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Effect of Alkali Treatment on the Composition of Mango Bark Wood Fiber for Structural Composite Application

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ABSTRACT
The modifications of mango bark wood fiber (MBWF) for the enhancement of its cellulosic component through alkali treatment was investigated in this work. The MBWF was modified using NaOH and the compositional content with regard to cellulose, hemicelluloses and lignin content were determined by chesson's method. The surface morphology and the established functional groups of the MBWF were investigated by employing the SEM and FTIR analysis, respectively. Results from the treatment process indicated that NaOH concentration of 12 wt% and time of 1.5 hour was observed to improve the cellulose content of the MBWF. The 12 wt% NaOH concentration was observed as the optimum at which the reduction in lignin and hemicellulose contents with corresponding increase in cellulose content were observed. The SEM analysis shows morphological changes of the treated MBWF fiber as a result of the NaOH treatment. The FTIR shows the prevalent functional group compositions that are associated with lignin, hemicellulose and cellulose in the MBWF. In this context, the MWBF indicated a prospect wood fiber in biocomposite application.

Kewwords: Mango Bark Wood Fiber, FTIR, SEM, Alkali treatment, composition

I. INTRODUCTION

The urgent need to replace synthetic fiber with readily available low cost natural fiber is steadily on the increase. The natural fiber consists of plant biomass of different kinds [1]. Thus, research interest has shifted to finding a cheaper lignocellulose natural fiber material that could be used in bio composite production.

Natural fiber with elemental compositions of lignin, hemicellulose and cellulose is a potential material for lignocellulose fiber The high cellulose content of most plant lignocellulose fiber is attractive to bio composite production. The dimension and structure of the hydrogen bond in the cellulose content in plant lignocellulose fiber is responsible for high resistance to microbial and termite attack and high tensile and elongation attack [2].

Recently, many researchers have examined the different modification methods of lignocellulose fiber that aims to improve the fiber matrix for bio composite production[3]. These include bleaching, acid hydrolysis, alkali pre-treatment, etc [4,5,6.7,8,9,10].

One of the most prominent modifications is via the alkalization process. The alkalization processes consist of treating the lignocellulose fiber in an alkaline solution [2,10]. Although, the lignin and hemicellulose contents contribute to the strength of the lignocellulose fiber, they are generally referred to as impurities that need to be reduced during the alkalization process. Some of the impurities also found in the lignocellulose fiber which is also removed during the alkalization process include the waxy oil covering the fiber surface and pectin [2,11–13].

Much enumerated studies have been extensively analyzed on the use of several reagents to boost the cellulose components of different natural fiber. These are highlighted: typha fiber (15); wood pulp(8), century fiber (14), rice husk (13), flask fiber(10), waste betel nut husk fibre (2), Mendong straw (16), oil palm fruit bunch(17), Pine wood(18), mango seed shell fiber(3,19), shea butter bark wood fiber(20), etc. However, this work investigates the effects of sodium hydroxide

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(NaOH) concentrations on the elemental compositions (cellulose, hemicellulose, and lignin contents) of mango bark wood (MBWF). FTIR and SEM analysis for untreated and NaOH treated MBWF was also examined.

II. MATERIALS AND METHODS

Material collection preparation

The mango bark wood fiber (MBWF) was collected from Wapan Nghaku in Wukari Local Government Area of Taraba State Nigeria. The MBWF was washed with distilled water and sun dried for 24 hours before crushing and sieved using a 850 µm sieve size. The fine grain of MBWF was stored in a sealed container at room temperature. Another material which was used in the pretreatment process before the analysis is the reagent such as NaOH and water the pretreatment and actual analysis is detailed below.

Pretreatment process of MBWF

The experiment investigated the effect of alkali treatment (NaOH) on the chemical properties of the MBWF. 10g of the crushed and sieved MBWF sample was soaked in the alkali solution with concentration of each alkali varied from 4wt%, 8wt%, and 12wt%. The solid part of the MBWF is washed at pH 7. The time of soaking was also varied from 1.5 hours, 3 hours, and 4.5 hours after which cellulose, hemicellulose and lignin contents were analyzed accordingly.

Analysis of the lignin and cellulose contents

The untreated and treated samples were analyzed for its lignin cellulose and hemicellulose contents at varying concentration and time using the Chesson method(20) as follows

(A) 1g of the dried sample containing 150 ml of distilled water was heated at 100°C for 1hour and weighed

(B) The mixture (dried sample containing 150 ml of distilled water) was dried, filtered and weighed until the weight was constant.

