}(\%) = \frac{W_2}{W_1} * 100\% \quad (6)$$

where w_1 denotes the number of extractive free samples collected for analysis, and w_2 denotes the mass of the cellulose residue. Moreover, 2 g of treated and untreated coffee husk fibers were placed in a break, 250 mL of 72% H_2SO_4 was slowly added and agitated for 2 h, and then the H_2SO_4 was diluted to 3% and refluxed for 1 h to measure lignin concentration. The filter residue was rinsed in hot water and dried in an oven at 105 °C for 2 h before being weighed (w_2). The lignin content was calculated using the residue weight [25]. Similarly, the treated and untreated coffee husk fibers were soaked in a 10% NaOH solution at ambient temperature to calculate the hemicellulose material of the fibers.

2.5. Fourier Transform Infrared Spectroscopy

The Fourier transform infrared spectroscopy approach has been used to identify the properties of organic compounds by passing infrared light through the sample fiber [28].

NaOH has been used as the remover of lignin, hemicellulose, and other foreign materials from the fiber's surface. Information was collected from 500 to 4000 range, with a resolution of 4 cm^{-1} 32 scans after embedding the sample with KBr pellets (1/200 mg) [29]. The FTIR spectrum made for 10% NaOH treated and untreated coffee husk fiber.

2.6. Statistical Analysis

The regression analysis was employed to predict the that the moisture content of coffee husk in individual's parameters. The standard deviation value determines where a difference is between the treated and untreated coffee husk. The test data were subjected to a one-way analysis of variance (ANOVA) with Duncan's post hoc test at a 95% credible interval. Technical triple analyses were carried out. IBM SPSS software was used to analyze the data using the ANOVA and Turkey ($p > 0.05$) procedures (Version 21). The Shapiro–Wilk test was used to ensure that data was normal. The regression of the sample is 6 and the residual is 3 and total is 9 samples. Finally, regression analysis of the data showed that the untreated coffee husk is the most water absorbed. The correlation is significant at the 0.01 level (2-tailed).

3. Results and Discussion

3.1. Physical Properties of the Experimented Fibers

Moisture Content

Table 1 shows the physical characteristics of treated and untreated coffee husk that was measured in this investigation.

Table 1. Physical characteristics of coffee husk.

Properties	NaOH Treated Coffee Husk	Untreated Coffee Husk
	$Tt_{10\%}$	Untt.
Moisture (%)	3.02 ± 0.07	5.43 ± 0.66
Density (g/cm^3)	5.26 ± 0.04	3.1 ± 0.05
Volatile Matter	82.24 ± 0.03	84.77 ± 0.16
Fixed carbon	92.22 ± 0.12	91.81 ± 0.04
Porosity	50 ± 0.05	46.42 ± 0.041

Where: **Tt** is treated coffee husk fiber and **Untt** is untreated coffee husk fiber.

The moisture content of coffee husk enhanced after NaOH treatment, as shown in the table. According to experiments, untreated coffee husk absorbs more moisture than treated fiber. Because of the alkali treatment, the hydrogen bond in the fiber structure has broken down, thus enhancing the fiber humidity uptake capabilities by lowering the hydrophilic hydroxyl groups of the fiber for good intersection with the matrix.

Figure 2 shows moisture content of NaOH treated and untreated coffee husk fiber over a time. As seen from the output, untreated has higher moisture content than the treated fiber. The moisture content of both fibers is decreased, but the amount they decreased are different. High moisture content of the fiber leads to weakening the fiber for composite materials production. The standard deviation of the samples shows that the treated coffee husk is better than the untreated samples.

