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# The Effects of Chemical Treatments on the Mechanical Properties of *Miscanthus X Giganteus* Fibre

Ihueze Christopher C. [1], Okoli Ndubuisi C.[1], Onwurah Uchendu O.[1*] and Okafor Christian E.[2]

[1]Department of Industrial and Production Engineering
Nnamdi Azikiwe University, Awka Nigeria
[2]Department of Mechanical Engineering
Nnamdi Azikiwe University, Awka Nigeria

[*]Corresponding Author's E-mail: ou.onwurah@unizik.edu.ng

**Abstract**

The various advantages of natural fibres over the synthetic counterparts, the increase in environmental consciousness and the need for sustainable development have stimulated people's interest in natural fibres and their industrial applications. However, natural fibres need chemical modifications in order to overcome their main disadvantages in composites, which are poor compatibility between fibre and matrix and relative high moisture sorption. This study examined the effects of chemical treatments on the mechanical properties (tensile strength and modulus of elasticity) of *miscanthus giganteus* fibre as a potential reinforcing material in polymer industry. *Miscanthus* fibres were successfully extracted using water retting method and subjected to both mercerization and acetylation. The mechanical properties of the raw, mercerized, and mercerized plus acetylated fibres were tested. The results showed that chemical treatments increased the tensile properties of *miscanthus* fibre. However, the double stage chemical treatment (mercerization + acetylation) showed better properties compared to mercerization only. The findings of this study revealed that a modified *miscanthus* fibre has high tensile properties, hence, a potential good reinforcing material for polymer industries.

**Keywords***:* *Miscanthus* fibre, Chemical treatments, Mechanical properties, Reinforcing material, tensile strength.

## 1. Introduction

The increase in environmental consciousness and the need for sustainable development all over the world have stimulated researchers interest in natural fibre and their applications in various fields. The use of natural fibre as reinforcement in polymer composites has got considerable attention in numerous applications because of the good properties and superior advantages over the synthetic fibre like relatively low weight, low processing cost, good relative mechanical properties such as tensile modulus and flexural modulus, improved surface finish of moulded composite parts, renewable resources, being abundant, biodegradability, high toughness, high flexibility and ease of processing (Madhu et al., 2019; Neher et al., 2020; Khalid et al., 2021a; Ihueze et al., 2022).

Natural fibres are classified into three categories: plant fibres, animal fibres and mineral fibres. Plant fibres are very important type of natural fibres and these are generally comprised mainly of cellulose, hemicellulose, lignin, pectin, waxes and water soluble substances. The composition may differ with the growing condition and test methods even for the same kind of fibre. The plants, which produce cellulose fibre can be classified into bast fibre (jute, flax, ramie, hemp, and kenaf), seed fibre (cotton, coir, and kapok), leaf fibre (sisal, pineapple, and abaca), grass and reed fibre (rice, corn, and wheat), and core fibre (hemp, kenaf, and jute) as well as all other kinds (wood and roots) (Faruk et al., 2012).

Natural fibres are currently used in various applications such as building materials, particle boards, insulation boards, automobiles, plastics, aerospace, electronics, oil and gas pipeline, etc. (Chandramohan & Bharanichandar, 2013;

Thukur and Thukur, 2014; Sanjay et al., 2016; Khalid et al., 2021a, 2021b; Ihueze et al., 2021). However, the main drawback of natural fiber composites, which affects its mechanical properties, durability, and applications in various sectors, includes poor fiber-matrix interfacial bonding, poor wettability, water absorption and moisture absorption (Yashas Gowda et al., 2019). It is therefore necessary to modify the fibre surface by employing chemical modifications to improve the adhesion between the fibre and matrix (Zakikhani et al., 2014; Mohammed et al., 2015).

A lot of researches have already been be carried out on some natural fibres like coir fibre (Verma et al., 2013), plantain fibre (Okafor et al., 2013; Ihueze et al., 2012, 2017a, 2017b, 2017c, 2021, 2022), bamboo (Zakikhani et al., 2014; Yu et al., 2014), jute (Das et al., 2018), sisal (Ibrahim et al., 2016), kenaf (Yousif et al., 2012; Suharty et al., 2016), Dioscorea alata stem fibre (Okafor et al., 2012) etc., in order to ascertain their usefulness in polymer composites industries. From the findings of the above researches, the above natural fibres are very useful reinforcing materials for polymer composites for various industrial and domestic uses.

