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Performance Enhancement of Biofibres: A Single Strand Specimen Route

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Abstract: This paper estimated the best route towards establishing the ultimate tensile strength of biofibres in this case, fibres of plantain pseudo stem, through the applications of strand of fibres and single fibre strand. These biofibres were extracted from the stem of harvested plantain tree through environmentally friendly biological retting and subjected to surface modifications of mercerization and acetylation. As strength of any engineering material has always been a criterion to determine its applicability in a particular engineering situation. All tensile strength experiments in this work were carried out in accordance with ASTM D3822-01. Through single strand tests, the performance enhanced biofibres were obtained at 2% NaOH concentration, 10% Acetic Anhydride concentration treatment at 50^{o}C acetylation temperature and dried at 80^{o}C oven temperature. Also the tensile properties of the developed biofibre when compared with other biofibres and e-glass fibre showed tremendous improvement in strength and modulus. This developed biofibre has wonderful properties that make it qualify to be applied as a reinforcement of polymer matrices or any other matrix for formation of engineering composite materials.
Keywords: Plantain pseudo stem, Tensile strength, Mercerization, Acetylation, Single fibre strand.

INTRODUCTION

Plantain stems are considered as wastes after matured fruits have been harvested from them. These agricultural by products are left in farms to only rut and decay whenever possible, but we are saying that this agricultural waste can be made to become an economical by-product and source of income for farmers.

The fact remains that, after harvesting of matured fruits from the plant, huge quantities of biomass wastes which include; pseudo stem, leaves, suckers etc., of over 90% of the plant is generated, hence something innovative needs to be done. In this research, fibres extracted from plantain pseudo-stem are to be used as bio or natural fibres for reinforcement of a polymer matrix composite to be used in production of composite pipe material for engineering applications. However, this article looks into the best route to determining the ultimate tensile strength of the plantain pseudo-stem fibre intended for reinforcement application after its surface modifications.

Fibres are the principal load carrying components of composites, the characteristics of these fibres significantly influence the effective mechanical properties of the composite produced from them. The mechanical and damage properties of individual constituents are essential in a micro-mechanical analysis of composites for effective properties and damage evaluation [1-3]. According to Ilankeeran *et al.* [4], most of high performance fibres are transverse isotropic with five mechanical constants to be determined in order to characterize the fibre. There are several techniques available to determine these constants. They adopted simple experimental techniques conducted on single fibre filaments to completely characterize their fibres such as ultimate axial strength, axial tensile Young's modulus and failure strain.

However, measuring the modulus and strength by conducting a tensile test on a strand of fibres (fibre bundle) is very popular in fibre manufacturing industries and researches. Kumar [5], puts it that, single fibre testing method is time consuming, also that strand of fibres (fibre bundle) testing is simple and can be done quickly and it gives the average properties, but there can be problems of aligning fibres in the stand with load applied, the friction between fibres, twisting of the fibres, etc.

This research validated the best route to determine the optimal axial tensile strength and Young's modulus of fibres in this case modified (mercerized and acetylated) plantain pseudo-stem fibre by comparing the results of tests conducted through the applications of strand of fibres (fibre bundle) and single fibre strand.

Fibre properties and composites design

The knowledge of basic mechanical properties of fibre and that of matrix is used in the evaluation of basic composite properties using some known classical equations. The rule of mixtures (ROM) equation for estimating the composite longitudinal modulus is classically expressed as

$$E_1 = V_{fr}E_f + V_{mr}E_{mr} \quad (1)$$

The transverse modulus is derived following the ROM model as

$$\frac{1}{E_2} = \frac{V_{fr}}{E_f} + \frac{V_{mr}}{E_m} \quad (2)$$

$$E_2 = \frac{E_f E_m}{V_{fr}E_m + V_{mr}E_f} \quad (3)$$

Where

E_1 = longitudinal modulus of composite (usually experimentally determined), E_2 = transverse modulus, E_f = elastic modulus of fibre, E_m = elastic modulus of matrix, V_{fr} = volume fraction of fibre, V_{mr} = volume fraction of matrix

The Halpin-Tsai equation for fibre reinforced composites is also expressed as

$$E_2 = E_m\left(\frac{1 + 2\beta V_{fr}}{1 - \beta V_{fr}}\right) \quad (4)$$

$$\beta = \frac{(E_f/E_m) - 1}{(E_f/E_m) + 2} \quad (5)$$

While the Brintrup equation is expressed as

$$E_2 = \frac{E'_m E_f}{E_f(1 - V_{fr}) + V_{fr}E'_m} \quad (6)$$

$$E'_m = E_m/(1 - \nu^2{}_m) \quad (7)$$

And ν_m is the Poisson's ratio of matrix material.

