

## RESEARCH ARTICLE



### OPEN ACCESS

Received: 05-04-2020

Accepted: 19-05-2020

Published: 23-06-2020

Editor: Dr. Natarajan Gajendran

**Citation:** Government RM, Okeke ET, Odera RS, Ani AK, Thaddaeus J, Ikechukwu NB (2020) Significance of alkaline treatment on the composition of mango seed shell fiber for polymer composite application. Indian Journal of Science and Technology 13(21): 2168-2174. <https://doi.org/10.17485/IJST/v13i21.81>

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**Funding:** None

**Competing Interests:** None

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## Significance of alkaline treatment on the composition of mango seed shell fiber for polymer composite application

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## Abstract

**Background/Objectives:** The minimization of unnecessary components from agro-waste is essential for performance and conversion into useful products in domestic and industrial utilization. The need for removal of these unwanted components is important to boost the strength of the agro-waste for its effectiveness as additive in agro-based polymer composite production as new engineering material for structural purpose. This investigation entailed the influence of NaOH concentration and soaking time on the key compositional content of novel mango seed shell fiber (MSSF). **Methods/findings:** The MSSF was treated with NaOH solution at 2.5, 5, 7.5 wt % concentration and soaking time of 2-6 hr. The compositions of MSSF were obtained for the treated and untreated sample by gravimetric method. The pure and NaOH treated MSSF were analyzed instrumentally employing Fourier transform infrared (FTIR) spectrometer to show the functionality of some substances present. From this result, the optimum composition of MSSF was observed at 5 % NaOH concentration and 4 hr soaking time. The maximum composition at this condition improved than the crude MSSF by 71.33% cellulose content, while the hemicelluloses and lignin content removal was lower than the raw fiber by 91.75 and 98.84%, respectively. **Application:** The results at this optimum treatment of MSSF composition can be recommended for agro-based polymer composite in indoor application. **Keywords:** Mango seed shell fiber; composition; NaOH; agro-wastes; alkaline treatment

## 1 Introduction

The use of agro-wastes in composite application is tremendously increasing. This is due to the advantages of lightweight, low cost and abundant availability<sup>(1,2)</sup>. The immerse deposits of agro-waste in the environs are alarming<sup>(3)</sup>. This is as result of human activities in the mechanized agro-industries<sup>(3)</sup>. Generally, an estimation of the product for

agro-wastes annually is about 140 billion metric tons which is a surplus raw material for polymer and other related industries.<sup>(4)</sup>

Currently, less attention is giving to agro-wastes for the conversion into different products. Long term plans should be adopted for the utilization of the wastes for manufacturing of various finished products due to the present state of the conversion of these raw materials is below standard<sup>(4,5)</sup>. The goal of demanding the usage of lignocellulosic source from agro-wastes in this present industrial evolutionary economy is to alleviate waste generates from plants in the environment<sup>(4,6)</sup>. Due to the huge availability of agro-wastes after the extraction of the edible components, making these fibers a major raw material during the period of harvest irrespective the exclusion of more land for cultivation purposes.<sup>(4,7)</sup>

The agro-wastes are examples of natural fibers. The fibers derive from these wastes are well-known as cellulose fibers or lignocellulose materials. The major sources of the wastes are grass, seeds, stem, fruit and stalk.<sup>(8)</sup> These wastes have essential specific characteristic<sup>(8–11)</sup>. As a result of large deposits of agro-wastes, agro-based industry applies non-traditional feedstocks which include baggase, coconut shell, oil palm waste and pineapple leaves. This is due to its viability and accessibility after experimentation, and these mentioned agro-wastes have confirmed to be used for composites, engineering components, and paper making<sup>(12)</sup>.