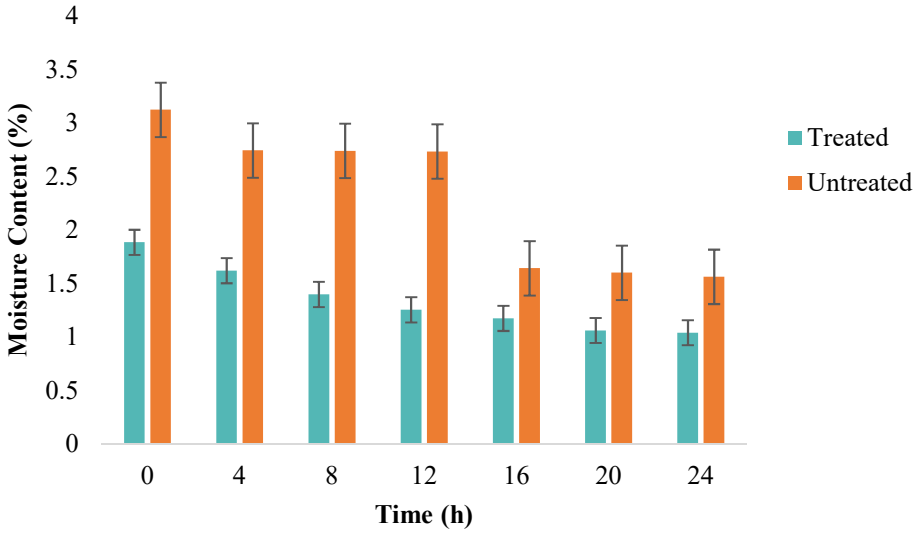


Figure 2. Moisture content of treated and untreated coffee husk fiber.

3.2. Chemical Properties of Coffee Husk Fibers

The wax, lignin, cellulose, and hemicellulose are fiber's most prevalent chemical compositions that impact its physical and chemical qualities. The chemical composition of 10% NaOH treated and untreated coffee husk fiber is shown in Table 2. The change in chemical properties came from NaOH treatment. NaOH treatment reduces the amount of lignin in the fiber, thus affecting the composite's characteristics and morphology. The lignin and hemicellulose content of coffee husk fiber treated with 10% NaOH was lowered by 73% and 52%, separately. The chemical composition points in Table 2 show that treated and untreated coffee husk fiber has high cellulose content.

Table 2. The chemical constituents of both untreated and NaOH-treated fibers.

Samples	Wax	Cellulose	Hemicellulose	Lignin	Ash Content of the Samples
Untt	7.51 ± 0.22	53.2 ± 0.07	17 ± 0.22	8 ± 0.03	3.42 ± 0.01
$Tt_{10\%}$	0.94 ± 0.12	56.58 ± 0.03	11 ± 0.12	4.75 ± 0.25	5.54 ± 0.02

3.3. Fourier Transform Infrared (FTIR) Spectrometry

The fiber comprises cellulose, hemicellulose, lignin, wax, and other contaminants, and the NaOH treatment was used to eliminate the hemicellulose, lignin, and other unwanted materials from the surface the fiber. FTIR was used to investigate the effects of NaOH treatment on chemical fiber compositions. Figure 3 depicts the whole infrared pattern of untreated and 10% NaOH-treated coffee husk fibers.

With a resolution of 32 scans, all spectra ranged from 4000 to 500 cm^{-1}. The alcohol compound or hydrogen-bonded OH stretching from cellulose, hemicellulose, and lignin was referred to as the broadband at around 3000–4000 cm^{-1}. The broadband at 3400–2850 cm^{-1} was credited to the C-H bond, commonly seen in alkane groups, whereas the intense peaks for C=O groups in ketone and carbonyl groups were indicated as peaks at 1730–1732 and 1450–1650 cm^{-1}, respectively, referring to hemicellulose and lignin compounds [25].

The spectrum of O-H absorption for coffee husk is around 3330.22 and 3317.98 cm^{-1} for treated and untreated coffee husk, respectively. Untreated coffee husk has a C-H stretching of 2913.82 cm^{-1}, and 10% NaOH-treated coffee husk has a stretching of 2910.88 cm^{-1}. The peaks at 1602.90 and 1602.66 cm^{-1} in untreated and alkali-treated coffee husk fiber were attributed to C=O stretching vibration of acetyl groups in hemicellulose compounds, as shown in Figure 3. The intensity of the peaks was not observed because coffee husk fibers were treated with 10% NaOH. As a result of the NaOH treatment, the hemicellulose components in the fiber are reduced.