(C) The residue was mixed with 150ml of 0.5 M sulfuric acid and heated at 100 °C for 1hour. The mixture was filtered, dried and weighed until weight was constant

(D) The residue was mixed with 10ml of 72%w/v sulfuric acid at room temperature for 4 hours and then heated for 1 hour and weighed.

(E) The residue was heated until it becomes ash and weighed. The cellulose and lignin contents were calculated as presented in equations 1 and 2, respectively

$$\% \text{ cellulose} = \frac{(C-D)}{A} \times 100\% \quad (1)$$

$$\%\text{lignin} = \frac{(D-E)}{A} \times 100\% \quad (2)$$

For hemicellulose

2g of the residue was placed in a beaker and 10ml sodium hydroxide was added. The residue was stirred with glass rod so that they would mix properly with the sodium hydroxide. The sodium hydroxide was added to the mixture periodically and the mixture temperature was kept at 20°C. About 33ml of distill water was added in the beaker and kept for 1hour. The residue was weighed (A). The residue was transferred to a cubicle and washed with 100ml of sodium hydroxide, 200ml of distill water and 15ml of acetic acid. The cubicle with the residue was dried and weighed (B). The hemicellulose content of MBWF was calculated using equation 3 below

$$\%\text{Hemicellulose} = \frac{\text{weight of residue (A)} - \text{weight of residue in cubicle (B)}}{\text{weight of residue (A)}} \times 100\% \quad (3)$$

SEM analysis

The surface morphologies of the untreated and treated at 12wt% NaOH concentration) were subjected to investigation using the SEM analysis. The analysis was used to study the internal structures of the MBWF. The analysis was performed using a ASPEX 3020 scanning electron microscope.

FTIR analysis

The FTIR spectra analysis of the treated and untreated MBWF at 12wt% NaOH concentration were analyzed using a SHIMADZU scientific infrared spectrophotometer model 8400s by recording the scan in transmission mode (%) in the range of 4000 to 400cm^{-1}.

Results and discussion

SEM analysis for MBWF

The untreated MBWF composition is indicated in Table 1. The cellulose content of UTN MBWF was 38.42 %, this had related value with straw (wheat) (38-45 %) (4,22), corn stalks (38.33-40.31 %) (4,23) and Kenaf (37.50-60.00 %) (4,24–26) . Also, the hemicellulose content of MBWF is 25.75 % which is relatively coincided with corn stalk [23], rice straw (23.22-28.45 %)[5,24] and seed flax (24-26 %)[26]. Furthermore, the MBWF

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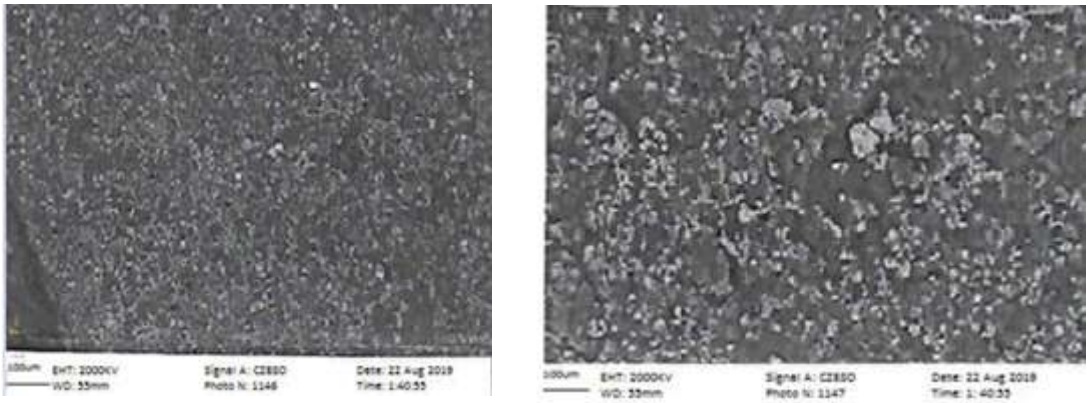
lignin content as was noticed in Table 1 is 26.18 %. The MBWF lignin content sustained the similar content with sugarcane fiber (24.35-26.30 %) [27] and urena lobata bast fiber (22.26-33.21 %)(28). The values obtained from Table 1 in this analysis, the chemical constituent of untreated MBWF displayed analogous characteristics to earlier existing fibers which can been seen as new fiber for utilization of the following domestic and industrial applications: paper making, reinforcement in polymer composite, etc. This can be concluded that MBWF will be applied for similar purpose [4,29].