The random modulus of composite is estimated based on the rule of mixtures assumptions and equation (8) expressed as

$$E_1 = E_f \times V_{fr} + E_m \times V_{rm} \quad (8)$$

Crawford [6] suggested that since short fibres are likely to be randomly oriented that their stiffness could be predicted with

$$E = E_r = E_{random} = 3E_1/8 + 5E_2/8 \quad (9)$$

E_2 is regarded as the transverse modulus of the aligned fibre composite and is determined according to equations (3), (4) and (6).

Hull [7] proposed that the shear modulus and Poisson's ratio for a random short fibre composite could be approximated by

$$G_{random} = \frac{1}{8}E_1 + \frac{1}{4}E_2 \quad (10)$$

The Poisson's ratio of aligned fibre composites is estimated with

$$\mu_{random} = \frac{E_r}{2G_r} - 1 \quad (11)$$

RESEARCH METHODS AND MATERIALS

When measuring the fibre tensile strength in the single fibre tensile test, there is a certain probability that the fibre fails within the adhesives, paper tape or tab. In order to address this issue of failing in the gripping area, Phoenix [8] proposed a model that depends on the fibre Weibull parameters and the fibre stress distribution within the load transfer zone. Numerical analyses indicate that the rate of fibre failure within clamp area (outside of guage length) increases rapidly with decreasing guage length. Assuming stress concentrations near the end of the fibre close to the grip (clamp), fibres are likely to fail due to the testing method rather than the flaw population alone [9]. Stoner *et al*. [10] and [11] have suggested a model to account for failures caused by end effect.

This paper for all intents and purposes wants to compare the tensile strength of natural fibres measured through fibre bundles and single fibres in accordance with ASTM D3822-01, where single strands of each concentration of the mercerized materials were carefully selected. The diameter of each strand was carefully measured using digital micrometer screw gauge. However, these single strands have different diameters ranging from 0.04mm to 0.09mm. After

the diameter is recorded, each strand was taped at both ends with paper tapes allowing a gauge length of 25mm. Then the specimen is now mounted on the tensile testing equipment and the computer interface into which necessary parameters have been keyed-in, is now ready for the test once the test icon is clicked-on in the interface.

Fibre Retting and Extraction

Plantain pseudo-stems used in extraction of fibres used in this research were sourced locally from Akigwe plantain plantation in Awka, south east Nigeria. The extraction of plantain fibres were centered on an environmental friendly process of biological (water) retting process. These fibres were manually extracted after 28 to 34 days of retting which was our established best retting period for maximum yield of good quality fibres. These extracted fibres are with low lignin content, long fibre length, uniformity, quick drying tendency with high tensile strength which is essential for industrial purposes.

Tensile tests on dried fibre

The strength of any engineering material has always been a criterion to determine applicability of a novel material in a particular engineering situation. Therefore, the use of tensile strength was applied in determining the effective drying temperature, treatment concentrations for the fibre material under development. This tensile tests were conducted at Polymer and Textile Engineering department laboratory, Nnamdi Azikiwe University, Awka, using a tensile machine named Kejian Instrument model KJ-1065 made by KEJIAN-TECH., China, with a capacity of 10kg (98.1N) breaking force.

Oven drying temperature

In order to obtain an approximate 0% moisture content for any fibre or fibre bundles an application of standard oven drying process is necessary. Meanwhile, in performing this drying temperature experiment, standard ovens and an electronic weighing balance were used. The oven drying temperature experiment was set at three drying temperatures of 50^0, 80^0, and 100^0C, hence three ovens used were labeled A, B, and C respectively. During the stage of developing our plantain pseudo stem fibres for this research, the bedrock experiment was the tensile experiment to determine the tensile strength of each fibre material at various stages of study. Invariably, the temperature that gave us the material with the best ultimate tensile strength was automatically adopted.