However, the Potential of other natural fibres, mostly weeds and wastes in the environment, for use in polymer composites needs to be explored. *Miscanthus X Giganteus* is one of the plant fibres which has been left out so far by researchers as a potential natural fibre that can be useful in polymer composite industry. It is a large perennial grass that is mostly seen as weed on the arable lands and waste in the environment. It is readily available, renewable, eco-friendly, biodegradable and contains a lot of fibres. In this study, the potential of *Miscanthus Giganteus* fibres for use as reinforcing material in polymer composite industries was explored.

## 2. Materials and methods

### 2.1 Materials

The following materials were used in this study: *miscanthus* fibres, sodium hydroxide, acetic acid and acetic anhydride.

*Miscanthus X giganteus* (Fig. 1) is a large, perennial grass that is mostly seen as weed on arable lands and waste in environments in the eastern Nigeria. It usually grows 8 – 12ft (240 – 360cm) tall each year. It is readily available, renewable, eco-friendly, biodegradable and contains a lot of fibres. The stems of *miscanthus* grass used for this study were obtained along the road and in plantations in Nnewi, Anambra State Nigeria.

**Fig. 1: *Miscanthus X Giganteus* Plant**

Sodium hydroxide (NaOH), also known as caustic soda, was used for mercerization of the extracted fibres. The essence was to remove unwanted soluble cellulose, hemicellulose, lignin, pectin, etc. from the fibres. Acetic acid was used to neutralize the NaOH used in mercerization of extracted fibres. Acetic anhydride was used for the acetylation of the fibres. Acetylation of natural fibres is necessary in order to stabilize the fibre cell walls against moisture, environmental degradation, improve dimensional stability and also to take off excess caustic soda. The above chemicals used in this study were obtained from Campal Scientific and Technological Company Onitsha, Anambra State Nigeria.

### 2.2 Fibre Extraction

Fibre retting process was employed in extraction of *miscanthus* fibres. *Miscanthus* stems collected were cut in sizes, washed in clean water to eliminate dirt and soaked in water bowl for 30 days at room temperature. After 30 days, the stems of *Miscanthus* were washed and fibres were extracted. The retting helped to separate the fibres from pectin, hemicelluloses and other impurities. The fibres extracted were washed with clean water and dried to constant weight in an oven for 150 minutes at oven temperature of $80^0$C. Fig. 2 shows the fibre extraction process and the extracted fibres.

**Fig. 2: Fibre Extraction Process**

### 2.3 Fibre Treatments

Both mercerization and acetylation were carried out in order to determine the effects of chemical treatments on the *miscanthus* fibres.

### 2.3.1 Mercerization

Six different samples of the extracted fibres were prepared and five of the samples were subjected to alkaline treatment (mercerization) using five different concentration of sodium hydroxide – 0.4%, 2%, 3.2%, 4% and 6% at room temperature for 60 minutes. After mercerization, the fibres treated with sodium hydroxide solution were neutralized with dilute acetic acid. The acetic acid was applied at 10%. That is, water to acetic acid ratio is 90:10. The resulted fibre were washed with a clean tap water until a pH of 7 was attained. The washed fibres were dried at room temperature for 48 hours followed by oven drying at 80$^0$C for 12 hours. Lab-Tech Ovum (Model no 01036, 230V, 600W) was used to reduce moisture content by 0.001% before subjecting them to tensile tests. Table 1 shows the samples – A(0% NaOH), B(0.4% NaOH), C(2% NaOH), D(3.2% NaOH), E(3.2% NaOH), E(4% NaOH) and F(6% NaOH).

**Table 1: Alkali Treatment of *Miscanthus* Fibres**

| Fibre Sample | Fibre treatment |
|---|---|
| A | Untreated *Miscanthus* Fibre (0% of NaOH) |
| B | 0.4% NaOH |
| C | 2% of NaOH |
| D | 3.2% of NaOH |
| E | 4% of NaOH |
| F | 6% of NaOH |

### 2.3.2 Acetylation

Three samples of *miscanthus* fibres were prepared and labelled G, H, and I, each sample was treated with 3.2% NaOH for 60 minutes and immediately subjected to acetylation. Sample G was treated with 5% acetic anhydride, Sample H was treated with 10% acetic anhydride, and sample I was treated with 15% anhydride for 60 minutes before they were removed, washed and dried in the oven at 80$^0$C for 24 hours.