The agro-wastes are composite of cellulose, hemicelluloses, lignin and other minor components<sup>(13)</sup>. The cellulosic material depends on species of plants, the time of germination and harvest<sup>(9,10)</sup>. Nevertheless, agro-wastes have to be chemically modified to extract the most important constituent otherwise regards as cellulose, the minimization of the hemicelluloses, lignin and nearly eradicates some minute components. This is to determine its suitable for different uses<sup>(3)</sup>. Therefore, agro-wastes with high cellulose content will yield superior ultimate tensile strength and elastic modulus.<sup>(11,12)</sup>

The cellulosic fibers produced from agro-waste can be treated in many ways in order to beef up the main component, reduces the two major impurities and other skeletal compositional constituents<sup>(1–3), (13–18)</sup>.

The processes for treating fibers include bleaching, alkalization, acidification and numerous process treatments. The treatment by breaching involves the use of chlorite<sup>(18)</sup>, the acidification process includes the immersion of plant wastes generates from the agro-wastes in HCl or H<sub>2</sub>SO<sub>4</sub> solution<sup>(19,20)</sup>, while alkalization treatment entails modification of the fibers in NaOH, KOH or Ca(OH)<sub>2</sub> solution<sup>(13,21)</sup>. The numerous stage treatment is basically engages O<sub>2</sub> or H<sub>2</sub>O<sub>2</sub> by eliminating unwanted constituents devoid of affecting the cellulosic agro-fibers<sup>(22)</sup>. Out of these methods, alkalization is the common and the efficient treatment process for degrading hemicelluloses, lignin and other composition of agro-wastes.<sup>(23,24)</sup>

During chemical treatment of agro-wastes, the interlinking between three main constituents of the fibers is broking. This treatment allows the solublization of unwanted components, the elimination of non-cellulosic materials in the natural fiber and leaving fiber with high degree of cellulose<sup>(13)</sup>. Also, treating the agro-waste reduces crystallinity, enhances pores and fiber surface<sup>(4,24–34)</sup>.

However, the chemical composition of agro-based fiber is based on the amount of reagent concentration solution to fiber ratio, immersion time and temperature of treatment<sup>(14–16)</sup>.

Previous studies have done extensive researches on the use of chemical modifiers to boast the cellulose content of agro-wastes for reinforcing agent in polymer composite for structural material in household use. Fahma et al.<sup>(14)</sup>, Johar et al.<sup>(17)</sup> and Jahan et al.<sup>(18)</sup> used H<sub>2</sub>SO<sub>4</sub> hydrolysis for oil palm, rice husk and jute fiber, respectively. Leitner et al.<sup>(19)</sup> induced wet chemistry for by-product of sugar beet chip. Sun et al.<sup>(31)</sup>, Barlianti et al.<sup>(20)</sup>, Shaihaidu and Soh<sup>(4)</sup>, and Iroba et al.<sup>(33)</sup> applied NaOH hydrolysis for wheat straw, rice straw and barley, respectively.

This work aimed to evaluate the composition of untreated novel agro-waste, MSSF, the variation of NaOH concentration and soaking time on its composition, determination of optimum process parameters that will yield the maximum cellulose, reduction of hemicelluloses and lignin content for its accessibility as alternative reinforcing fiber in polymer composite production for furniture in domestic electronic support.

## 2 Experimental section

### 2.1 Supply of mango seed shell fiber (MSSF)

The mango seed shell fiber (MSSF) was extracted at Wapam-Ngaku of Taraba State in Wukari Local Government Area.

### 2.2 Procurement OF NaOH

The 98% pure NaOH was supplied by LOBA Chemie Laboratory reagents and fine chemicals Pvt. Limited in Mumbai of the Republic of India. The NaOH was purchased in Ogbete Main Market, Ogbui at Eastern part of Nigeria, Enugu State

## 2.3 Preparation of MSSF

The MSSF was thoroughly washed with running water and dried in the sun for period of 8 hours in 7 seven days. The MSSF was pulverized to powder form and subjected to 20 mesh size (850  $\mu\text{m}$ ) sieving process. The MSSF of 10g was soaked in NaOH solution at 2.5, 5.0, 7.5 wt % concentration for 2, 4, 6 hr time. The mixture of MSSF and alkaline solution was later, filtered and rinsed with distilled water for five times to remove unwanted component of the fiber. The MSSF was further sun-dried for 8 hours for determination of the composition for the treated and untreated fiber by gravimetric method.