Mercerization Treatment

The first mercerization treatment on the fibres was performed using 1mole (4%) concentration of Na OH which was reported by many researchers such as [12-14] as optimum concentration for the natural fibre they studied. Unfortunately, mercerization at this concentration was found to have adverse effect on the plantain fibre thereby weakening the fibre strands. Because of this finding, the extracted fibres were then mercerized at three different concentrations below 4% (I mole). These concentrations are 0.1mole (0.4%), 0.5mole (2%), and 0.8mole (3.2%) of NaOH solutions. These developed fibres were tested for tensile strength as fibre bundles (strand of fibres) and single fibre strand after drying at our established drying temperature.

Acetylation Treatment

The treatment of natural fibres with acetic anhydride solution is known as Acetylation and sometimes called Esterification. Since, we have established optimum concentration of NaOH for the mercerization treatment, we go further to treat these fibres with acetic anhydride solution. Some portion of the mercerized fibres were treated with acetic anhydride solution at different concentrations of 5%, 10%, and 15% under two different temperatures of 30^0C (room temperature) and 50^0C.

In other to maintain an environmental temperature of 50^0C, a water bath was used in the treatment process. The water bath used in this treatment is an AISET water bath model DK-420-YLJYE-100 made by G. Bosch, Germany, and was made available at Industrial/Production Engineering department laboratory, Nnamdi Azikiwe University, Awka. After acetylation treatments at different concentrations, the material samples were subjected to tensile, applying the same equipment parameters and in accordance with ASTM D3822-01 standard as in the previous tensile tests.

Fig-1: Bundles of Fibres of different concentrations and diameters set for testing

Fig-2: Samples after Tensile Testing Process

Fig-3: Single strand after testing

RESULTS AND DISCUSSION

Oven drying temperature

After the extraction of fibres, we carried out the investigation into the establishment of the optimum temperature at which these fibres will be dried. Three ovens were used set at 50^0C, 80^0C, and 100^0C respectively, after drying to almost zero percent (0%) moisture content, the fibres were subjected to tensile tests and the results were analyzed with bar charts as shown in Figs. 4 to 6. For the tensile tests, different samples were prepared with three distinct diameters obtained through twisting of fibre bundles of different number of strands. The three diameters are 0.5mm, 1.0mm, and 1.5mm measured with a digital micrometer screw guage with five (5) replications of each diameter.

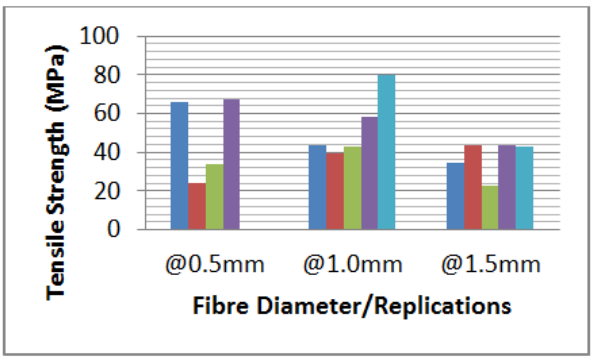

Fig-4: Strengths at drying temperature of 50^{0}C at varying diameter of fibres

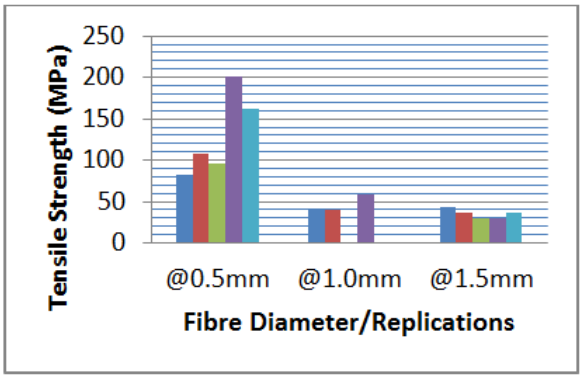

Fig-5: Strengths at drying temperature of 80^{O}C at varying diameter of fibres

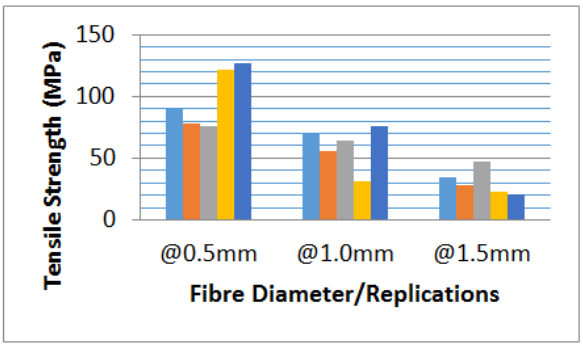

Fig-6: Strengths at drying temperature of 100^{O}C at varying diameter of fibres

From the results recorded in the experiment of oven drying temperature suitable for the material under development, it was shown that fibres dried in oven temperature of 80^{0}C gave optimum strength of 201.340MPa. This implies that the temperature for drying these fibres subsequently through the research will be at 80^{0}C; hence this oven temperature is adopted.