### 2.4 Tensile Tests

Tensile tests were performed according to ASTM D638 using Hounsfield Tensometer at a crosshead speed of 1mm/min. Tests were carried out on Hounsfield Tensometer model –H20KW with magnification of 4:1 and 31.5kgf beam force. Each specimen was loaded to failure. 4 samples of each of the fibres treated with different concentrations of NaOH were subjected to tensile test to ascertain the tensile strength, modulus of elasticity and elongation at peak of *miscanthus giganteus* fibres.

## 3. Results and Discussion

### 3.1 Tensile Test Results of the Mercerized Samples

Table 2 shows the tensile strength, the modulus of elasticity and the elongation at peak of *miscanthus* fibres treated with different NaOH concentrations. Four replications of each sample were tested for each material property and the mean value in each case was obtained.

For the tensile strength, Fig. 3 shows vividly the trend in tensile strength of the *miscanthus* fibres as a result of chemical treatments. Sample A (the untreated fibres) has the least tensile strength of 128.059MPa. When the fibres were subjected to chemical treatments from 0.4% to 6% NaOH concentrations, the tensile strength of the fibres began to increase from 0% NaOH concentration till it got to 3.2% NaOH concentration, which has the peak value of tensile strength (627.450MPa) and gradually, the tensile strength began to decrease till it got to 6% NaOH concentration with tensile strength of 282.977MPa. From the figure, it shows that 3.2% is the right concentration of sodium hydroxide needed in mercerization of *miscanthus* fibres in order to obtain the desired tensile strength. 4% NaOH concentration is equally adequate for treatment of *miscanthus* fibres as it also gave high tensile strength, but, 3.2% NaOH concentration was selected as the best concentration for *miscanthus* fibre treatment and was used for the subsequent alkali treatments of the fibres in this study. This finding also agrees with the previous studies that found that alkali treatment improves the tensile strengths of natural fibres when compared with the untreated natural fibres (Hossain et al., 2013; Norul Izani et al., 2013; Cai et al., 2015; Yan et al., 2016). Faruk et al. (2012), Cai et al. (2015) and Yan et al. (2016) have reported poor tensile strengths of the various natural fibers they examined as a result of poor surface roughness which resulted to poor mechanical interlocking and strongly recommended proper chemical treatment in order to enhance the mechanical properties of the fibers concerned. Norul Izani et al. (2013) studied the effect of chemical treatment on the morphological and tensile strength of the oil palm empty fruit bunches EFB fiber. The treatments were by the types of treatments, 2% sodium hydroxide (NaOH) and combination of both NaOH and boiling water. The chemical treatment by NaOH led to enhancing the fiber surface topography, thermal stability, and tensile strength of the fiber, while the chemical treatment using NaOH and water boiling caused the higher thermal properties of the EFB fibers compared to untreated fibers.

Although, concentration of alkali treatment that gave their optimum tensile strength in each of the studies varies, but all recorded improvement in the tensile strength in relation to the untreated fibre. Hence, alkali treatment is needed in order to improve the tensile properties of *miscanthus giganteus* fibre.

**Table 2: Tensile Strength of the Mercerized Fibers**

| **Fibre Sample** | **Fibre treatment** | **Fiber Property** | **Replication** | | | | |
|---|---|---|---|---|---|---|---|
| | | | 1 | 2 | 3 | 4 | Mean |
| A | Untreated *Miscanthus* fibre (0% NaOH) | Tensile Strength (MPa) | 104.813 | 130.406 | 122.256 | 154.762 | 128.059 |
| | | Modulus of Elasticity (GPa) | 3.868 | 7.126 | 5.247 | 7.370 | 5.903 |
| | | Elongation (%) | 2.7 | 1.8 | 2.3 | 2.1 | 2.2 |
| B | 0.4% NaOH | Tensile Strength (MPa) | 238.391 | 334.347 | 539.166 | 388.682 | 375.147 |
| | | Modulus of Elasticity (GPa) | 8.669 | 12.158 | 16.692 | 11.886 | 12.351 |
| | | Elongation (%) | 2.8 | 2.8 | 3.2 | 3.3 | 3.0 |
| C | 2% NaOH | Tensile Strength (MPa) | 470.652 | 629.413 | 344.602 | 485.745 | 482.603 |
| | | Modulus of Elasticity (GPa) | 11.507 | 13.594 | 7.658 | 9.147 | 10.477 |
| | | Elongation (%) | 4.1 | 4.6 | 4.5 | 5.4 | 4.7 |
| D | 3.2% NaOH | Tensile Strength (MPa) | 267.746 | 456.401 | 815.677 | 969.974 | 627.450 |
| | | Modulus of Elasticity (GPa) | 7.296 | 13.229 | 20.091 | 26.358 | 16.744 |
| | | Elongation (%) | 3.7 | 3.5 | 4.1 | 3.7 | 3.8 |
| E | 4% NaOH | Tensile Strength (MPa) | 309.488 | 529.888 | 785.419 | 684.632 | 577.357 |
| | | Modulus of Elasticity (GPa) | 8.253 | 14.130 | 18.835 | 21.328 | 15.637 |
| | | Elongation (%) | 3.8 | 3.8 | 4.2 | 3.2 | 3.8 |
| F | 6% NaOH | Tensile Strength (MPa) | 180.742 | 374.888 | 297.921 | 278.358 | 282.977 |
| | | Modulus of Elasticity (GPa) | 6.232 | 10.472 | 9.398 | 8.512 | 8.654 |
| | | Elongation (%) | 2.9 | 3.6 | 3.2 | 3.5 | 3.3 |

