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Effect of Process Variables on the Composition of Chemically Modified *Nauclea latifolia*Wood Bark Fiber for Paper Production

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Abstract

The shortage of sustainable fiber sources in the chemical industry highlights the need for alternatives to synthetic fibers, which have been widely used for years. This study examined the effect of NaOH treatment on the chemical composition of a novel fiber obtained from Nauclea latifolia bark wood (NLWBF). The objective was to investigate the influence of NaOH concentration and soaking time on NLWBF. Soaking time and NaOH concentration were varied from 1.2 to 3.6 hours and 3.4 to 10.2 % w/v, respectively. After collection, the NLWBF was processed into powdered form and treated to remove impurities. Both modified and unmodified NLWBF samples at optimum treatment conditions were structurally analyzed using Fourier Transform Infrared Spectroscopy (FTIR) to identify the organic functional groups present. The initial cellulose (CL), hemicellulose (HM), and lignin (LN) contents of NLWBF were 36.83%, 29.71%, and 31.29%, respectively. After chemical modification, the maximum cellulose content of 69.06% was obtained at 3.6 hours and 6.8% w/v NaOH. The optimum hemicellulose content was 18.14%, recorded at 2.4 hours and 6.8% w/v. FTIR spectra revealed the presence of OH, –COOH, C=O, C–O, and C–O–C groups, with noticeable shifts after NaOH treatment, confirming chemical modification. These changes indicated a reduction in hemicellulose and lignin, thereby enhancing the cellulose content of NLWBF. The results demonstrate that NaOH-treated NLWBF, with its improved cellulose composition, holds strong potential as a sustainable raw material for industrial paper.

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1.0 Introduction

Natural fibers derived from plants have been widely applied in various industrial sectors, including paper making, tissue production, polymer composites, and motor components (Netral et al., 2012; Dungani et al., 2016; Shuhaida & Soh, 2016; Naidu et al. 2013; Naidu et al. 2017). In recent times, these fibers have become essential raw materials for innovative product development, particularly in composite materials worldwide (Obasi, 2015). Other notable applications of natural wood composites include electronic packaging and building materials (Oushabi et al., 2017; Indu et al., 2017). Wood fibers, often considered waste, serve as alternatives to mineral fibers and can be utilized in finished products (Government et al., 2019).

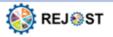
The primary component of wood fiber is cellulose, while hemicellulose (HM) and lignin (LN) are present in smaller proportions. These non-cellulosic components often need to be removed to minimize compatibility issues in composite production (Reddy *et al.*, 2013; Oushabi *et al.*, 2017). Non-uniformity of fibers, combined with the presence of these components, can result in high water absorption in composites over time (Government *et al.*, 2017; Government *et al.*, 2018).

Traditionally, composites have relied on oxides and carbonates of metals or synthetic fibers, but these materials present drawbacks such as heavy weight, high cost, complex processing requirements, and limited accessibility (Komuraiah et al., 2014; Government et al., 2019). To improve fiber–polymer matrix compatibility, cellulosic fibers must pretreated to enhance their strength interfacial adhesion (Government et al., 2019). The efficiency of composite production is strongly related to the cellulose content of the fiber (Oushabi et al., 2017; Palamae et al., 2017; Shuhaida & Soh, 2016; Barlianti et al., 2015).

Pretreatment plays a crucial role in partially eliminating unwanted constituents in fibers, enhancing the major components while minimizing the minor ones. This makes fiber modification highly important. Common modification methods include alkalization, acetylation, and bleaching, among others. Among these, alkalization remains the most widely used process. It involves introducing alkali into water to form an aqueous solution for treating fibrous materials. Typical alkalizing agents used to improve compatibility and increase cellulose (CL) content include KOH, NaOH, Ca(OH)2, and Mg (OH)2 (Naidu et al., 2013; Obasi, 2015; Naidu et al., 2017; Lazić et al., 2017).

Several studies (Oushabi et al., 2017; Palamae et al., 2017; Shuhaida and Soh, 2016; Barlianti et al., 2015) have reported increases in cellulose content and reductions in lignin as a result of chemical treatment of wood fibers. According to scholarly works, numerous experiments have been conducted on various fibers for commercial applications. Examples include pinewood (Chen et al., 2014), rice straw (Shuhaida and Soh, 2016), flax fiber (Lazić et al., 2017), century fiber (Reddy et al., 2013), sugarcane bagasse (Ahmadi et al., 2016), waste betel nut husk fiber (Sari et al., 2017), Pterocarpus angolensis (wukwa) (Setswalo et al., 2017), oil palm empty fruit bunch (Palamae et al., 2017; Muryanto et al., 2015), mendong straw (Suryanto et al., 2013), mango seed shell fiber (Government et al., 2020b), and shea butter wood bark (Government et al., 2019; Government et al., 2020a; Okeke et al., 2023).

