

## Effect of Alkaline Pre-Treatment on the Surface Modification of Napier Grass Fibres for the Properties of Medium Density Fibreboard (MDF)

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### ABSTRACT

*In this study, Napier grass (Pennisetum purpureum) fibres which hold 59.19% cellulose, 21.35% hemicellulose and 7.26% lignin were comprehensively characterized to evaluate their potential as building material for medium density fibreboard (MDF). The purpose of this work was to examine the effects of sodium hydroxide solution as an alkali pre-treatment in determining the best condition for lignin removal in Napier fibres. The treatments were conducted using NaOH concentrations of 0.5, 3.0, 5.5, 8.0, 10.0 and 10.5 wt. %. The morphology of the fibres was observed using scanning electron microscope (SEM) and its chemical compositional changes was confirmed by Fourier transform infrared spectroscopy (FTIR) analysis. The study showed that the morphological observation demonstrated that the best concentration for the removal of lignin in Napier fibre was 10.5 wt. % NaOH solutions. It has been found that as the alkali concentration increased, the surface of the fibre becomes rougher which reduced the void content in the fibres. The fibres that were alkali-treated using NaOH solutions of various concentrations as well as the untreated one were subjected to single fibre testing. From the results of the single fibre testing, it is revealed that the 10.5% alkali-treated Napier grass fibres yielded the maximum and highest strength. This further signifies that alkali pre-treatment has significant impact on the production of MDF board from the non-wood fibres biomass such as Napier fibre.*

### Keywords

Napier grass, NaOH, Medium Density Fibreboard.

### Introduction

The recent growth in eco-friendly consciousness has created great interest in the use of natural fibres as alternative non-wood fibres materials for medium density fibreboard (MDF). This is basically because of their low environmental effect, low cost, and relatively noble specific properties. Researchers have been determined to develop biodegradable building material using renewable agro-based resources. MDF is one of the greatest commonly used wood-based boards to manufacture structure and housing components such as furniture parts for interior uses. In current years, production of MDF has significantly greater than before and has a main market share in the wood industry [1]. MDF has numerous advantages such as evener surface, easier machinability, and is a supreme board material as substrate for thin overlays used in indoor conditions [2]. MDF can be manufactured from variety of natural fibres but lumber or wood, because of its comparative great quantity and

constant availability, is still the most needed raw material. On the other hand, growing response of forest resources for diverse uses has run to the deficiency of wood supply. Consequently, there is a requirement to find substitute raw materials or wide-ranging use of wood capitals containing harvesting residues, annual plants, lumber and furniture plant residues, residues of pulp plants, and recycled paper [3]. Great response for MDF in the world marketplace has led MDF manufacturers to raise their production volume. This has caused in an increased option for raw materials to achieve the market demand.

In Malaysia, MDF manufactures use rubber wood as their main raw material, which, though is shrinking. Thus, manufacturers need to look for alternative assets that are in continuous supply and moreover, are economical [4]. Extensive reviews showed by facts of publications have well-defined the transformations of natural fibres with respects to their mechanical properties and its applications. Numerous authors documented the application of natural fibres such as corn [3], empty fruit bunch of oil palm,

coconut coir [5] and bagasse fibres [6], as reinforcement fibres in MDF fabrications. Malaysia has plenty of agro waste material that has not been fully exploited to an all-out production. Fibrous material from agricultural residues is believed to have great potential for replacing wood in MDF composite materials. As this issue turn out to be an interesting one, another fibre from non-wood material would give a good key on save our environment from being extinguish together developing of human life. One of the most potential and possible plant residue materials for manufacturing MDF is Napier grass stems, which can play a major role in providing the balance between supply and demand. Thus, this study aims to investigate the development of MDF by using agricultural residues which is Napier grass to be as the fibres bond with urea-formaldehyde as a binder.