Table 1: Composition of MBWF (untreated)

Wood Fiber	Cellulose (%)	Hemicellulose (%)	Lignin (%)
MBWF	38.42	25.75	26.18

The SEM micrograph presented in Figure 1(a)-(b) revealed the morphologies of the untreated and alkaline treated MBWF at 12wt% NaOH concentration, respectively. It can be observed that the alkaline treated MBWF revealed rougher surface as compared to the untreated MBWF (1,10). The rougher surface observed in the alkaline treated could be attributed to the removal of the waxy and oily layer on the surface of the fiber.

The SEM micrograph of the alkaline treated MBWF at 12wt% NaOH concentration also showed an increase in the pore spaces on the fiber surface (Figure 1b). The untreated MBWF showed a slippery and oily surface as mentioned in Figure 1(a). This was due to the accumulation of impurities on the fiber surface. However, the observed rough surface of the alkaline treated MBWF fiber indicated its potential benefits as reinforcing material in the composite manufacturing.



(a) (b)

Figure 1: SEM micrograph of crushed MBWF (a) untreated (b) treated at 12wt% NaOH concentration

Figures 2 (a-b) shows the FTIR analysis result for the untreated and treated MBWF, respectively.

` The band around 1644.37cm^{-1} (Figure 2(a) was attributed to the C=C bond in cellulose. This band reappeared in figure 2(b) at 1605.79 cm^{-1} after the alkaline treatment and was attributed to the conjugated C=O stretching in lignin and cellulose [10,14,29]. The band at 1742.74 cm^{-1} in the treated MBWF in Figure 3 was attributed to the C=O stretching vibration in ketone and carbonyl.

The indicative peaks of hemicellulose and pectin in both the treated and untreated MBWF (figures 2(a) and 2(b)) at a wavenumber of 2925.15 cm^{-1} and 2933.83 cm^{-1}. These peaks were attributed to medium intensity of the C=O stretching vibration within the near infrared region of the MBWF. The absorption bands in the region between 3419.90 cm^{-1} and 2350.34 cm^{-1} in the untreated and treated MBWF respectively were attributed to the vibration of O-H bond. However, increasing the alkali concentration was found to decrease this bond [15,30].

The carbonyl group of C=O stretching vibration was observed in the spectrum of the treated MBWF from 1456.30 cm^{-1} to 1372.40 cm^{-1} (figure 2(b). The bands at 1443.77 cm^{-1} for the untreated MBWF (figure 2(a)) and those that appeared at 1033.88 cm^{-1} and 465.82 cm^{-1} in the treated MBWF (figure 2(b)) were characteristics of –O-CH$_3$ and C=C band in lignin. Finally,

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transformational changes in functional group of the MBWF were observed from Figure 2(a) to 2(b) which is an indication for partly extraction of impurities from the organic fiber after soaking of NaOH solution.