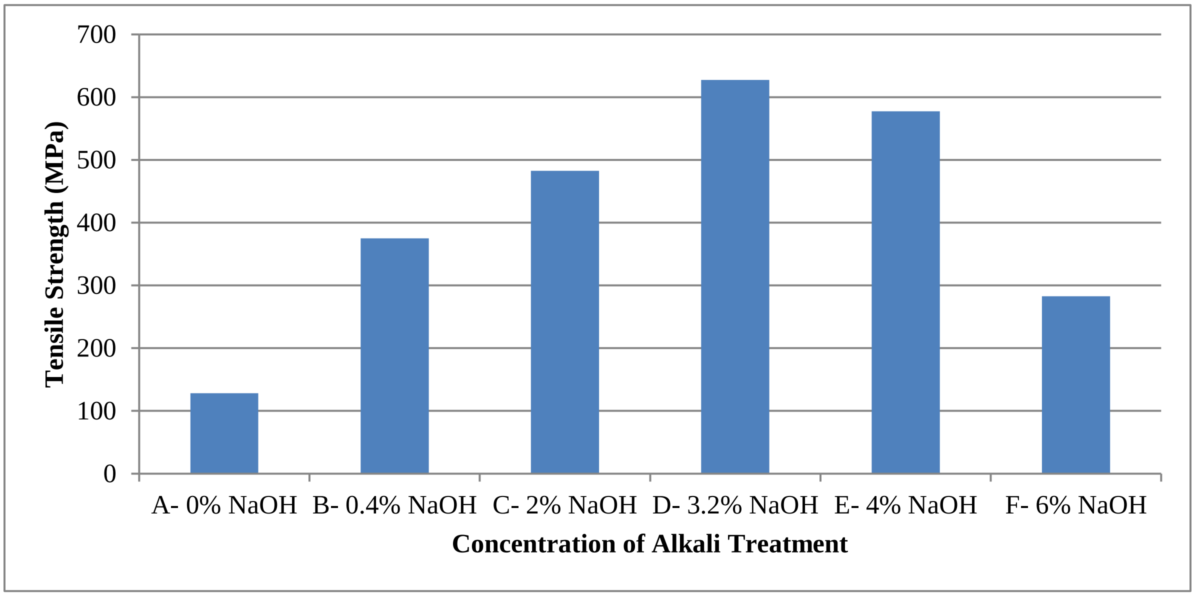

**Fig. 3: Tensile Strength of Mercerized Samples**

Fig. 4 shows the modulus of elasticity of *miscanthus* fibres treated with different concentrations of NaOH. The figure shows that the maximum modulus of elasticity (16.744GPa) occurs at Sample D, while Sample A has the least modulus of elasticity (5.903GPa). There was an increasing trend in the modulus of elasticity of *miscanthus* fibers till it got to Sample D, which has the peak value, and the trend began to decrease until it got to Sample F (8.654GPa).