Mercerization treatment and tensile tests with Fibre Bundle

Like the drying temperature tensile experiments, the specimens were prepared in terms of fibre bundles of 0.5mm, 1.0mm, and 1.5mm diameters and each replicated five times. The bundles are paper taped at both ends to hold the fibres in place basically at the guage length distance of 25mm in accordance with ASTM D3822-01. The tensile testing equipment was set at 1.667mm/s testing speed for 30 seconds.

Table-1: Results for 0.5M (2%) NaOH Solution Concentration at 0.5mm Diameter

Replications	Force @ Peak (gf)	Elong. @ Peak (mm)	Tensile Strength (gf/mm^2)	Tensile Strength (MPa)	Elongation percentage @ peak(%)	Area (mm^2)	Gauge-Length (mm)

R1	4574.080	1.881	23337.143	228.859	7.525	0.196	25.000
R2	5220.040	1.869	26632.857	261.179	7.475	0.196	25.000
R3	2818.051	2.263	14377.811	140.998	9.050	0.196	25.000
R4	6815.024	1.794	34770.531	340.983	7.175	0.196	25.000
R5	5016.075	1.894	25592.219	250.974	7.575	0.196	25.000

The results of these tensile experiments showed that for 3.2% (0.8 mole) concentration, tests were successfully carried out at the three fibre bundle diameters, but the ultimate (optimum) tensile strength for the mercerized fibres was recorded as 340.983 MPa at 0.5M (2%) alkaline concentration (Table 1). But there was an interesting observation during the tensile experiment; the tensile testing apparatus was able to break all ranges of diameter for 0.8M (3.2%) concentration treatment. This equipment has a capacity of 10kg or 10000gf (98.1N) breaking force, however, it was not able to break the materials of 0.5M concentration at 1.0mm and 1.5mm diameter and that of 0.1M concentration at 1.0mm and 1.5mm diameter. This shows that the force needed to break these materials is more than what the tensile machine could produce. Rather, when these specimens were forced to break, the following observations were recorded (Figs. 7 and 8). Meanwhile, this phenomenon does not necessarily mean that the tensile strength could exceed the optimum already recorded as this engineering parameter (tensile strength) is a function of force and area.