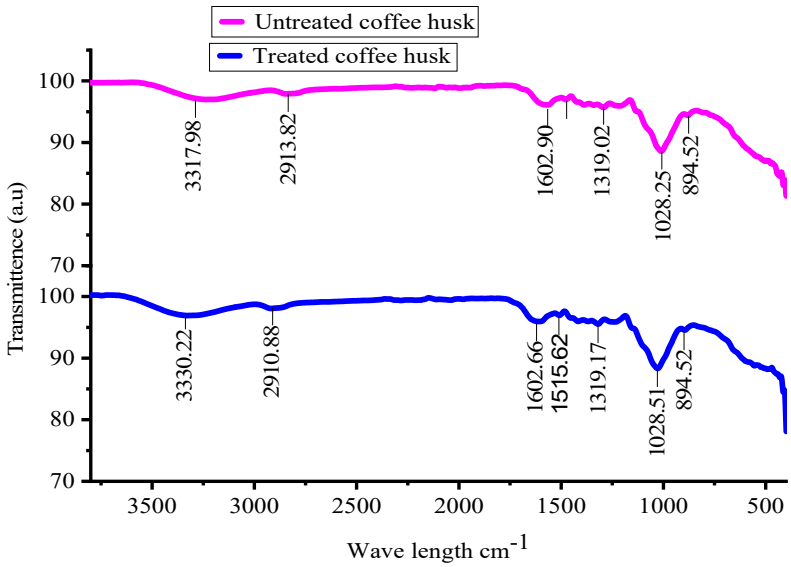


Figure 3. FTIR spectrums of untreated and alkali-treated coffee husk fiber.

The peaks 1460–1650 cm^{-1} represent the aromatic structure of lignin found in fiber. In untreated coffee husk fiber, the peak 1602.90 cm^{-1} can be found. The fact that the strength of the peaks dropped after NaOH treatment proves that lignin was lowered. C-H bending of hemicellulose, O-H stretch of cellulose, and C-O stretching of the NaOH group are all visible at 1300–1500 cm^{-1}. The peaks at 1319.02 and 1515.62 cm^{-1} are caused by acetyl (lignin) C-O stretching and intense elimination by a 10% NaOH chemical treatment. Carbon–hydrogen bending of hemicellulose compound is responsible for the peaks at 1319.02 and 1319.17 cm^{-1}, which were eliminated by NaOH treatment. The hydrogen bond group in cellulose and the occurrence of b-glyosidic of the monosachorides on untreated coffee husk fiber can be attributed to at the peak 1028.51 cm^{-1} and 1028.26 cm^{-1}. Similarly, similar chemicals can be found on treated fiber, thus demonstrating that the treatment does not remove them entirely. The peaks at 3330 to 1028 cm^{-1} represent the stretching vibration of –O-H and C-O-C present in lignocellulosic materials at intervals in the coffee waste. The well-defined peaks at 2913 and 1319 cm^{-1} are the characteristic peaks for C-H bond stretching and bending vibrations, respectively [28,30,31]. Associate degree FTIR spectrum of Alkali-treated coffee husk provided in Figure 2, with the peak necessary amendment at intervals the spectra. The absence of the peak at 1319 cm^{-1} is attributed to uronic organic compound teams and shows a decrease in lignin and hemicellulose in the coffee husk [32,33].

The untreated CH–FTIR spectra, shown in Figure 3, show a distinct peak at 1319 and 1515 cm^{-1}, thus indicating the presence of –C-N bonds of aromatic amines attributed to untreated CH. Furthermore, the increase in peak intensity at 1602 cm^{-1} after the surface assimilation indicates an increase at intervals in the number of –C=C bonds resulting from the cyclic aliphatic compound [32].

4. Conclusions

This work presented a study on the physical and chemical characterization of 10% NaOH treated and untreated coffee husk fiber for composite materials production. Physical and chemical characteristics, such as density, porosity, moisture content, lignin, cellulose, hemicellulose, and ash, were investigated. Based on the findings of the above investigation, the following conclusion may be attained:

- The chemical properties of coffee husk fiber were significantly treated by NaOH for a better interfacial surface with the polymer matrices.
- The treated coffee husk shows greater reduction in lignin and hemicellulose, 73% and 52%, respectively.

- Water absorption is minimum for treated coffee husk than untreated coffee husk fiber.
- The FTIR information shows a reduction of lignin and hemicellulose, compared to an untreated husk due to chemical treatment.
- The peaks 1460–1650 cm^{-1} represent the aromatic structure of lignin found in fiber. In untreated coffee husk fiber, the peak 1602.90 cm^{-1} can be found. In untreated coffee husk fiber, the peak 1602.90 cm^{-1} can be found.
- The peaks at 1602.90 cm^{-1} and 1602.66 cm^{-1} in untreated and alkali-treated coffee husk fiber were attributed to C=O stretching vibration of acetyl groups in hemicellulose compounds
- Regression analysis of the data showed that the untreated coffee husk is the most water absorbed.
- The study's findings can then be used to build coffee husk fiber polymer composites for a variety of applications.

Author Contributions: The idea, experimental work, the analysis of data, and the writing throughout the work was carried out by B.T.A. Examination and correction and supervision throughout the work was carried out by H.A. and G.S.T. Correction of the manuscript, review and editing, proofreading, and funding acquisition by N.H. All authors have read and agreed to the published version of the manuscript.