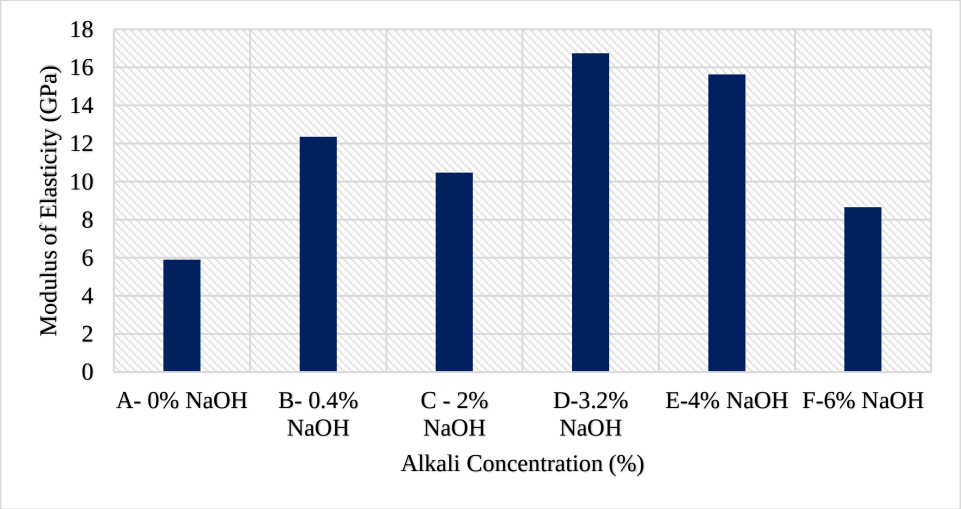

**Fig. 4: Modulus of Elasticity of Mercerized Samples**

Fig. 5 shows elongation at peak of *miscanthus* fibers treated with different concentrations of NaOH. The figure shows that maximum elongation occurs at Sample C while Sample A, the untreated fibers, is the sample that showed the least elongation at peak.

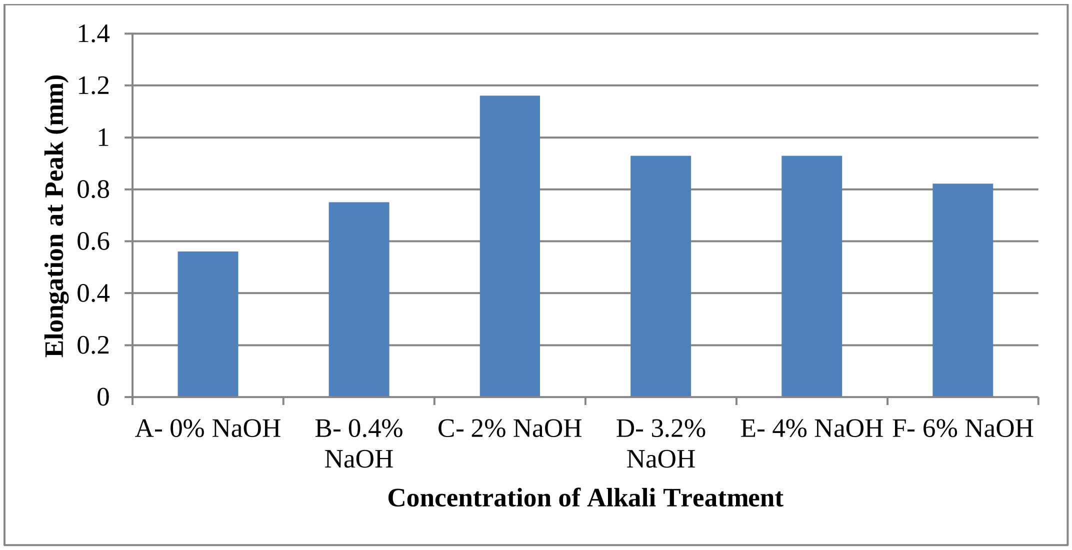

**Fig. 5: Elongation at Peak of the Mercerized Samples**

In order to ascertain the effects of chemical treatment on *miscanthus* fibres, the mechanical properties of the untreated and the treated fibres were compared. Table 2 shows that there is a great improvement in the mechanical properties of the treated samples over the untreated sample. Treating *miscanthus* fibre with 3.2% sodium hydroxide concentration gives 390% increase in the tensile strength of the fibre and 184% increase in the modulus of elasticity of *miscanthus* fibres over the untreated fibers. This finding is in agreement with previous studies that examined the effects of chemical treatments on natural fibres (Hossain et al., 2013; Chandramohan, 2014; Venkatesh et al., 2013). Venkatesh et al., 2013 reported 30% increase in the tensile properties of natural fibres as a result of alkaline treatment. Yan et al. (2016) reported 17.8% and 16.9% increase in the tensile and the modulus of elasticity of coir fiber respectively after alkali treatment.