Napier grass, also identified as Elephant or Uganda grass, is one of the most important tropical forage crops [7]. Napier grass was first presented in Malaysia in the 1920's from East Africa and is now become the most popular fodder grass in dairy production in Malaysia Kong, 1988. Ecological livestock production is highly rely on the availability of quality feed and fodder resources. Chee & Peng reported, about 9 750 tonnes of Napier grass silage was produced as an option feedstuff supply for ruminants to overcome forage shortages in Malaysia. In addition, with the faster development of the ruminant industry, the request for Napier hay and chaff has enlarged significantly. At present, our country imported Napier hay at a high price of RM800 to RM1400/tonne, depending on type, quality and country of source. Chaff is imported at RM1500/tonne. The study by Kong, which concluded that in Sabah, there are two farming sectors, namely the smallholders and the large commercial holdings. The smallholders usually have farm sizes of about 1.5 ha in the northern paddy areas while along the coastal region and around milk collecting centres in inland areas; farm size varies from 0.3 ha to 1–2 ha. Generally about 59% of the farmers have no land for livestock activities, 17% own less than 1 ha and 16% run their operations on 1–2 ha of farm land. Therefore, due to the fact that most smallholder livestock own small and fragmented pieces of land, grasses such as Napier grass offer a best-fit alternative to other feed options, as these are high yielding forages which require a minimum amount of inputs and land. This is reflected that there is a prospect for locally produced Napier hay or chaff to be commercialized into this market and at the same time the stems of the Napier could be processed to become fibres for MDF fabrication in local and broad furniture industry.

However, there are uncertainties concerning the characteristics of natural fibres such as their hydrophilic nature, high moisture absorption, low reactivity, and poor compatibility with polymeric matrices, all of which impact their mechanical properties [8]. These doubts can be resolved through morphology modification such as alkali treatment to improve the MDF interfacial adhesion between natural fibres (Napier fibres) and its adhesives (urea-formaldehyde) as well as to enhancing its mechanical and physical [9].

In this study, Napier grass, also scientifically known as *Pennisetum purpureum* fibre, is composed of 59.19% cellulose, 21.35%

hemicellulose and 7.26% lignin. The purpose of the alkali treatment is to eliminate or at least to minimize the lignin content, torn apart the fibres in the fibrils, and form a closely packed cellulose chain to generate the release of the internal strain, which subsequently may improves the mechanical properties of the Napier fibre [5]. Following the alkali treatment, the effective surface area of the Napier fibre available for wetting by the urea-formaldehyde adhesives might be enlarged as the increased of the fibres fibrillation which subsequently believe, enhances the bonding between the fibre-binder interfaces within the MDF board structure [6]. Hence, the process of pre-treatment in Napier fibre, before other treatment methods, is a vital stage in order to obtain more cellulose and hemicellulose accessibility for fibre-binder reaction in MDF fabrication.

The key objective of this study was to explore and discover the prospect and possibility of using Napier grass fibres as a green MDF fibre reinforcement. To the greatest of our awareness, this is the first time a general description of this fibre has been piloted and this investigation should offer novel information to the research community.

## Materials and Methods

### Fibre modification

**Materials:** Napier grass stem with the height of 3 to 5 meters was harvested from a local farm at Stesen Pembiakan Ternakan Sebrang, Keningau, Sabah, Malaysia located on the northern part of Borneo Island. Its altitude is 1,250 meters above sea level and the farming areas are about 800 hectares. The Napier fibres were physically extracted from its grass internodes after exposed to water retting procedure. Firstly, the stalks or stems were washed and crushed using a hammer to detach the fibres strands. Afterward, the short grass stems were immersed in a water storage tank containing with tap water for about 3–4 weeks to enable the bast fibres to detach from the woody centre. Finally the fibre strands were thoroughly cleaned using distilled water immediately to remove any impurities and were sun-dried 3–5 days to ensure removal of moisture content of the fibres. Subsequently, the extracted fibres were dried again in an oven at 103°C for 5 hours to achieve a moisture content of less than 10 wt.%. To keep the material for delignification, it was vacuum stored in a sealed plastic bag in desiccator as shown in Figure 1.