## 2.4 Determination of chemical composition of MSSF

The compositional characterization of the treated and untreated MSSF was done at Divine Analytical laboratory, Nsukka, Enugu State of Nigeria. The composition of chemical modified and pure MSSF was done by Chesson-Datta gravimetric method<sup>(20)</sup>. A mixture 1 g of alkalized and non-alkalized dried MSSF (V) and 150 mL de-mineralized water were boiled in a beaker at a 100°C for 1 hour, respectively. The residue was obtained after separation by filtration of the mixture of MSSF and the deionized water after the heating. The residue was later rinsed with 300 mL of warm de-mineralized water. This residue was dried in an oven to a constant weight (W). The residue and 150 mL of 1 M H<sub>2</sub>SO<sub>4</sub> mixture was heated in the oil bath for 1 h at 100°C. The mixture was later filtered, rinsed with 300 mL of de-mineralized water and dried to a new constant residue (X). This residue was soaked with 10 mL of 72 % H<sub>2</sub>SO<sub>4</sub> at normal atmospheric condition for 4 h and 150 mL of 1 M H<sub>2</sub>SO<sub>4</sub> was further added to the mixture. The mixture was refluxed in the oil bath for 1h. The solid product during reflux was rinsed with 400 mL of de-mineralized water, oven-heated at 105 °C and weighed for stable weight (Y). The solid was heated again until ash surfaced and finally weighed (Z). The determination for the composition of MSSF was repeated both the treated and untreated flour. The stated relationship, Eq. (1), Eq. (2) and Eq.(3) were applied to evaluate the cellulose, hemicellulose and lignin content, respectively:

$$\% \text{ cellulose} = \frac{(Y - X)}{V} \times 100 \quad (1)$$

$$\% \text{ hemicellulose} = \frac{(X - W)}{V} \times 100 \quad (2)$$

$$\% \text{ lignin} = \frac{(Z - Y)}{V} \times 100 \quad (3)$$

## 2.5 FTIR examination of MSSF

The analysis of MSSF for both treated and untreated was examined with FTIR system spectrum (MODEL NO. BX).

## 3 Results and Discussion

**Table 1** presents the composition of untreated MSSF. From this Table, the untreated MSSF displayed 56.74 % cellulose content, 32.84 % hemicelluloses and 6.87 % lignin content. The result for cellulose content of MSSF was comparable to kenaf (37.50-63.00 %) <sup>(35-40)</sup> and abaca (56-63 %) <sup>(21)</sup>. Also, percentage of hemicelluloses for the crude MSSF indicated similar feature with hard woods (25-35 %) <sup>(22)</sup>. The MSSF content with respect to lignin was related to pineapple fiber (5.35-12.33 %) <sup>(23)</sup> Since the composition of crude MSSF was close to some existing agro-waste based fibers, this means that crude MSSF is eligible for reinforced polymer composite, paper and engineering material <sup>(3)</sup>.