### 3.1.2 Tensile Properties of Acetylated Samples

Figs. 6 and 7 show the tensile strength and the modulus of elasticity respectively of the acetylated fibre samples G, H and I. Sample G has the lowest tensile strength of 759.827MPa and modulus of elasticity of 20.080GPa, followed by sample I with tensile strength of 1085.800MPa and modulus of elasticity of 22.993GPa, and sample H has the highest tensile strength of 1261.719MPa and the modulus of elasticity of 33.867GPa. From the figures, it shows that treating mercerized *miscanthus* fibres with 10% acetic anhydride concentration gives better tensile properties than treating with 5% or 15% acetic anhydride concentration.

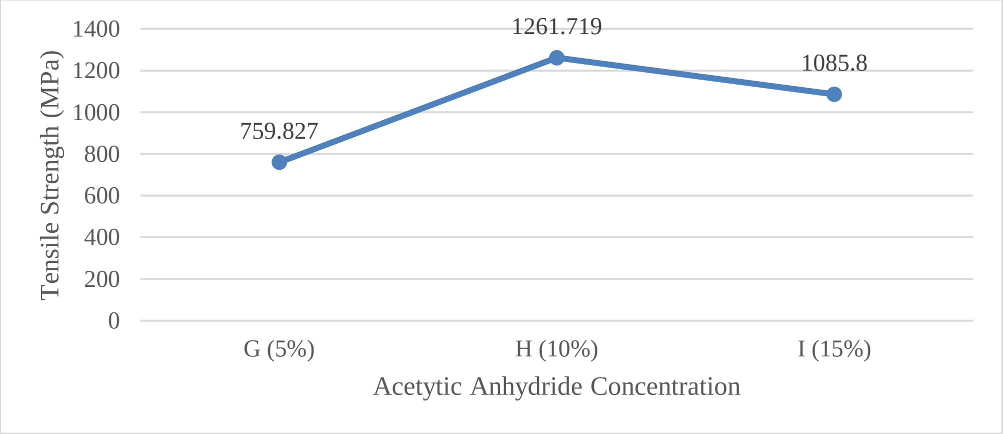

**Fig. 6: Tensile Strength of the Acetylated Samples**

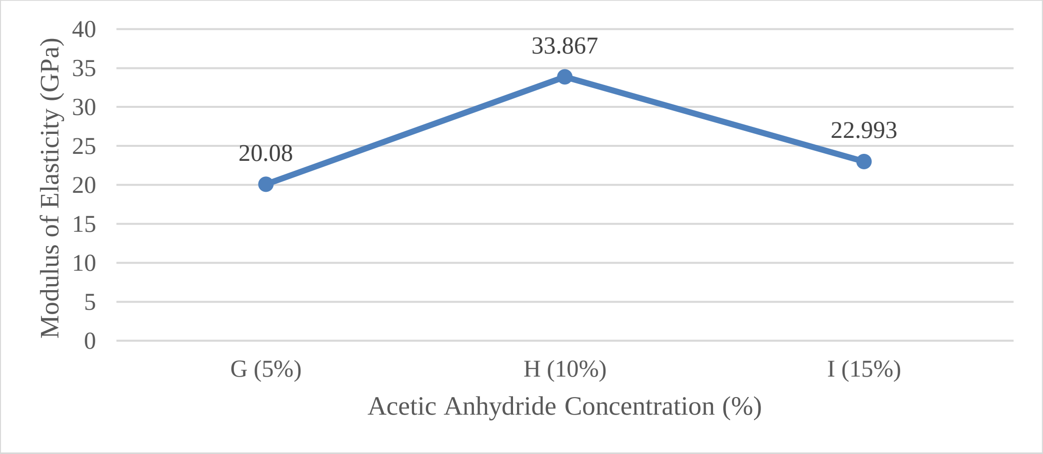

**Fig. 7: Modulus of Elasticity of the Acetylated *Miscanthus* Fiber Samples**

### 3.1.3 Comparison of the Effects of Different Chemical Treatments on the Tensile Properties of *Miscanthus* Fibres

Fig. 8 shows the comparison of the tensile strengths of untreated fibre, mercerized fibre (alkaline treatment only), and mercerized plus acetylated (fibre subjected to alkaline treatment followed by acetic anhydride treatment). The mercerized plus acetylated fibre has the highest tensile strength of 1261.719MPa, followed by mercerized fibre with tensile strength of 627.450MPa, and the least is untreated fibre with tensile strength of 128.059MPa. It can be seen from the figure that the tensile strength of *miscanthus* fibres increased as a result of chemical treatment of fibres, and the double stage treatment (mercerization + acetylation) produced the highest tensile strength of the *miscanthus* fibre. The mercerization gave 390% improvement in