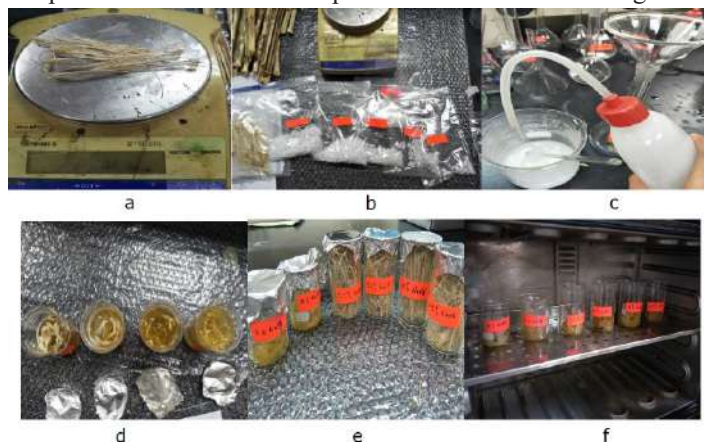


**Figure 1:** (a) harvested Napier grass stem (b) washed Napier stem (c)

crushing for fibre extraction, (d) Soaked Napier grass fibres in tank, (e) cleaned fibre strand with distilled water, (f) sun-dried Napier fibre strand, (g) oven-dried Napier fibre, (h) vacuum stored fibre.

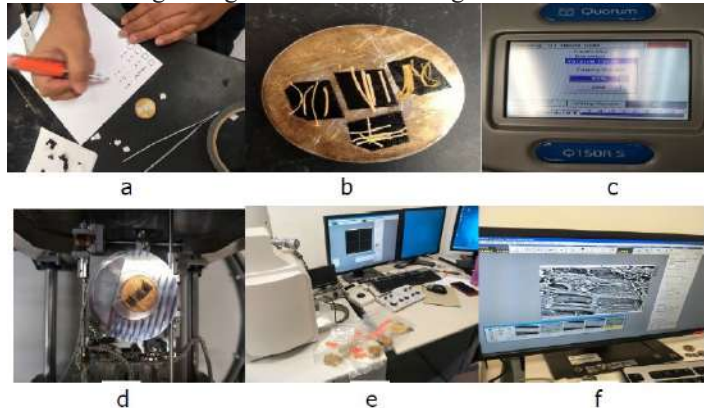
### Alkali pre-treatment with sodium hydroxide

Chemical constituents of Napier fibres are given in Table 1. A sodium hydroxide (NaOH) solution was used to chemically modify the surface of the Napier grass fibres. The treatments were conducted using NaOH concentrations of 0.5, 3.0, 5.5, 8.0, 10.0 and 10.5% (w/w) to perform delignification in Napier fibres. Weighed samples of 20.0 g dry fibres were immersed in the NaOH solutions respectively and incubated at 60°C for 4 hours. The samples were finally washed several times with distilled water until neutral pH was achieved to remove absorbed alkali from the fibres. The treated Napier fibres were then dried at room temperature for overnight followed by oven drying at 103–105°C for 10 hours. The material was characterized to determine its composition. All contents were expressed on a dry basis throughout this work. A sample of material after alkali pre-treatment is shown in Figure 2.



**Figure 2:** (a) 20.0 g of Napier fibres were weighed, (b) Sodium hydroxide pellets were weighed according to each concentration, (c) preparation of sodium hydroxide solution, (d) 20.0 g of material was immersed in the NaOH solution, (e) samples were placed in room temperature for 10 minutes, (f) samples were incubated at 60°C for 4 hours.

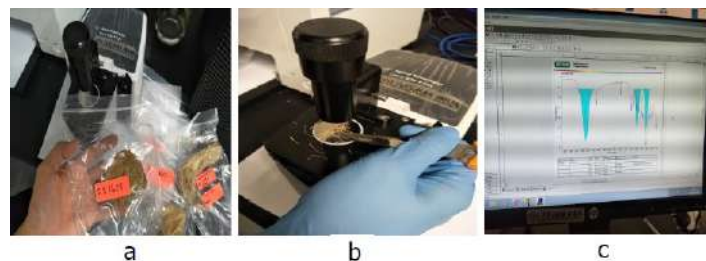
The surface morphologies of the untreated and treated Napier fibre strands were observed using an SEM (HITACHI Model S-3400N) as shown in Figure 3. Prior to SEM analysis the samples were coated to obtain an optimum image. The scanning images were expanded with an accelerating voltage of 15.0 kV and magnification of 100–700×.



**Figure 3:** (a)-(f). Sample preparations and coating for SEM analysis.

### Compositional changes of Napier fibre characterisation by Fourier Transform Infrared Spectroscopy (FTIR)

The Agilent Technologies Cary 630 FTIR was used to originate the FTIR spectra of the untreated and treated Napier fibre as shown in Figure 4. All the spectra were recorded in the wavenumber range of 600–3500  $\text{cm}^{-1}$ , working in ATR (attenuated total reflectance) mode.



