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Effects of Sodium Hydroxide on the Compositions of Shea Butter Bark Wood Fiber for Polymeric Composite Production in Structural Application

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ABSTRACT: The efficiency of natural fiber rely on pre-treatment for the reduction of unwanted components in its accessibility for industrial application. This study investigates the effects of sodium hydroxide on the chemical compositions of shell butter bark wood fiber (SBBWF). The SBBWF was pre-treated with NaOH at 3, 6 and 9 wt% for 18 hours. The SBBWF for untreated and treated were subjected to gravimetric method and fourier transform infra-red spectrometer (FTIR) to determine the chemical composition and organic functionality present, respectively. The changes in the prevalent functional group of the alkali treated and untreated SBBWF were also investigated. After treatment, the improvement of cellulose and mimimization of the hemicellulose and lignin content was noticed. Results from the elemental compositional analysis indicated that SBBWF contained high cellulose content (33.89%) followed by hemicellulose (33.67%) and lignin (8.66%). The optimum cellulose, hemicellulose and lignin contents were obtained at 3%wt and 9 wt% sodium hydroxide, respectively. The FTIR result showed that the modification of the SBBWF as a disappearance in functional group was observed.The disappeared functional groups were present in untreated SBBWF and positional change in the transmittance with sodium hydroxide treated SBBWF. Finally, the shea butte bark wood fiber is highly recommended for biocomposite production in structural application.

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Extensive research over the years has established that wood fiber has great potentials in the production of composites due to its high cellulose content(Government *et al*., 2021; Government *et al*., 2020; Mike *et al*., 2020). Natural fiber sourced from wood are generally easy to treat, cheaper offers better dimensional stability in comparision with mineral and inorganic fillers, the high cellulose content in wood fiber could serve as suitable reinforcement in the production of biocomposites (Government *et al* 2021). The structure of hydrogen bond in wood fiber provides high resistance to microbial attack and high tensile strength (Laka *et al*., 2015). The main composition of natural fiber is cellulose, hemicellulose and lignin (Goda et *al* 2006; Haque *et al*., 2019; Matsunaga *et al*, 2018). The first serves as a booster to the properties of a polymer when used for composite manufacturing, while the other two is taken to be gelly-like content which is originally linked contaminants in the fiber (Ahmadi *et al*., 2016; Ikramullah *et al*., 2018). There is the need to increase the major component of fiber (cellulose) and partially eradicate those natural generated unwanted part of the fiber (hemicelluloses and lignin) (Palamae *et al*., 2017; Reddy *et al*., 2013) However, lignocelloluse from wood fibers needs various forms of treatment to enhance and modify their usability in various industrial aplication. Alkali treatment is one of the widely established method for

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cellulose preparation (Government *et al*., 2021). The alkali treatment process entails subjecting the wood fiber in an interaction with a basic solution, which is aimed at removing impurities and reducing the lignin and hemicellulose cotents (Laka *et al*., 2015). SBBWF is sourced from a shea butter wood bark tree (Akihisa *et al*., 2010). Shea butter trees are predominantly utilized for herbal drugs in Nigeria (Government *et al*., 2020). The part of the tree can be applied as additives for biocomposite. It is advisable before employing SBBWF for viable venture; it is better to immerse it on chemical medium on the basis of improving cellulose content using alkaline treatment process (Government *et al*., 2018; Government *et al*., 2021, 2020). Several investigations have carried out to fast-track the cellulosic content of species for lignocelluloses fiber by chemical method in the fiber-polymer matrix composite for marketable application. These involve oil palm (Fahma *et al*., 2010), wheat straw (Sun *et al*., 1995), rice straw (Harun and Geok, 2016; Kim and Han, 2012) and jute (Jahan *et al*., 2011), etc. But SBBWF has not be been worked upon on any scholarly report. Accordingly, this work investigates the effects of sodium hydroxide concentrations on elemental compositions (cellulose, hemicellulose and lignin contents) of shea butter bark wood fiber (SBBWF).