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References

1. Bekele, A.E.; Lemu, H.G.; Jiru, M.G. Experimental study of physical, chemical and mechanical properties of enset and sisal fibers. *Polym. Test.* **2021**, *106*, 107453. [CrossRef]
2. Cangussu, L.B.; Melo, J.C.; Franca, A.S. Chemical Characterization of Coffee Husks, a By-Product of Coffea arabica Production. *Foods* **2021**, *10*, 3125. [CrossRef] [PubMed]
3. Franca, A.S.; Oliveira, L.S. Coffee processing solid wastes: Current uses and future perspectives. *Agric. Wastes* **2016**, *12*, 90–96.
4. Tolesa, L.D.; Gupta, B.S.; Lee, M. Treatment of Coffee Husk with Ammonium-Based Ionic Liquids: Lignin Extraction, Degradation, and Characterization. *ACS Omega* **2018**, *3*, 10866–10876. [CrossRef]
5. Calvo-flores, F.G.; Dobado, J.A. Lignin as Renewable Raw Material. *ChemSusChem* **2010**, *3*, 1227–1235. [CrossRef]
6. Lee, R.A.; Berberi, V.; Labranche, J.; Lavoie, J. Bioresource Technology Lignin extraction—Reassessment of the severity factor with respect to hydroxide concentration. *Bioresour. Technol.* **2014**, *169*, 707–712. [CrossRef] [PubMed]
7. Constant, S.; Basset, C.; Dumas, C.; Di Renzo, F.; Constant, S.; Basset, C.; Dumas, C.; Di Renzo, F.; Robitzer, M. *Reactive Organosolv Lignin Extraction from Wheat Straw: Influence of Lewis Acid Catalysts on Structural and Chemical Properties of Lignins To Cite This Version: HAL Id: Hal-01093037 Lewis acid Catalysts on Structural and Chemical Properties of Lignins*; Elseiver: Amsterdam, The Netherlands, 2018; Volume 65, pp. 180–189.
8. Serna, L.V.D.; Alzate, C.E.O.; Alzate, C.A.C. Bioresource Technology Supercritical fluids as a green technology for the pretreatment of lignocellulosic biomass. *Bioresour. Technol.* **2015**, *199*, 113–120. [CrossRef] [PubMed]
9. Fan, J.; Njuguna, J. An Introduction to Lightweight Composite Materials and Their Use in Transport Structures. In *Lightweight Composite Structures in Transport: Design, Manufacturing, Analysis and Performance*; Elsevier Inc.: Amsterdam, The Netherlands, 2016; pp. 3–34.
10. Catalán, E.; Komilis, D.; Sánchez, A. Environmental impact of cellulase production from coffee husks by solid-state fermentation: A life-cycle assessment. *J. Clean. Prod.* **2019**, *233*, 954–962. [CrossRef]
11. Collazo-Bigliardi, S.; Ortega-Toro, R.; Chiralt, A. Improving properties of thermoplastic starch films by incorporating active extracts and cellulose fibres isolated from rice or coffee husk. *Food Packag. Shelf Life* **2019**, *22*, 36–100. [CrossRef]
12. Vadivel, V.; Brindha, P. Antioxidant property of solvent extract and acid/alkali hydrolysates from rice hulls. *Food Biosci.* **2015**, *11*, 85–91. [CrossRef]
13. Feng, N.; Guo, X.; Liang, S. Adsorption study of copper (II) by chemically modified orange peel. *J. Hazard. Mater.* **2009**, *164*, 1286–1292. [CrossRef] [PubMed]