**Figure 4:** (a)-(c). Sample preparations for FTIR analysis.

### Tensile testing of a single Napier fibre

The single Napier grass fibres were subjected to tensile testing using an Instron micro-tester and their tensile strength was determined according to ASTM D3822-07 standards. 50 mm was marked as the gauge length of each fibre and a 2 kN load cell was used for the testing. Ten samples were tested and the average mass, length, diameter, area, linear density and failure at break were determined by using a digital optical microscope and used to calculate their breaking tenacity, linear density, and single tensile strength.

## Results and Discussion

### Untreated and treated Napier fibre chemical composition characterisation

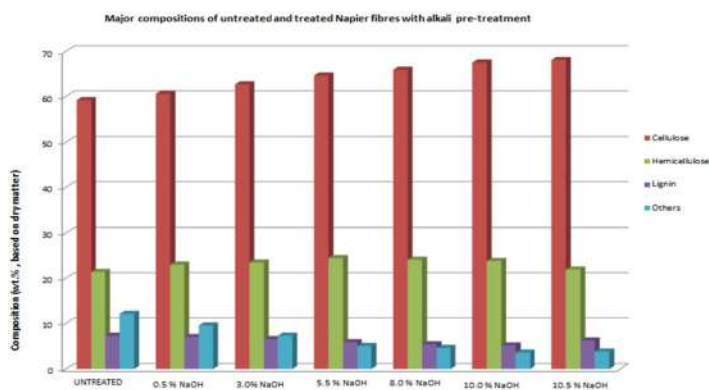
The major compositions of untreated and treated Napier fibres variation is shown in Table 1. It can be observed that the main compositions are cellulose, hemicellulose and lignin. The cellulose steadily increases from 59.19% of untreated material to 68.09% for 10.5 wt. % solution. It is also can be seen that lignin considerably decreases from 12.20% of the untreated to 3.80% of the treated fibre with 10.5 wt. % sodium hydroxide solution. The Napier fibres in which were treated with a greater proportion concentration of alkali solution showed better removal of lignin. Meanwhile, there are certainly no exact values about the percentage concentration of alkaline solution to be used in the chemical treatment of any lignocellulosic natural fibres. Therefore, most findings regarding the chemical treatments of natural fibres are founded on several experimental trials, which procedure varying amounts of alkali solutions.

Figure 5 indicates the estimated chemical composition of both untreated and alkali treated fibres. The chemical analysis of untreated fibres from Table 1 indicates the presence of cellulose, hemicellulose, and lignin. The examination of the results shown from figure 5 illustrates that alkali treatment reduced the lignin fraction extensively until 10.0% NaOH, however the increased of lignin and the reduced of hemicellulose was observed for 10.5%

alkali treated fibres. Hence, more investigation is required to validate and confirm such relationships. Generally, natural fibres are treated with alkali to get rid of weak amorphous constituents so that the fibre preserves crystalline components, as a result, improving the strength of the fibre [10].

Specimens	Composition (wt.%, based on dry matter)			
	Cellulose	Hemicellulose	Lignin	Other
untreated	59.19	21.35	7.26	12.2
0.5	60.57	22.95	6.98	9.50
3.0	62.76	23.42	6.54	7.28
5.5	64.71	24.39	5.87	5.03
8.0	65.97	24.03	5.41	4.59
10.0	67.53	23.76	5.12	3.59
10.5	68.09	21.88	6.23	3.80