MATERIALS AND METHODS

Processing of SBBWF: The research took place in Wukari Taraba State, Nigeria from May 16, 2017 to August 15, 2017. The organic fiber used for this research work is shea butter bark wood fiber (SBBWF) (Vitellaria paradoxa), which was obtained from Tella, Gassol Local Government Area, Taraba State Nigeria. The fibers bark from the shea butter wood tree was pilled, cut into pieces and sundried for 8 weeks after, which it was manually crushed with local mortar and pestle to obtain a smaller size. It was further ground into a fine particle size using an electric blender (Vitamix A3500), after which it was sieved using 850 µm particle size sieve. The alkaline or mercerization treatment was conducted using 10 grams of the prepared SBBWF fiber at Federal University Chemical Laboratory. The SBBWF fiber was soaked in various NaOH concentrations (3%wt, 6%wt, and 9%wt) for 18 hours in a plastic container, which was aimed to enhance its modification. After which it was rinsed with plenty of distill water and allowed to settle. The supernatant of the solution was removed using a transparent filter cloth to separate the fillers from impurities. In order to reduce the moisture content, the SBBWF was oven dried at 90˚C for 17 hours. However, the process was repeated for the untreated fiber but without alkaline treatment in order to ascertain the cellulose, hemicellulose, and lignin contents of the treated and untreated fiber. The untreated and treated SBBWF are shown in Figure 1 and 2, respectively.

Fig 1: Untreated SBBWF

Fig 2: Treated SBBWF

The untreated and treated SBBWF: Figure 1 and 2 present the untreated and treated SBBWF sample, respectively. It was depicted that appearance, more roughness and clustering of the SBBWF increased before and after modification were seen in Figure 1 and Figure 2, respectively. This occurrence was attributed to better intermingling and stickness of the SBBWF after immersion in aqueous sodium hydroxide as described by Matsunaga *et al*.(2018); Ikramullah *et al*.(2018); Reddy *et a*l.(2013); Sun *et al* .(1995) ; Kim and Han (2012).

Cellulose Content Determination: 10g of dried sample of the NaOH treated SBBWF was added to a solution containing nitric acid and ethanol mixture (95% purity). The mixtures were filtered, using whatman filter paper before rinsing the residue with warm water. This was in order to eliminate the residual acid. The residue was oven dried to constant weight (P_1) at 100˚C(Mahyati *et a*l., 2013). The cellulose content in the SBBWF was calculated using Eq. (1)

$$\%\text{cellolose content} = \frac{P_1}{P} \times \frac{100}{1} \tag{1}$$

Where P_1 is the weight after oven dried and P is the weight of the SBBWF used.

Determination of the Hemicellulose Content (Neutral detergent fiber method): A reflux quantity was used in preparing the natural detergent fiber 10g of the air dried NaOH treated SBBWF was mixed with 50ml sodium lauryl sulphate solution. The mixtures were filtered and the residue rinsed with warm distilled water. The residue was dried in an oven at 100˚C for 8 hours, after which the neutral detergent fiber weight of SBBWF was determined (Mahyati *et al.*, 2013). The difference in weight of the neutral detergent fiber weight of SBBWF and the acid detergent fiber weight of SBBWF as expressed in Eq. (2) was used to determine the hemicellulose content.

$$\%\ \text{hemicellose} = \frac{\text{NDSBBWF}-\text{ADSBBWF}}{\text{P}} \times \frac{100}{1} \quad (2)$$

Where NDSBBWF is the neutral detergent fiber weight of SBBWF and ADSBBWF is the acid detergent fiber weight of SBBWF, which was obtained from the hydrolysis of the same mass of treated SBBWF sample.

Determination of the Lignin Content: 10g of treated SBBWF was heated with 5ml of 72% w/w H_2SO_4 solution for 4hours to hydrolyze the cellulose and hemicelluloses. After the hydrolysis, precipitate was filtered, rinsed with warm distilled water and ethanol to remove acid present. The solid deposit was weighed after oven-dried at 105 ˚C for 24 hours. This filtrate left is called the acid detergent SBBWF. The filtrate later transferred to a constant pre-weighed porcelain crucible and heated at 600 ˚C for 5 hours. After cooling, it was weighed and ash content was determined(Mahyati et al., 2013). The percentage lignin was calculated using Eq (3)

$$\%\text{lignin content} = \frac{P_2 - P_3}{P} \times \frac{100}{1}. \quad (3)$$

Where P_2 is the weight of deposit at 105 ˚C after cooling; P_3 is the weight of residue at 600 ˚C after cooling; P is the weight of the SBBWF used.

FTIR test: The FTIR properties were tested using FTIR spectrometer model 8400 S. A mixture of 50mg potassium bromide salt and 1.5mg SBBWF was injected in the device. The wavelength of spectrums, which was equivalent to prevalent functional groups of the tested samples were recorded.

Statistical analysis: The experimental data was statistically examined using design expert version 7.0 for the analysis of variance (ANOVA) and results were significant at 0.05 probability value.