the tensile strength of *miscanthus* fibre, and double stage treatment while gave 885% improvement in the tensile strength over the untreated *miscanthus* fibre. Previous research also reported improvement on the mechanical properties of natural fibres as a result of chemical treatment. Mokaloba and Batane (2014) reported 173% and 435% increase in shear strength of sisal fibre as a result of mercerization and acetylation respectively; and 12.04% and 14.08% improvement in the tensile strength in both cases respectively.

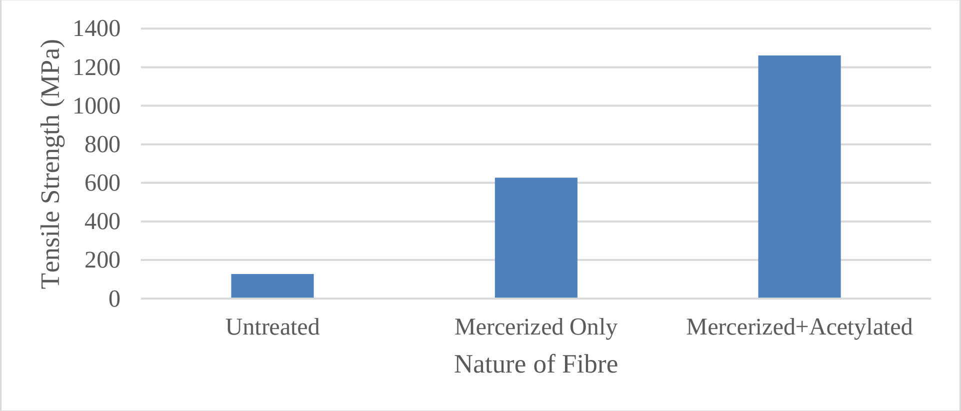

**Fig. 8: Comparison of the Tensile Strengths of treated and untreated Fibres**

Fig. 9 shows the comparison of the modulus of elasticity of untreated fibre, mercerized fibre, and mercerized plus acetylated. The fibre subjected to mercerization + acetylation has the highest modulus of elasticity of 33.867GPa, followed by fibre subjected to mercerization only with modulus of elasticity of 16.744GPa, and the least is the untreated fibre with the modulus of elasticity of 5.903GPa. Both treatments improved the modulus of elasticity of *miscanthus* fibres as both have modulus of elasticity that are greater than that of the untreated fibre.

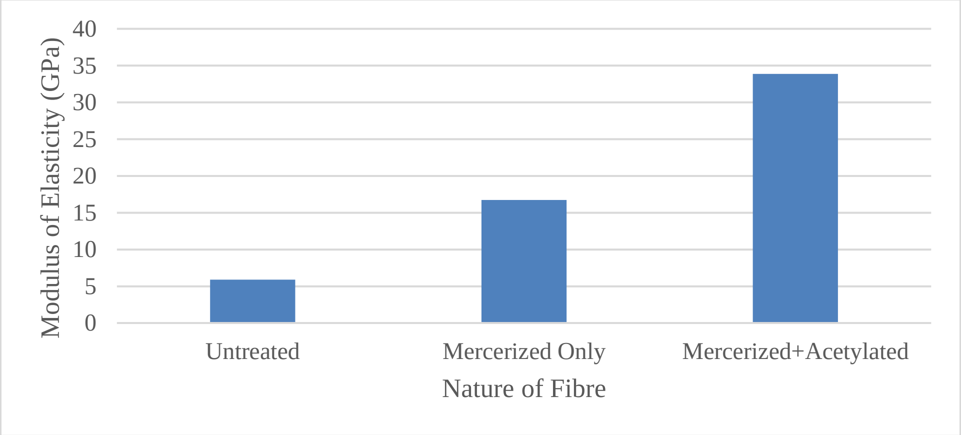

**Fig. 9: Comparison of Modulus of Elasticity of treated and untreated Fibres**

It can be deduced from the foregoing that both tensile strength and modulus of elasticity of *miscanthus* fibres increased as a result of chemical treatments. Furthermore, acetylation of mercerized *miscanthus* fibres increases both the tensile strength and the modulus of elasticity of an already mercerized fibres. This shows that double stage chemical treatments of *miscanthus* fibres enhance the tensile properties of the natural fibre.