**Table 1:** Major compositions of untreated materials with alkali pre-treatment.

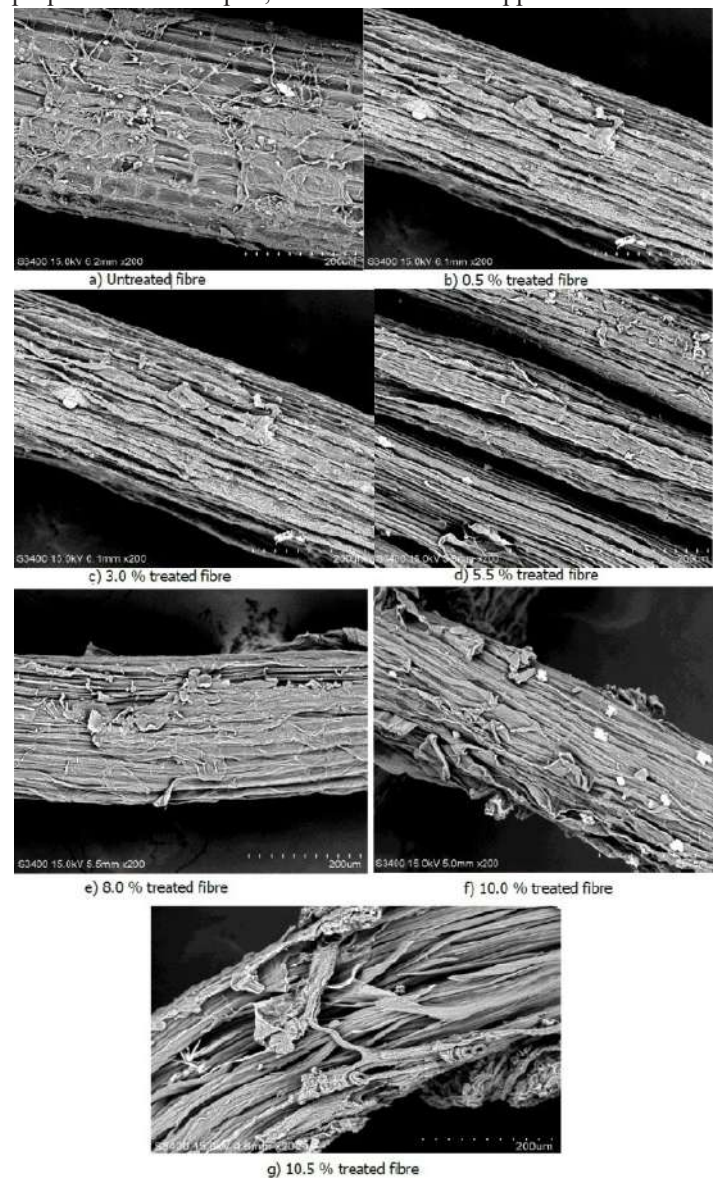


**Figure 5:** Composition of Napier fibre for the untreated and with various alkali pre-treatment.

### Surface morphology of untreated and treated Napier fibre by SEM analysis

The SEM micrographs of the morphology surfaces of the untreated and alkali treated Napier fibres are shown in Figure 6. It was essential to analyse the surface morphology of the fibres to detect the alterations that occurred on the surface of the Napier fibre after the alkali treatment. The SEM images revealed that the Napier fibre had a multi cellular arrangement. Figure 6 (a) shows the SEM images of the untreated fibre. The white film surrounded by the images denotes the existence of impurities on the surface of the Napier fibre. Figure 6 (b) shows the SEM image of the 0.5% alkali-treated fibre. It can be seen that the white layer decreased as this fibre is spotless than the untreated fibre due to the removal of surface impurities after the treatment. Nevertheless, some surface impurities can still be observed. Figure 6 (c–f) shows the 3.0, 5.5, 8.0, 10.0, and 10.5% alkali-treated fibres. The surfaces from the 3.0 until 10% alkali treated fibres had almost a similar form of surfaces but they were cleaner with fewer impurities. Figure 6 (g) shows the 10.5% alkali-treated fibres. The impaired roughness on the surfaces of the fibres can be observed. The 10.5% alkali treated fibres demonstrated an extra twisted and coarser surface than the 10.0% alkali-treated fibres. If these fibres were selective used in MDF, these rough surfaces would be predictable to promote and