**Table 1.** Chemical composition of untreated MSSF

Agro-waste	Cellulose (%)	Hemicellulose (%)	Lignin (%)
MSSF	56.74	32.84	6.87

**Figure 1** (a-b) presents the FTIR diagram of untreated and treated MSSF, respectively. The presence of OH group was situated between 3845.71 to 3411.42  $\text{cm}^{-1}$  and 3720 to 3497.14  $\text{cm}^{-1}$  for **Figure 1** (a) and 1(b) before and after soaking in sodium hydroxide solution at 5 wt% and 4 hrs time, respectively. Also, the presence of COOH bonding emerged at the untreated and sodium hydroxide absorbed MSSF with corresponding crests at 2937.14  $\text{cm}^{-1}$  and 2925.71  $\text{cm}^{-1}$  to 2857.14  $\text{cm}^{-1}$  in **Figure 1** (a) and **Figure 1** (b), respectively. As pointed out in the pure MSSF as shown in **Figure 1** (a), the presence of lignin and hemicelluloses can be traced at C=O and C-O bonds in the fresh MSSF at peaks of 1736.37  $\text{cm}^{-1}$  and 1281.86  $\text{cm}^{-1}$ , respectively. There are little observable traces of the lignin and hemicellulose bonds in the **Figure 1** (b) after alkalization with NaOH solution. This can be

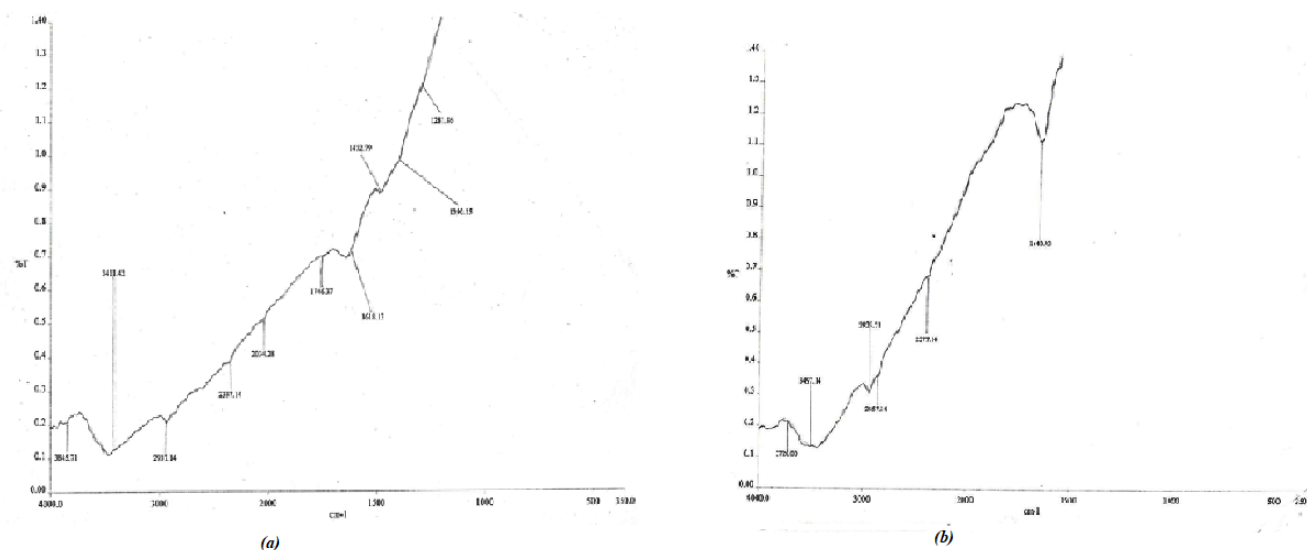


Fig 1. FTIR spectra of (a) pure MSSF and (b) treated MSSF at 5 wt% NaOH and 4 hr soaking.

concluded that these weak components that held the bonds of MSSF has been drastically minimized and the cellulosic content in the composition of MSSF has improved after modification with NaOH as justified by works in elsewhere<sup>(41–44)</sup>.