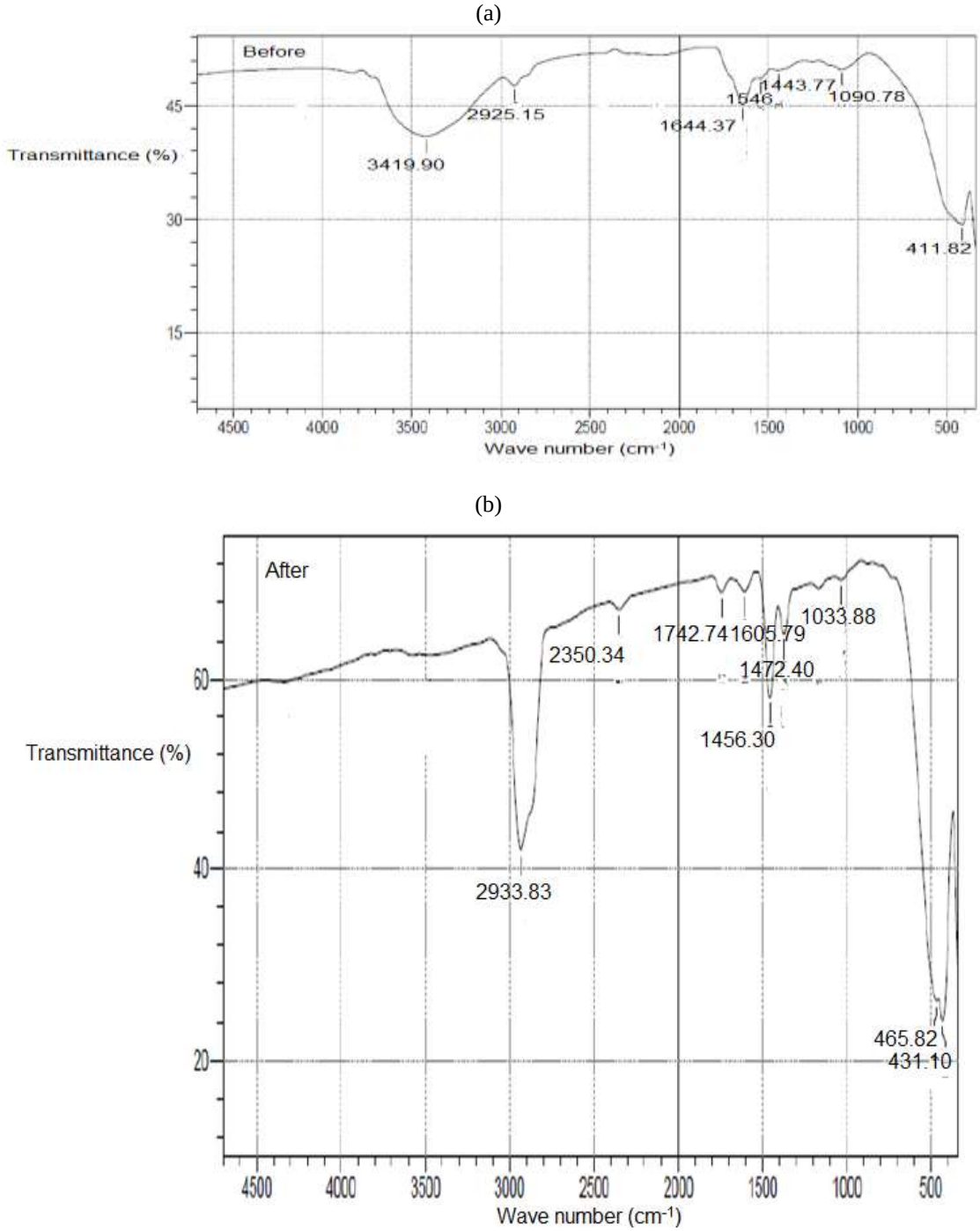

Figure 2: FTIR spectroscopy of the (a) untreated (b) treated MBWF at 12 wt% NaOH concentration

To investigate the effects of time and NaOH concentration on the hemicellulose content of MBWF, the soaking time were varied from 1.5 hrs, 3 hrs, and 4.5 hrs. The NaOH concentrations were also varied from 0 wt%, 4 wt%, 8 wt%, and 12 wt%, at 25°C. The 0 wt% NaOH concentration represents the untreated MBWF, which was used for comparisons with the treated samples. The

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untreated and treated hemicellulose content of MBWF at different time and NaOH concentration is presented in Figure 3.

In comparison with the hemicellulose content of the untreated MBWF (25.75 %), it was observed that the reductions in the hemicellulose contents of the MBWF after treatment with NaOH at 1.5 hrs were 18.2 %, 15.1 %, and 10.09 % at 4 wt%, 8 wt%, and 12 wt%, respectively. At 3 hrs, the reduction in the hemicellulose content after treatment with 4wt%, 8wt%, and 12wt% NaOH concentration were 18.0 %, 16.8 %, and 13.1 %, respectively. Similarly, the hemicellulose reductions at 4.5 hrs were closely related to the ones observed at 3 hrs (Figure 3). However, the deductions made from the hemicellulose content after the NaOH treatment showed that the optimum time of 1.5 hr and 12 wt% NaOH concentration gave the best reduction (10.9%) in the hemicellulose content of MBWF. Furthermore, the observation demonstrated that the hemicellulose content reduction is favored by decreasing the treatment time and increasing alkali concentration. Similarly, in previous study, Ikramullah et al.,[15] reported 1 hour treatment time of Tyha fiber in NaOH solution was the optimum as it does not harm the components of the fiber. Earlier scholars reported close results[1,4,7–9,12,15,31].