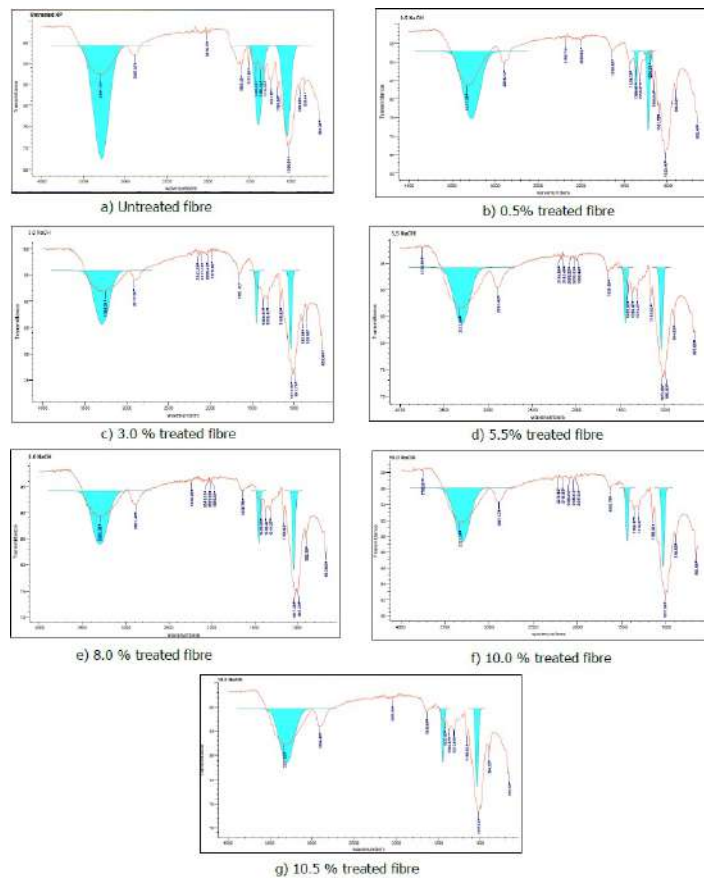
uphold good interfacial bonding between the fibres and the binder [11]. However, if the surfaces were coarser and grainier than a preferred level, this could decrease the mechanical properties of the fibres [12]. The Napier fibres contain a multi-cellular structure, which indicates a porous structure. The study done by Ridzuan et.al. [13] a hollow void recognised as a lumen exists inside the unit of the fibre demonstrates that the lumen structures vanish as the concentration of alkali treatment increases. In the other words, through the alkali pre-treatment it destroyed the cellular assembly of the Napier fibre, and therefore condensed the void content of the fibres. This can result in lower water absorption and explain the reduction of diameter for alkali-treated fibres [9]. Therefore, the alkali treatment may improve the mechanical and physical properties of the Napier, and thus enables its applications in MDF.



**Figure 6:** (a–g) Surface morphologies of untreated and alkali treated Napier fibres.

**FTIR spectra of the untreated and alkali treated fibres analysis**  
To confirm the changes in the composition on alkali treatment

of the Napier grass fibres, FTIR technique was used. The FTIR spectra of the untreated and alkali treated fibres are presented in Figure 7. From Figure 7(a), it can be seen that the untreated Napier fibre reveals precise bands at approximately 4000 to 1000  $\text{cm}^{-1}$  within its spectra. The small peaks at  $2887.67\text{cm}^{-1}$  linked with the existence of structure C–OH [13] and the peak at  $3307.12\text{cm}^{-1}$  can be described to the presence of  $\beta$ -glycosidic linkages between the monosaccharides [14]. The intense band, centred at  $2895.13\text{cm}^{-1}$ , is related with the C–O stretching modes of the hydroxyl and ether groups in the cellulose [15]. The peak centred at  $1030.91\text{cm}^{-1}$  indicates the C=C aromatic stretching with conjugated C–C bond and this peak is recognised to lignin content of the fibre [16]. The absorption band centred at  $1638.65\text{cm}^{-1}$  can be attributed to the C=O stretching vibration of the acetyl groups in the hemicelluloses [9]. The peak at  $1422.40\text{cm}^{-1}$  is a representative band for the C–H stretching vibration of CH and CH<sub>2</sub> in the cellulose and hemicelluloses components [17]. FTIR is used to determine the compositional changes that occur during the alkali treatment [18]. The bands for the 5.5 and 8.0 % alkali-treated fibres are relatively alike to those of the untreated fibre with slightly change. The bands at  $1420.53$ ,  $1314.27$ ,  $1366.47$ , and  $1636.78\text{cm}^{-1}$  had disappeared for the alkali-treated fibres. This indicates that the compositional changes of Napier fibre had occurred. Therefore, the FTIR investigations validate that the composition of the Napier fibres can be altered as the alkali concentration increases [19].