14. Wan Ngah, W.S.; Hanafiah, M.A.K.M. Removal of heavy metal ions from wastewater by chemically modified plant wastes as adsorbents: A review. *Bioresour. Technol.* **2008**, *10*, 3935–3948. [CrossRef] [PubMed]
15. Pehlivan, E.; Altun, T.; Parlayici, S. Modified barley straw as a potential biosorbent for removal of copper ions from aqueous solution. *Food Chem.* **2012**, *135*, 2229–2234. [CrossRef] [PubMed]
16. Selatnia, A.; Bakhti, M.Z.; Madani, A.; Kertous, L.; Mansouri, Y. Biosorption of Cd2+ from aqueous solution by a NaOH-treated bacterial dead Streptomyces rimosus biomass. *Hydrometallurgy* **2004**, *75*, 11–24. [CrossRef]
17. Naeem, H.; Bhatti, H.N.; Sadaf, S.; Iqbal, M. Uranium remediation using modified Vigna radiata waste biomass. *Appl. Radiat. Isot.* **2017**, *123*, 94–101. [CrossRef]
18. Ashrafi, S.D.; Kamani, H.; Mahvi, A.H. The optimization study of direct red 81 and methylene blue adsorption on NaOH-modified rice husk. *Desalin. Water Treat.* **2016**, *57*, 738–746. [CrossRef]
19. Chowdhury, S.; Mishra, R.; Saha, P.; Kushwaha, P. Adsorption thermodynamics, kinetics and isosteric heat of adsorption of malachite green onto chemically modified rice husk. *Desalination* **2011**, *265*, 159–168. [CrossRef]
20. Ebrahimian Pirbazari, A.; Saberikhah, E.; Badrouh, M.; Emami, M.S. Alkali treated Foumanat tea waste as an efficient adsorbent for methylene blue adsorption from aqueous solution. *Water Resour. Ind.* **2014**, *6*, 64–80. [CrossRef]
21. Ferreira, D.P.; Cruz, J.; Fangueiro, R. *Surface Modification of Natural Fibers in Polymer Composites*; Elsevier Ltd.: Amsterdam, The Netherlands, 2019; Volume 276, pp. 13–23.
22. Rohit, K.; Dixit, S. A Review—Future Aspect of Natural Fiber Reinforced Composite. *Polym. Renew. Resour.* **2016**, *7*, 43–59. [CrossRef]
23. Kabir, M.M.; Wang, H.; Lau, K.T.; Cardona, F. Applied Surface Science Effects of chemical treatments on hemp fibre structure. *Appl. Surf. Sci.* **2013**, *276*, 13–23. [CrossRef]
24. Ajouguim, S.; Abdelouahdi, K.; Waqif, M.; Stefanidou, M.; Saâdi, L. Modifications of Alfa fibers by alkali and hydrothermal treatment. *Cellulose* **2019**, *26*, 1503–1516. [CrossRef]
25. Bekele, A.E.; Lemu, H.G.; Jiru, M.G. Exploration of Mechanical Properties of Enset—Sisal Hybrid Polymer Composite. *Fibers* **2022**, *10*, 14. [CrossRef]
26. Gonçalves, B.M.M.; de Oliveira Camillo, M.; Oliveira, M.P.; Carreira, L.G.; Moulin, J.C.; Neto, H.F.; de Oliveira, B.F.; Pereira, A.C.; Monteiro, S.N. Surface treatments of coffee husk fiber waste for effective incorporation into polymer biocomposites. *Polymers* **2021**, *13*, 3428. [CrossRef] [PubMed]
27. Pratap, G.K.; Shantaram, M. Green synthesis of silver nanoparticles (Ag-nps) from olea dioica roxb., leaf extracts and its biological activity. *Iforest-Biogeosci. For.* **2019**, *39*, 544–549. [CrossRef]
28. *TAPPI T211 T211*; Ash in Wood, Pulp, Paper and Paperboard: Combustion at 525 °C. TAPPI Test Methods. TAPPI: Atlanta, GA, USA, 2007; Volume T211 om–02, p. 5.
29. Guo, J.Z.; Li, B.; Liu, L.; Lv, K. Removal of methylene blue from aqueous solutions by chemically modified bamboo. *Chemosphere* **2014**, *111*, 225–231. [CrossRef]
30. Gebresemati, M.; Gabbiye, N.; Sahu, O. Sorption of cyanide from aqueous medium by coffee husk: Response surface methodology. *J. Appl. Res. Technol.* **2017**, *15*, 27–35. [CrossRef]
31. Zhu, G.; Xing, X.; Wang, J.; Zhang, X. Effect of acid and hydrothermal treatments on the dye adsorption properties of biomass-derived activated carbon. *J. Mater. Sci.* **2017**, *52*, 7664–7676. [CrossRef]
32. Kocaman, S.; Karaman, M.; Gursoy, M.; Ahmetli, G. Chemical and plasma surface modification of lignocellulose coconut waste for the preparation of advanced biobased composite materials. *Carbohydr. Polym.* **2017**, *159*, 48–57. [CrossRef]
33. Huang, R.; He, L.; Zhang, T.; Li, D.; Tang, P.; Zhao, Y.; Feng, Y. Fabrication and adsorption behavior of magnesium silicate hydrate nanoparticles towards methylene blue. *Nanomaterials* **2018**, *8*, 271. [CrossRef]