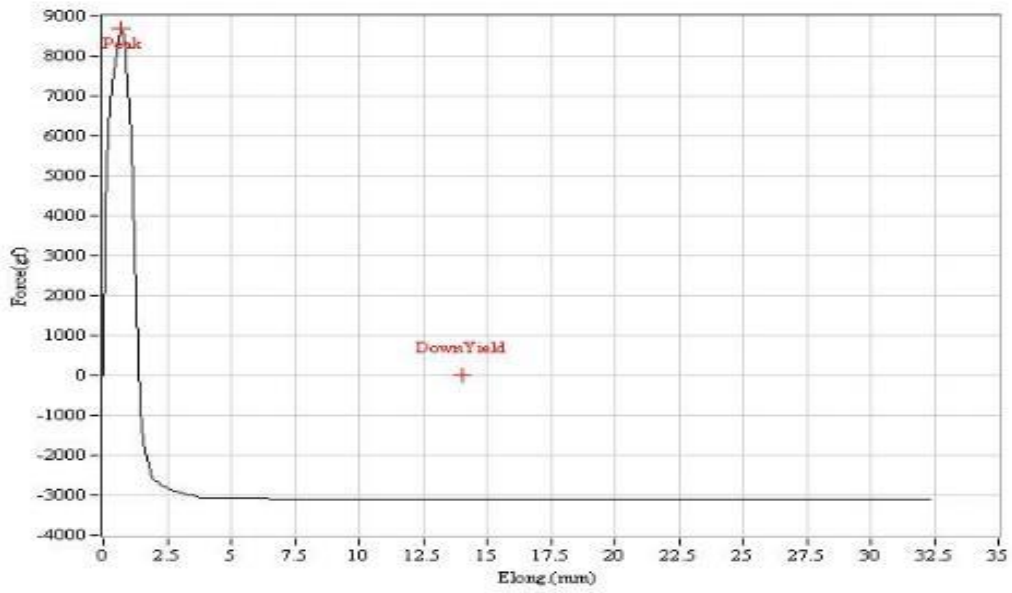

Fig-7: Plot of Over Loaded Machine at 0.5M concentration and 1.0mm diameter

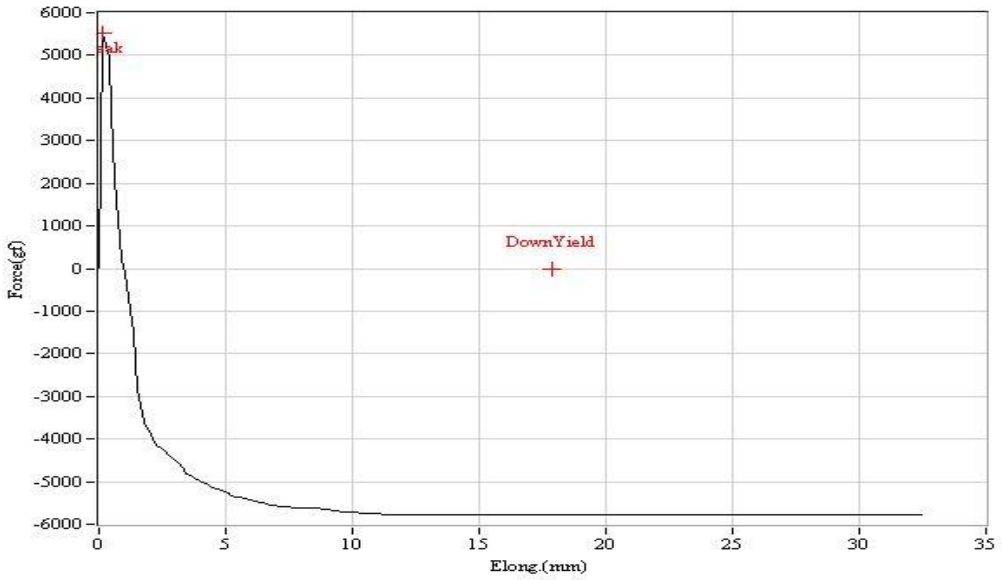

Fig-8: Plot of Over Loaded Machine at 0.1M concentration and 1.0mm diameter

The above figures of Figs. 7 and 8 indicate that when the maximum breaking force of 10000gf is exceeded the plot goes to the negative value, hence on addition of these values (positive and negative) gives the force needed to actually break the specimen (for instant, Fig. 7 is 11,350gf). This is an indication that the Tensile strength measured using strand of fibres (fibre bundle) could not actually give the optimum tensile strength of the fibre understudy.

Tensile tests with single fibre strand

Mercerization or alkaline treatment of the fibre was carried out at different concentrations of NaOH solutions, which are 0.4%, 2%, and 3.2% concentrations and untreated of 0%. The results obtained from tensile tests conducted on these modified single fibre strands were recorded and analyzed with bar chart of Fig. 9, then at optimum concentration 2%, relevant graphs were plotted as shown in Figs. 10, and 11.

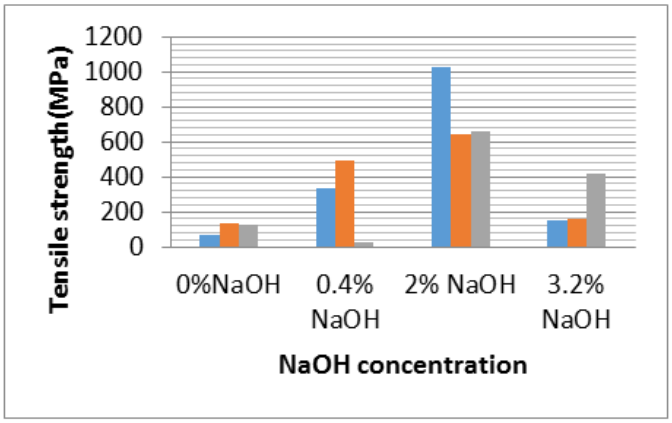

Fig-9: Strengths at Varying Na OH Concentration

Various graphical plots at the recorded optimum concentration point of 0.5M are represented as follows;