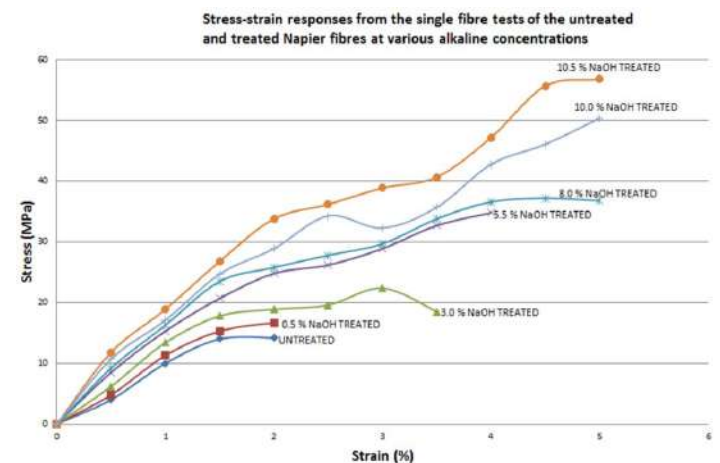
**Figure 6:** (a-g) FTIR analysis of untreated and alkali treated Napier fibres.

The single tensile properties of the untreated and alkali treated

fibres are presented in Table 2. The tensile strength of the fibre was measured for the 10 identical samples, by a ratio of the average load to average area. The Napier fibre diameter varied between 188.4  $\mu\text{m}$  and 211.5  $\mu\text{m}$ . The results show that the 10% NaOH-treated fibre exhibited the highest breaking tenacity of  $>6000\text{ N/tex}$ . The data shows that for the alkali treated fibres the maximum unit break increased significantly while the % elongation at break increased slightly. In untreated fibres, hemicellulose and lignin continue to spread in the interfibrillar area untying the cellulose chain from one another [20]. The cellulose chains are continuously in a state of constraint. Removal of lignin after alkali treatment, detached internal restraint and the fibrils developed are more capable of relocating themselves in a compact [10]. These indications lead to a closer filling of the cellulose chains, which enhanced the fibre strength and its tensile properties. These explanations were principally consistent with the chemical analysis data and SEM analysis.

fibres Specimen condition (%)	Mass (mg)	Length (mm)	Diameter ( $\mu\text{m}$ )	Area ( $\text{mm}^2$ )	Linear Density (Kg/m)	Unit break (N/Tex)
untreated	3.9	119.5	188.4	0.027	0.000282	457
0.5	4.8	120.1	190.2	0.029	0.000287	475
3.0	5.7	125.4	224.2	0.034	0.000312	506
5.5	6.8	128.1	257.2	0.051	0.000572	978
8.0	6.3	132.8	262.5	0.058	0.000591	1192
10.0	4.6	148.6	193.4	0.022	0.000279	5489
10.5	4.8	153.7	211.5	0.028	0.000316	5806

**Table 2:** Single fibre test (Tensile Test): Alkaline-treated single Napier grass.



**Figure 8:** Stress-strain analysis of untreated and alkali treated Napier fibres.

Figure 8 illustrates the responses of stress–strain from the single Napier fibre tensile tests of the untreated and treated fibres at various alkaline concentrations. Based on the figure, the maximum strength of 58.2 MPa was achieved by the 10.5% NaOH-treated Napier fibre. However, the lowest strength recorded is at 13.6 MPa, for the untreated Napier grass fibre specimens. This indicates that the strength increase at almost 70% between the untreated fibre and the 10.5 % NaOH-treated fibre. This results are based

on the explanation from the alkalisation process that breaks the fibre bundles into smaller fibrils, increasing the effective surface area for energy dissipation [10]. The growth in surface area allows effective adhesion interaction between the fibre and the binder, which should improve the interfacial bonding between both components in the fabricated MDF board [9].

## Conclusion

The morphological and compositional properties Napier fibres were investigated. Based on the findings of this study, it can be stated that there was dissimilarity in the Napier fibre surfaces after the alkali pre-treatment. The elimination of impurities (white film) by the alkali pre-treatment leads to the formation of fibrillation within Napier fibre in which the surface of the fibre become rougher. The available surface area is the key element for the extent of surface modification in Napier fibre morphology. Therefore, the focus of this research should be on the development of delignification method on the basis of the lignin and cellulose profile of Napier grass fibre. Subsequently, the main goal of the present work was to investigate the potential utilization of Napier fibres in MDF board production to improve raw material shortages in MDF industry.

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