RESULTS AND DISCUSSIONS

Elemental Compositions of SBBWF: Table 1 shows the elemental compositions (cellulose, hemicellulose and lignin contents) for SBBWF. As shown in Table 1, the SBBWF contained higher proportions of cellulose (33.89%) when compared with hemicellulose and lignin of 33.67% and 6.66%, respectively. Similarly, Mohanty *et al.* (2000) reported that bagasse has cellulose content of 28.3 to 55%, hemicellulose content of 20 to 36.3% and lignin content of 7 to 13%, respectively. These values entailed that the SBBWF has similar compositional properties of other existing cellulosic material from plant. Therefore, SBBWF is potential material for composite reinforcement.

Table 1: Elemental Compositions of the Untreated SBBWF

	Cellulose content (%)	Hemicellulose content (%)	Lignin (%)
Untreated Natural fiber	33.89	33.67	6.66

Effect of NaOH concetrations on the celluolose, hemi cellulose and lignin contents Celluolose: According to Figure 3, it was observed that at 0% NaOH concetration, which indicated the untreated SBBWF with the cellulose content of 33.89%. Consequently, as the concentrations of NaOH increased from 0 to 3 and 6 to 9 wt%, the cellulose content improved from 33.89 to 67.112 %, and reduced from 53.33 to 41.332 %, respectively. Also, the high cellulose content of SBBWF correlated well with the increase in stiffness and strength of the natural plant fiber after pre-treatment (Thygesen *et al*, 2007). This observation could be that high concentration of NaOH destroys the cellulose content which led to the conversion of amorphous to crystalline cellulose (Vilay *et al.*, 2008). The results showed that the optimum cellulose content was obtained at 3% NaOH. This implies that at 3% NaOH concentration, impurities were removed from the fiber without damaging the fiber surface. Basically, the NaOH treatment process was carried out to enhance the interfacial properties of SBBWF (Oushabi *et al.*, 2017). This was mentioned by previous researcher (Matsunaga *et al.*, 2018; Ikramullah *et al.*, 2018; Reddy *et al.*, 2013) ; Kim and Han, 2012; Government *et al.*, 2021; Government *et al.*,2020).

Hemicellulose: Figure 4 summarizes the effects of NaOH concetrations on the hemicellulose content. The hemicellulose content of the untreated SBBWF as shown in Figure 4 was 25.676 %. As observed, at 3% wt NaOH, the hemicellulose reduced to 14.122 %. Also as the NaOH concetrations increased from 6% to 9%, the hemicellulose content reduced from 12.656 to 12.585 %, respectively. This reduction might be due to chemical modification of the fiber[22](Satyanarayana *et al.*, 1990). However, the high NaOH concentrations might be causing an increase in water absorption of SBBWF as a result of pores formation on the SBBWF.

The hemicellulose content suggest a reduced water absorption capacity as perious studies indicated that reduced water absorption capacity is responsible for poor mechanical prperties (Thygesen *et al.*, 2007; Government *et al*, 2020; Government *et al.*, 2021). This has been reported elsewhere (Government *et al.*, 2020; Government *et al.*, 2021; Reddy *et al*, 2013; Ikramullah *et al.*, 2018)

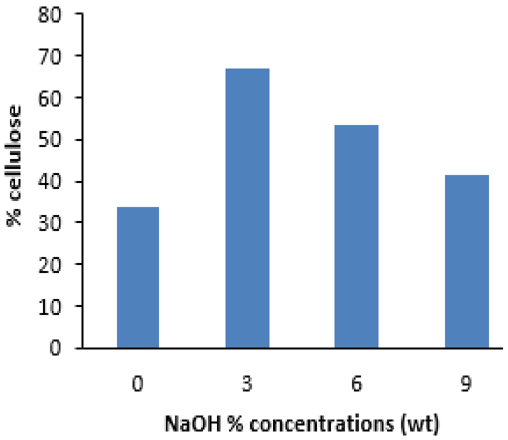

Fig 3: Effect of NaOH concentration on the %cellulose of the SBBWF.