Novel non-woody lignocellulosic biomass fiber (NLWBF) remains scarce in scholarly research, which motivates its investigation in this study. The research evaluates NLWBF as an inexpensive and renewable alternative fiber, capable of competing with conventional fibers reported in the literature for large-scale paper production. To achieve this, the NLWBF was processed and its components - lignin (LN),



hemicellulose (HM), and cellulose (CL) were analyzed both before treatment and after modification with sodium hydroxide (NaOH) at varying concentrations and durations. The optimized composition, obtained at the highest concentration and treatment time, was further characterized using Fourier Transform Infrared Spectroscopy (FTIR). The findings demonstrate the potential of NLWBF for commercialization in the paper manufacturing industry.

2.0 Materials and Methods

2.1 Material Collection

The NLWBF was obtained from the Federal Government College, Wukari, Taraba State. The sample was washed with distilled water, sundried for 10 days (8 hours daily), crushed into a semi-powder form using a mortar, and sieved through a 20-mesh (850 µm) sieve.

2.2 Pre-treatment of NLWBF

The NLWBF was crushed, sieved, and weighed (20 g, 20 g, and 15 g) for the raw and modified fiber compositions, respectively. The samples were soaked in NaOH solution at varying concentrations of 3.4, 6.8, and 10.2 % (w/v). After soaking, the solid fraction was rinsed several times with distilled water at pH 7 to remove residual NaOH. The immersion times were varied as 1.2, 2.4, and 3.6 hours. The moisture in the mercerized NLWBF was removed by oven-drying at 105 °C for 7 hours, after which cellulose (CL), hemicellulose (HM), and lignin (LN) contents were analyzed (Muryanto et al., 2015).

2.3 Determination of CL and LN Content

The untreated and pre-treated NLWBF samples were analyzed for lignin (LN) and cellulose (CL) content under varying NaOH concentrations and immersion times using Chesson's method (Mahyati *et al.*, 2013; Government *et al.*, 2019; Government *et al.*, 2020b; Okeke *et al.*, 2023), as follows:

- (F) 1 g of dried NLWBF was dissolved in 150 mL of distilled water. This was boiled at 100 °C for 1 hour and reweighed.
- **(G)** The mixture (dried NLWBF and distilled water as in step F) was dried, filtered, and weighed until a constant weight was achieved.
- **(H)** The residue from step F was treated with 150 mL of 0.5 M H₂SO₄, heated at 100 °C for 1 hour, filtered, and reweighed.
- (I) The residue was mixed with 10 mL of 72 % (w/v) H₂SO₄ for 4 hours at room temperature, followed by heating for 1 hour, after which the weight was recorded.
- (J) The final residue was incinerated to ash, and the final weight was measured.

The cellulose and lignin contents of NLWBF were then calculated using Eq. (1) and Eq. (2), respectively.

CL content (%) =
$$\frac{(H-I)}{F} \times 100$$
 (1)

LN content (%) =
$$\frac{(I-J)}{F} \times 100$$
 (2)

2.5 Determination of HM content

A 2 g portion of the NLWBF residue was measured and placed in a beaker containing 10 ml of NaOH. The mixture was agitated at 300 rpm using an orbital shaker to ensure thorough mixing. NaOH was periodically injected into the mixture at 20 °C. Subsequently, 33 ml of distilled H₂O was added to the beaker, and the mixture was allowed to stand for 1 hour. The residue was then weighed (F). Afterward, the residue was transferred to a cubicle, washed sequentially with 100 ml of NaOH, 200 ml of distilled water, and 15 ml of acetic acid. The cubicle containing the residue was dried and weighed (G). The heavy metal (HM) content of NLWBF was determined using Eq. (3).

$$HM\ content\ (\%) = \frac{residual\ weight\ (F) - residual\ weight\ in\ cubicle\ (G)}{residual\ weight\ (F)} \times 100\% \tag{3}$$



2.6 FTIR analysis

The modified and unmodified NLWBF samples at optimum conditions were analyzed using a SHIMADZU 8400s FTIR spectrometer. A mixture of 50 mg of KBr and 1.5 mg of NLWBF was finely ground in a mortar and pressed into a pellet using a die. The pellet was then placed in the FTIR spectrometer and scanned. The instrument was equipped with a TGS detector, and the scanning frequency was maintained at 0.1 cm⁻¹. Spectra were recorded in transmission mode with a resolution of 4 cm⁻¹, over a range of 800 – 3500 cm⁻¹.