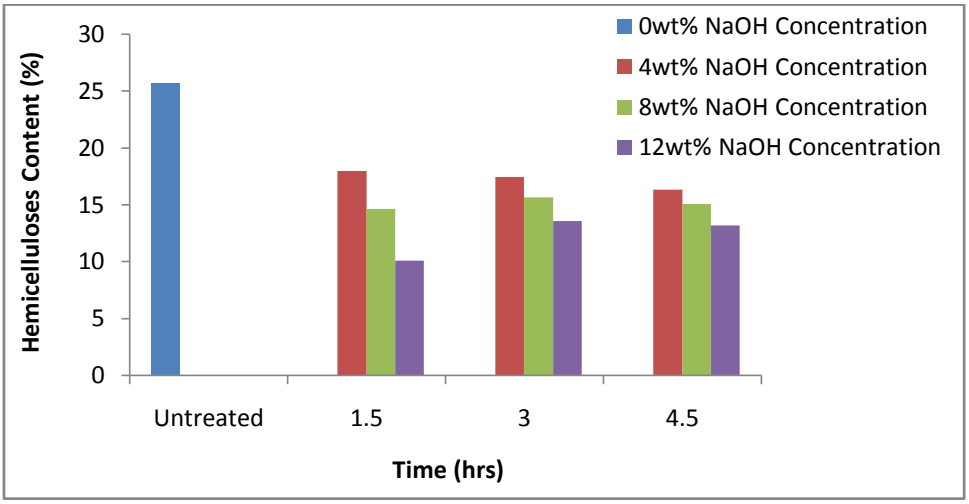

Figure 3. Effect of soaking time at different NaOH concentration on hemicellulose content of MBWF

Effect of time and NaOH concentration on the cellulose content of MBWF

From figure 4, it can be found that the alkali (NaOH) treatment of the MBWF enhanced the cellulose content. Initially, the cellulose content of the untreated MBWF was 39 % (Figure 4). However, after treatment in NaOH concentrations of 4 wt%, 8 wt%, and 12 wt% at 1.5 hours, the enhancement in cellulose contents of the MBWF were 50 %, 55 % and 60 %, respectively (Figure 4).

The results obtained for the cellulose content of MBWF after 3 and 4.5 hours and at 4 wt%, 8 wt%, and 12 wt% NaOH concentrations also showed an enhancement in the cellulose content (figure 5). Comparing the time of treatment (1.5, 3 and 4.5 hours) and NaOH concentrations (4wt%, 8 wt%, and 12 wt %), it was observed that the optimum enhancement of the cellulose content of the MBWF was obtained at 12 wt% NaOH concentration and 1.5hours (Figure 4). At these optimum conditions, the cellulose content enhancement was 60%.

The enhancement observed in the cellulose content of the MBWF as a result of alkali treatment translates to an improvement in the tensile strength and elongation of the MBWF[14]. The enhancement further indicates that the fibril content of the hemicellulose was removed making the MBWF suitable for composite production [14]. The reductions in the lignin and hemicellulose contents of MBWF in Figures 5 and 4 suggest the elimination of the non-productive active sites in the MBWF, which will contribute to the improvements of the cellulose content [30]. Related outcomes have been stated previously.[1,4,7–9,12,15,31]

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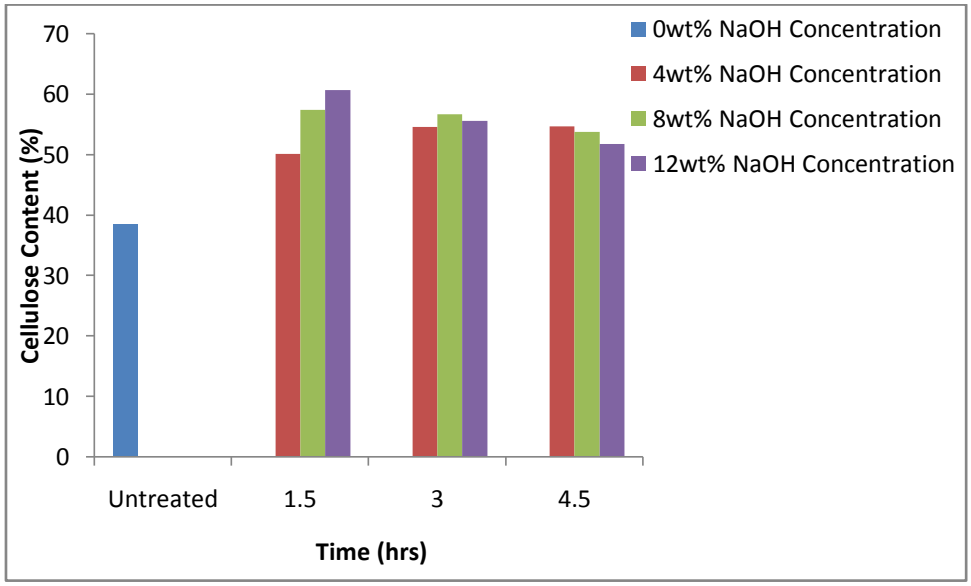