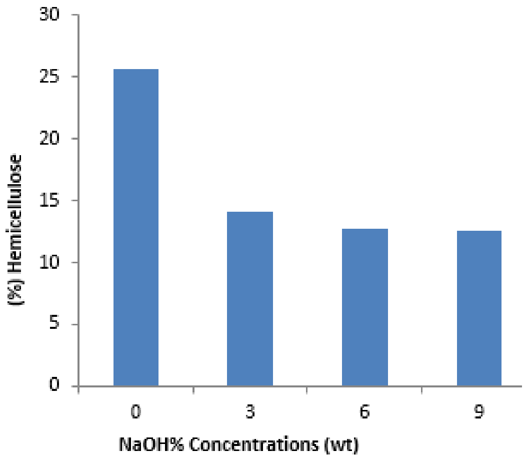

Fig 4: Effect of NaOH concentration on the % Hemicellulose of the SBBWF

Lignin: Figure 5 indicates the effects of NaOH concetrations on the lignin content. The percentage lignin content for the untreated SBBWF was 8.661% but reduced to 4.785 %, 4.042 % and 2.52 % at 3%wt, 6%wt and 9%wt, NaOH concentrations, respectively. This was due chemical modification of the SBBWF fiber (Xue *et al.*, 2007). Also, partial dissolution of the lignin and surface impurities due to the increase in NaOH concentration might be responsible for the reduced lignin content. In addition, an enhanced fiber structure of SBBWF with less wax was observed at 3 %wt compared with the untreated SBBWF (Setswalo *et al*, 2017). Also the neucleophilic reaction between the lignin and the high NaOH concentration results in fragmentation and dissolution of the lignin macromolecule. However, the observed low lignin content could also result in a change in the rigidity of the natural wood fiber. The low lignin content of SBBWF is also associated with amount of residual lignin, which is related to the kappa number (bleacheability of wood pulp for paper making) (Goda *et a*l., 2006). Therefore the lower the kppa number, the suitability of the wood fiber in paper making. This work has been stated by previous work (Government *et al*, 2020; Government *et al*, 2021; Reddy *et al*, 2013; Ikramullah *et al.*, 2018).

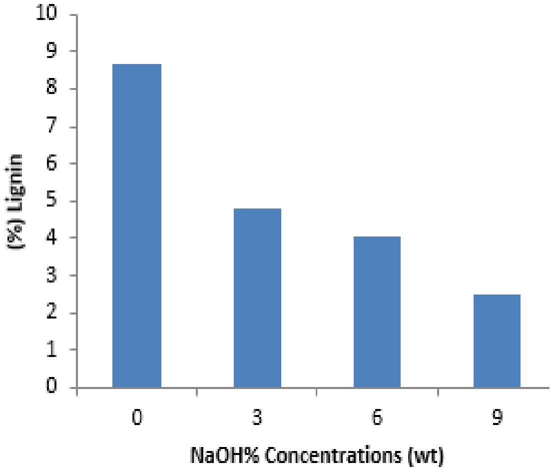

Fig 5: Effect of NaOH concentration on the % Lignin content of the SBBWF

FTIR Analysis of Untreated and NaOH Treated SBBWF: The broad spectrum of the untreated SBBWF indicated fourteen distinctive peaks with corresponding transmittance (%) as shown in Figure 6. The presence of carbonyl groups of fatty acid might be responsible for the sharp peaks observed at 3693.8 to 3265.1 cm^{-1}, which is a characteristic of the O-H bond. The sharp peak observed at 2922.1 $\text{cm}^{-1}$is assigned to the asymmetric deformation of lignin. The band located at 2113.4, 1982.9 and 1871.1 cm^{-1} represents the characteristics of the C=C bonding of aromatic benzene ring in SBBWF. An anhydride compound was domiciled at 1434.8 cm^{-1} allotted to C=O stretching. The peaks located at 1315.8, 1364.2cm^{-1} and 1606.5 cm^{-1} are assigned to $-CH_2$ in plane bending and skeletal vibration for stretching of benzene ring in lignin, respectively. The absorption band found at 1032.3 is a characteristics of C-O stretching vibration assigned to the hydroxyl group of hemicellulose, cellulose and lignin.