3.0 Results and Discussion

Table 1 presents the chemical composition of NLWBF for the unmodified fiber. The cellulose (CL), hemicellulose (HM), and lignin (LN) contents were 36.83%, 29.71%, and 31.29%, respectively. The cellulose and lignin values are comparable to those of coconut coir (36.62–43.21%) (Agu *et al.*, 2014; Dungani *et al.*, 2016; Okeke *et al.*, 2023) and oil palm fibers (18.30–33.55%) (Agu *et al.*, 2014; Dungani *et al.*, 2016; Government *et al.*, 2019). These results suggest that NLWBF possesses suitable characteristics for papermaking, making it a promising alternative material compared to previously studied fibers

Table 1: Composition of NLWBF

CL content (%)	HM content (%)	LN content (%)
36.83	29.71	31.29

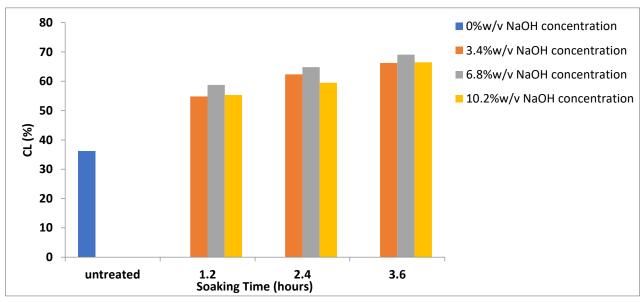


Figure 1: Effect of NaOH on the CL content of NLWBF

Figure 1 presents the effect of NaOH modification parameters on the cellulose (CL) content of NLWBF. At a constant soaking time of 1.2 hr., the CL content of NLWBF increased from 38.68% to 55.32% as the NaOH concentration rose from 0 to 10.2%. This enhancement is attributed to the weakening of the bonding forces within the fiber matrix when immersed in NaOH solution, which facilitates

the removal of impurities. Similarly, at a NaOH concentration of 6.8%, the CL content improved from 36.83% to 69.06% as the soaking time increased from 0 to 3.6 hr. The observed improvement in cellulose content with extended soaking time is due to the progressive removal of impurities from the fiber. The maximum CL content (69.06%) was recorded at 3.6 hour and 6.8% w/v NaOH concentration. The findings



from this study are consistent with results reported in previous studies (Government et al., 2020a; Government et al., 2020b; Government et al., 2019; Oushabi et al., 2017; Palamae et al., 2017; Shuhaida & Soh, 2016; Barlianti et al., 2015; Wang et al., 2016; William & Patrik, 2017). Figure 2 shows the effect of NaOH pretreatment on the holocellulose (HM) content of NLWBF. The HM content decreased from 29.71% in the raw NLWBF to 21.20% with an increase in NaOH concentration from 0 to 10.2% w/v at 1.2 hr. A similar reduction was observed at 2.4 hr. At constant concentration, the HM

content of NLWBF showed a steady decrease from 0 to 3.6 hr. The optimum HM content was obtained at 2.4 hr and a concentration of 6.8% w/v, corresponding to a reduction of 18.14%. is attributed reduction NaOH modification, which minimizes gummy components associated with NLWBF. Comparable findings have been reported in previous studies (Marwan & Nasim 2016; Sari et al., 2017; Government et al., 2019, 2020a, 2020b; Oushabi et al., 2017; Palamae et al., 2017; Shuhaida & Soh, 2016; Barlianti et al., 2015).

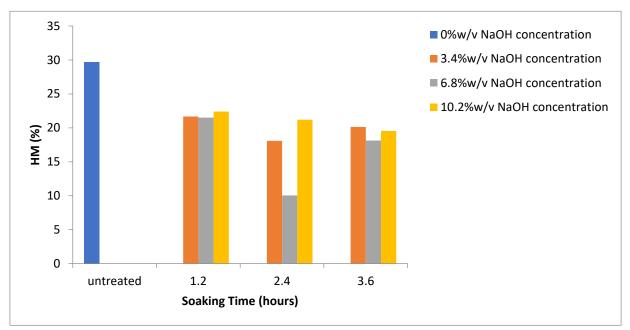


Figure 2: Effect of NaOH on the HM content of NLWBF

Figure 3 illustrates the effect of NaOH parameters on the lignin (LN) content of NLWBF. As shown, the LN content decreased sharply from 31.29% to 12.79% when the NaOH concentration increased from 0 to 6.8% w/v. A similar reduction in LN content was observed at soaking times of 1.2 h and 2.4 h, respectively. Furthermore, at a constant concentration, prolonged soaking time further enhanced the reduction of LN content in NLWBF. These trends are consistent with the influence of process parameters on the cellulose content of NLWBF. Comparable findings have also been reported by other researchers (Lazic et al. 2017; Government *et al.*, 2020a; Government *et al.*,