Figure 4: Effect of soaking time at different NaOH concentration on cellulose content of MBWF

Effects of time and NaOH concentration on the lignin content of MBWF were investigated by varying the treatment from 1.5 hrs, 3 hrs, and 4.5 hrs. The NaOH concentrations were also varied from 0 wt%, 4 wt%, 8 wt%, and 12 wt%, at 25 °C. As presented in Figure 5, the reduction in the lignin content of MBWF at different time and NaOH concentrations were compared with the lignin content of the untreated MBWF.

As observed, the reduction in lignin content of MBWF was evident at different NaOH treatment and time. The results showed that at 1.5 hours and 12 wt% NaOH concentration, the reduction in lignin content of MBWF fiber was 15 % from the initial 26 % of the untreated MBWF (Figure 5). Also, at 1.5 hours NaOH treatment, the lignin reduction was 20 % and 17 % at 4 wt% and 8 wt% NaOH concentration, respectively.

However, after 3 and 4.5 hours, the reduction in the lignin content of MBWF at 4 wt% and 8 wt% and 12 wt% were not significant (Figure 5). In comparisons with the untreated MBWF (26%) in Fsigure 5, it was observed that a significant reduction in the lignin content of MBWF was obtained at 1.5 hours and 12 wt% NaOH concentration. The reduction in the lignin content of MBWF could be attributed to the breakage of the internal structure of the lignin[30]. The outcomes from this study were closely obtained by earlier researchers [1,4,7–9,12,15,31].

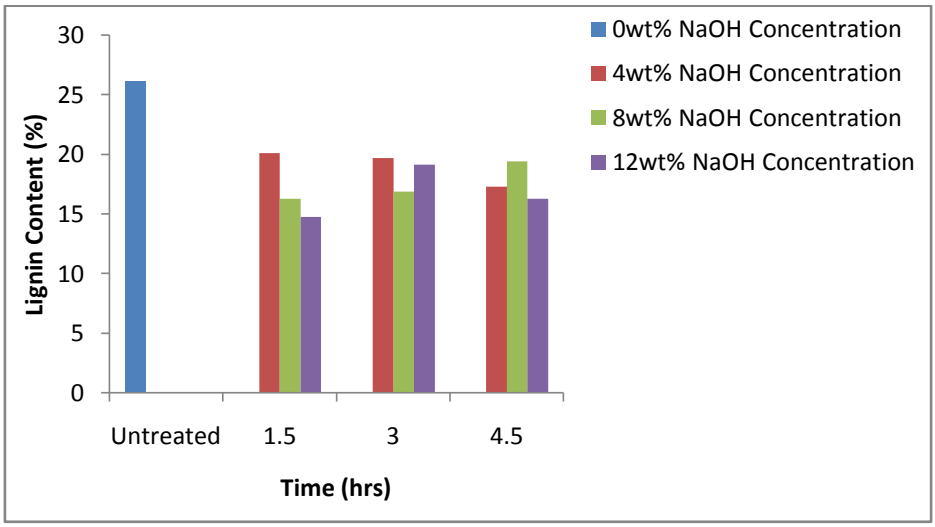

Figure 5: Effect of soaking time at different NaOH concentration on lignin content of MBWF

III. CONCLUSION

The alkali (NaOH) treatment of the mango bark wood fiber has been achieved and reported in this work. In variably, time of immersion and NaOH concentration is a determinant in the MWBF composition. It was found that the alkali treatment permits the reduction in the lignin and hemicellulose content of the MBWF. The MBWF treated with 12wt% NaOH concentration was found to give the optimum tensile strength and dimensional stability of the fiber as it resulted in an increase in the cellulose content. This study shows that increasing the NaOH concentration significantly decreased the lignin and hemicellulose content while improving the cellulose content. The result from this research shows that the MBWF at optimal state of pre-treatment is a credible substitute for bio-composite application.

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