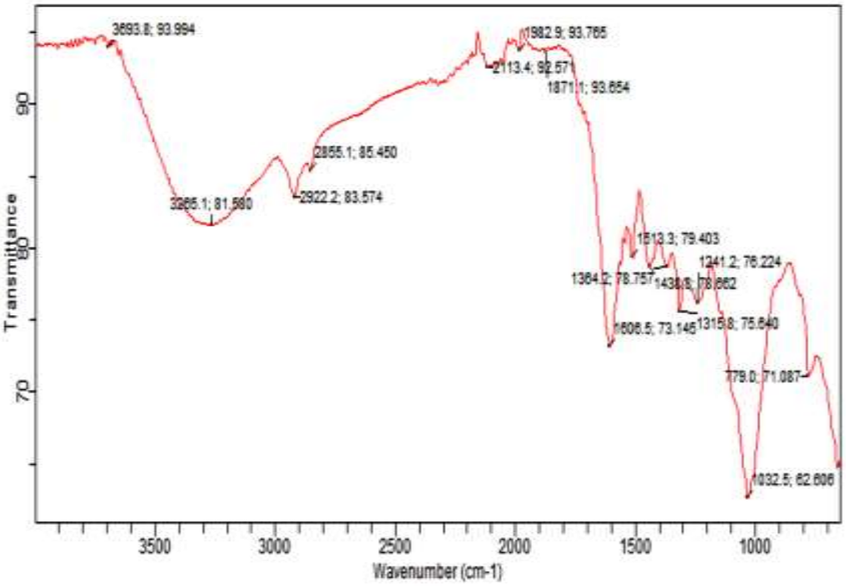

Fig 6: FTIR Spectrum for untreated SBBWF

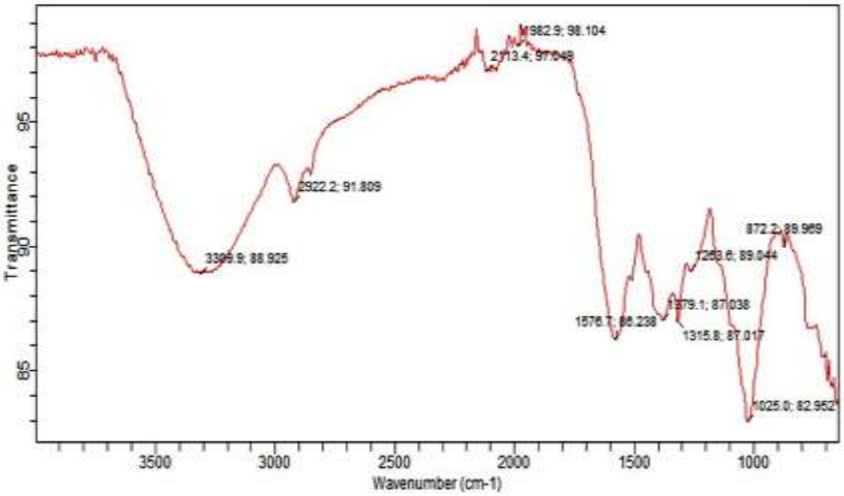

Fig 7: FTIR for NaOH treated SBBWF.

Figure 7 shows the FTIR spectrum of treated SBBWF. The spectrum pattern showed nine distinguishing peaks in the range of 3309.9 to 872.2cm^{-1} alongside their transmittance (%). The O-H bond reoccurred as a result of the SBBWF elongation after treatment with NaOH at rapid variation in the transmitting percentage. The lignin from C=C bond reduced after treatment with NaOH by increasing transmittance from 92.57 to 97.049% *(*Agarwal and Reiner, *2009;* Ikhlef *et al*., 2012). The absorption band located at 1315 to 1025.0cm^{-1} is a characteristic of C-C stretching and C-O stretching, which were attributed to the carboxyl group of hemicelluloses with also a change in the peak transmittance (Ismail *et al*., 2002). In comparision, it was observed that the peak involving C=O radical in the untreated SBBWF dissapperared and high disparity in the percentage transmittance (O-H and C-O groups from 93.995 to 88.995% and 62.606 to 88.995%) were noted after treatment with NaOH indicating that the alkali might have modified the interfacial properties of the SBBWF, respectively (Oushabi *et al*., 2017). This finally results in the improvement of cellulose and reduction of hemicelluloses and lignin component of SBBWF. The properties of modified SBBWF will enhanced such as tensile strength and modulus for the applicability in composite production. Similarly, Rezour et al. (2010) reported that the composite of treated rice husk in alkali media had high tensile strength. This result maintains similar range as existing works (Government *et a*l., 2020; Government *et al*, 2021; Reddy *et al*., 2013; Ikramullah *et al*., 2018; Oushabi *et al*., 2017).

Conclusions: The elemental compositions of the SBBWF showed that SBBWF had higher content of cellulose in comparison with the hemicellulose and lignin contents. The high cellulose content \was associated with increase in tensile strength and stiffness resulting in dimensional stability of the fiber. FTIR spectra pattern was to characterize the treated and untreated SBBWF. The FTIR showed disappearance of C=O group and change in transmittance (%) in the treated SBBWF. The NaOH treatment enhances cellulosic content and reduced other constituent of SBBWF.

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