2020b; Government *et al.*, 2019; Oushabi *et al.*, 2017; Palamae *et al.*, 2017; Shuhaida & Soh, 2016; Barlianti *et al.*, 2015; Brinchi *et al.*, 2013). Figure 4 presents the FTIR spectra of raw and NaOH-treated NLWBF. The peak observed at 3431 cm⁻¹ and 2962 cm⁻¹ corresponds to –OH and –COOH groups, indicating the presence of phenolic and carboxylic compounds in the unmodified NLWBF. A peak at 1730 cm⁻¹, attributed to lignin (LN) and hemicellulose (HM), is associated with C=O and C–O stretching in the raw fiber. Additional peaks at 1410 cm⁻¹ and 1230 cm⁻¹ were assigned to N=N and C–O ester groups, corresponding to azo and aliphatic ester stretching, respectively. The



chemically purified NLWBF exhibited a peak at 1016 cm⁻¹, representing C–O alcohol/phenol stretching, which coincides with alcohol and phenolic groups. These signals confirm the presence of impurities in the raw fiber, including pectin, rennin, and fractions of HM and LN. Peaks at 1095 cm⁻¹ and 897–724 cm⁻¹ were attributed to C–O–C asymmetric stretching and C–H plane vibrations in the cellulose component of raw NLWBF. After NaOH modification, most of the peaks present in the untreated fiber

diminished or disappeared, and noticeable shifts in peak positions were observed. This indicates the removal of inefficient components and impurities, enhancing the purity of the NLWBF. The treatment improved the cellulose (CL) content, making the NLWBF more suitable for industrial applications. Similar findings have been reported in previous studies (Chen *et al.*, 2014; Ikranullah *et al.*, 2018; Oushabi *et al.*, 2017; Government *et al.*, 2020a; Government *et al.*, 2020b).

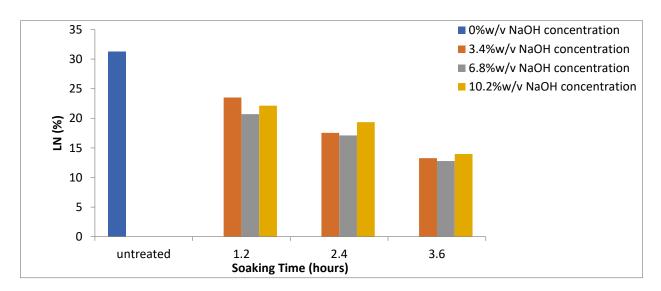


Figure 3: Effect of NaOH on the LN content of NLWBF

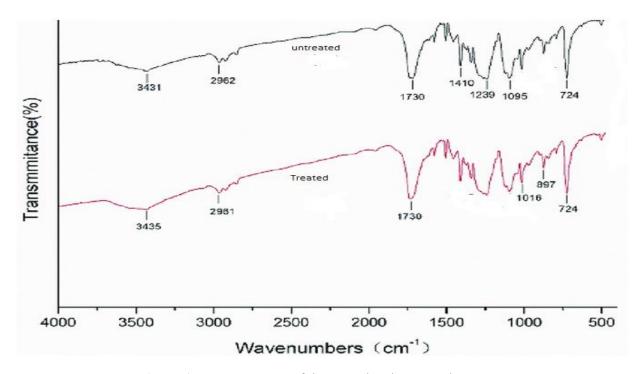


Figure 4: FTIR spectrum of the treated and untreated NLWBF



4.0 Conclusion

The modification of compositional components of novel NLWBF for papermaking was investigated. Alkaline modification of NLWBF enhanced the CL content and partially reduced other components. The chemical compositions of both the modified and unmodified NLWBF were established. Pre-treatment variables significantly influenced the chemical composition of NLWBF. Chemical treatment improved the cellulose content while reducing the hemicellulose (HM) and lignin (LN) fractions. FTIR analysis confirmed enhancement of the cellulosic components through observable alterations in the structural arrangement. The best cellulose content was obtained at higher concentration of 6.9v/w % NaOH and lower time of soaking at 3.6 hrs. These results demonstrate that NLWBF is a promising alternative raw material for largescale paper production.

Recommendation

The results derived in this research demonstrate that NLWBF at optimum state is an unconventional material in paper production.

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