## 4.0 Application of *Miscanthus Giganteus* Fibres

Natural fibers reinforced composites are emerging very rapidly as the potential substitute to the metal or ceramic based materials in applications that also include automotive, aerospace, marine, sporting goods and electronic industries (Thakur and Thakur, 2014). Natural fibers possess characteristics that are comparable to conventional materials. Properties like light weight, low material cost, renewability and being environment friendly are most important properties of fibers which make them to be used in the engineering applications (Neher et al., 2020; Khalid et al., 2021; Ihueze et al., 2022).

*Miscanthus giganteus* plant is readily available, renewable, eco-friendly, biodegradable and contains a lot of fibres. *Miscanthus* plant, which is mostly seen as weed on arable lands and waste in environments in the eastern Nigeria, needs to be put into useful applications in the industries. In this study, the mechanical properties of *miscanthus* fibre were assessed to ascertain its useful in natural fibre reinforced polymer composite for possible automotive applications. From the results obtained above, the mechanical properties of *miscanthus* fibre is adequate enough for reinforcement of polymers for industrial applications when compared with the mechanical properties of other natural fibres that have been used in similar applications as can be seen in Table 3. This implies that *miscanthus X giganteus* is a very good material for natural fibre/polymer composite industry and its usefulness in reinforcing polymers for automotive applications should be thoroughly harnessed. In the next phase of this study, optimal composition of *miscanthus* fibre reinforced polypropylene composites for motorcycle headlamp casing application will be determined and applied for production of motorcycle or motor headlamp casing.

**Table 3: Comparison of the Tensile Properties of *Miscanthus X Giganteus* Fibre and Natural Fibres**

| S/N | Natural Fibre | Tensile Strength (MPa) | Young Modulus (GPa) | Source |
|---|---|---|---|---|
| 1 | Kenaf | 930 | 53 | Codispoti (2014) |
| 2 | Hemp | 690 | 70 | Ilomaki (2012) |
| 3 | Sisal | 561 | - | Mokaloba and Batane (2014) |
| 4 | Fax | 345-2000 | 15-80 | Catto et al. (2014) |
| 5 | Jute | 393-800 | | Raja et al. (2017) |
| 6 | Bamboo | 500-700 | 30-50 | Abilash and Sivapragash (2013) |
| 7 | Coconut | 140-225 | 3-5 | Sinha et al. (2017) |
| 8 | Cotton | 400 | 12 | Codispoti (2014) |
| 9 | Abaca | 717 | 18.6 | Cai et al. (2016) |
| 10 | Ramie | 560 | 24.5 | Sonar et al. (2015) |
| 11 | *Miscanthus X Giganteus* | 1261.7 | 33.9 | The Present Study |

## 5. Conclusion

The effects of chemical treatments on the tensile properties of *miscanthus* fibres have been examined in this study. The present research demonstrated that chemical treatment clearly affected the mechanical properties of fibres obtained from *miscanthus giganteus* plant. The following were arrived at in this study:

a. Both mercerization and acetylation increased the tensile properties of *miscanthus* fibres.
b. 3.2% Sodium hydroxide concentration is the appropriate alkali concentration for the treatment of *miscanthus* fibres as it gave the highest tensile properties compared to other concentrations.
c. In double stage chemical treatment (alkali + acetic anhydride), 10% acetic anhydride gave the best tensile properties when compared with 5% and 15% concentrations.
d. Acetylation of already mercerized fibres increases both the tensile strength and the modulus of elasticity of the fibres.
e. The double stage chemical treatment (alkali + acetic anhydride) showed better tensile properties compared to alkali treatment only. Mercerization only made 390% increase in tensile strength of the fibre when compared with the untreated; while 885% improvement over the untreated fibre was made using the double stage treatment.
f. *Miscanthus X giganteus* fibre is a potential good reinforcing material for polymer industries as can be seen from its high tensile properties.

g. Its availability, eco-friendly, renewability, low cost and higher tensile strength provide it with an opportunity to replace the existing materials.

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