Figure 2 captures the effect of alkali treatment on cellulose content of MSSF. The cellulose content improved when the NaOH solution concentration increases from 0 % to 7.5 %. The percentage of the cellulose steadily increases by 57.35 % when compared to the crude MSSF from 0 to 7.5 % NaOH concentration of alkali modification at 2 hr. The enhancement of cellulose was displayed due to chemical modification by NaOH reduces other components in the cellulosic agro-waste by breaking the bond of the fiber, allowing other components to move out from the alkali solution<sup>(12)</sup>. Similar improvement was recorded at soaking time of 4 hr and 6 hr by 63.46 % and 48.03 % than the crude MSSF, respectively. In addition, high cellulose production of the MSSF was felt when the residence time increases due to more immersion of NaOH solution which aids removal of unwanted material from the fiber. The enhancement of cellulose was displayed due to chemical modification by NaOH reduces other components in the cellulosic agro-waste by breaking the bond of the fiber, allowing other components to move out from the alkali solution. The optimum cellulose yield was discovered at 5 % concentration of NaOH solution for 4 hr soaking time. At this condition, the cellulose content of treated MSSF is higher than the crude by 71.33 %. The improvement in the cellulosic components of MSSF at this point is due to the partly removal of unwanted material without fiber destruction<sup>(24)</sup>. Early researchers had felt this trend.<sup>(4,25–27)</sup>

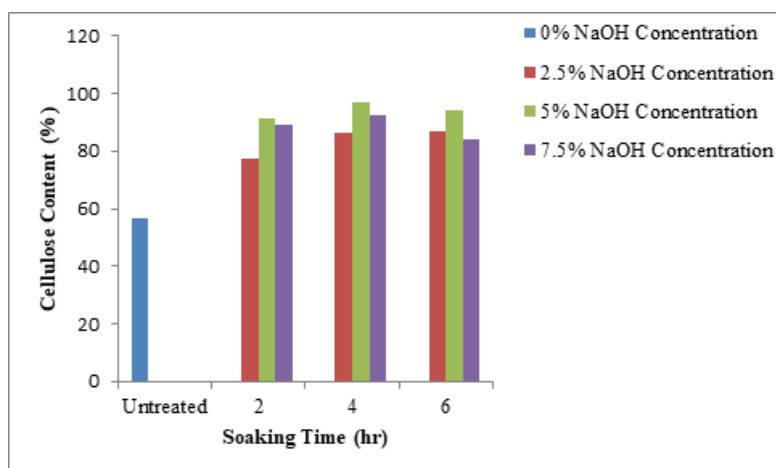


Fig 2. Effect of NaOH treatment on cellulose content of MSSF

Figure 3 indicates the variance of MSSF hemicellulose content with alkali treatment parameters. The hemicellulose component of MSSF declines with increase in NaOH concentration from 0 to 7.5 %. The hemicellulose of crude MSSF reduces by 75.15 % after the ranges of NaOH treatment at 2 hr. This observed trend is due to hemicelluloses which is the second largest constituent breaks down from the MSSF bond after immersion of alkali solution, hence, leading to the reduction of this component from the fiber<sup>(12)</sup>. For 4 hr and 6 hr of soaking time, it reduces by 86.97 and 83.62 % when the concentration increases from 0 to 7.5 %, respectively. Furthermore, there are also significant hemicelluloses drop when the resident time increases. This attributed fact justifies that long exposure of the MSSF in the alkali modification process minimizes more of the hemicelluloses from the fiber. The maximum hemicelluloses of crude MSSF removal occur at 5 % concentration and 4 hr soaking time by 91.75 %. Famous scholars have exhibited these trends.<sup>(4,25–27)</sup>

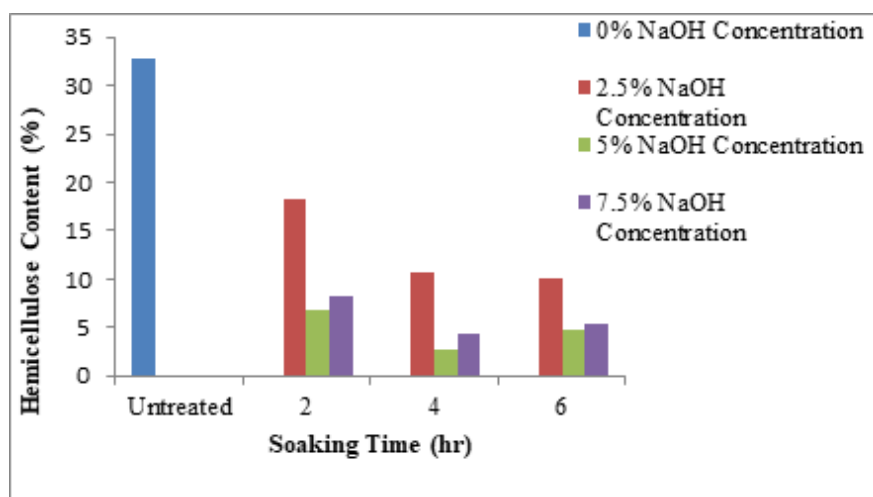


Fig 3. Variation of NaOH treatment on hemicelluloses content of MSSF

Figure 4 illustrates the lignin content removal on the effect of alkali modification of MSSF. From this picture, it was noticed that the MSSF lignin content minimizes during the immersion of MSSF into NaOH solution concentration from 0 to 7.5 %. The crude MSSF observed a fall in lignin content aftermath of treatment from 0 to 7.5 % by 62.74 % at 2 hr of soaking time. This is due to disruptions of MSSF bond which beef up the cellulose and further perk up the pore spaces of the fiber<sup>(12)</sup>. The rise of these spaces, drops the crystallinity and later take away the lignin from the MSSF.<sup>(4,28,29)</sup> In addition, the lignin in MSSF also experiences additional improvement in its crude removal for further soaking time at 4 hr and 6 hr NaOH treatment by 71.32 and 67.39 %, respectively. These observable trends could be as a result of more saturated NaOH solution as the resident time increases which releases more lignin from the MSSF. The utmost lignin reduction for MSSF crude was observed at 5 % NaOH solution concentration and 4 hr soaking time by 98.84 %. At this point, all other impurities had been removed from the MSSF. These outcomes toe similar sequence with past investigators.<sup>(4,32,33,45–48)</sup>

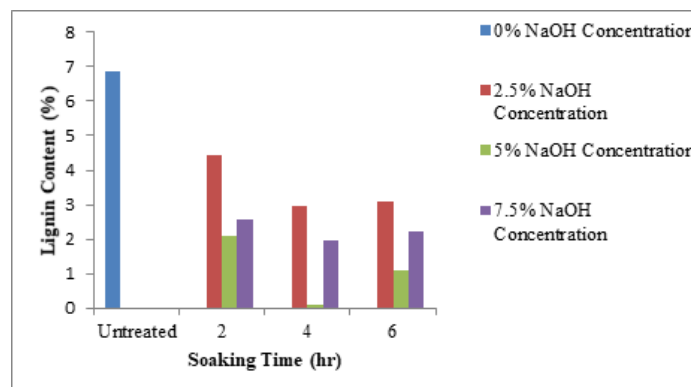


Fig 4. Influence of NaOH treatment on Lignin content of MSSF.



## 4 Conclusion

The study on alkali treatment on improving cellulose yield, hemicellulose and lignin removal from novel agro-based-waste, MSSF by NaOH had been performed for discovery of new reinforcing fiber in polymer composite for domestic structural application. The composition of crude MSSF has been established. The operation parameters: NaOH solution concentration and soaking time influenced the composition of MSSF. The outcome in this research showed that the percentage yield of the cellulose surpassed the crude, while the effective removal of hemicelluloses and lignin content lowered than the raw MSSF after treatment, respectively. The results obtained from this work indicated that the raw and treated MSSF can be recommended in agro-based polymer composite production for indoor application.

## Acknowledgement

The authors acknowledged Prof. Onukwuli, O.D of Department of Chemical Engineering, Nnamdi Azikiwe University, Awka for nurturing us with this area of research and Prof. Egwakhide, P.A of Chemical Sciences of Federal University Wukari, Taraba for the opportunity for this study.

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