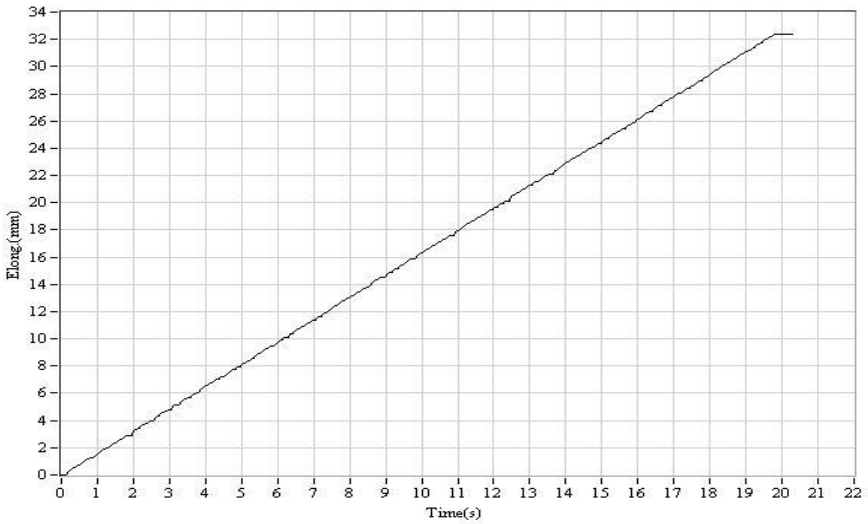

Fig-10: Elongation Vs Time Plot at Optimum Strength of 2% concentration

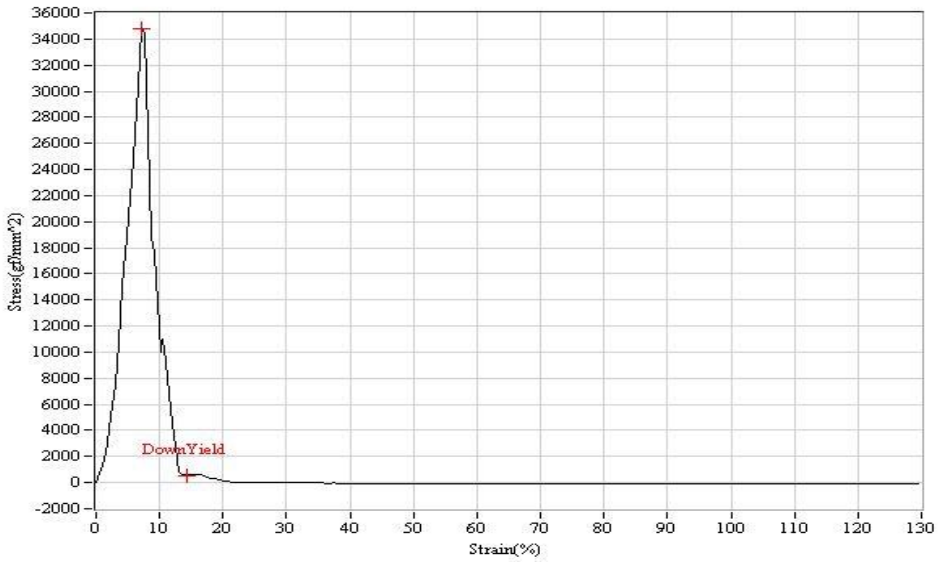

Fig-11: Stress Vs Strain Plot at Optimum Strength of 2% Concentration

This research reports that the ultimate (optimum) tensile strength of the Plantain fibre under study is 1024.986 MPa recorded at 0.5M (2%) NaOH solution concentration and 1% acetic acid treated. This particular concentration and drying temperature of 80^0C were taken as benchmark for other fibre treatments of such throughout the research. However, it is established that there was a tremendous increase in strength of about 770% through alkaline treatment at optimum concentration.

Acetylation Treatment – Single Strand

It is now very clear that we have follow the harder right instead of the easier wrong, therefore, we are adopting the use of single fibre strand for the rest of our tensile strength experiments. The mercerized fibres were also treated with acetic anhydride at two acetylation temperatures of 30^0C and 50^0C for 5%, 10%, and 15% concentrations of acetic anhydride solutions. Their strengths were further evaluated with results recorded and also analyzed with bar charts of Figs. 12, and 13 respectively. While Figs. 14 to 15 are plots at optimum tensile obtained from 10% acetic anhydride concentration and 50^0C acetylation temperature.

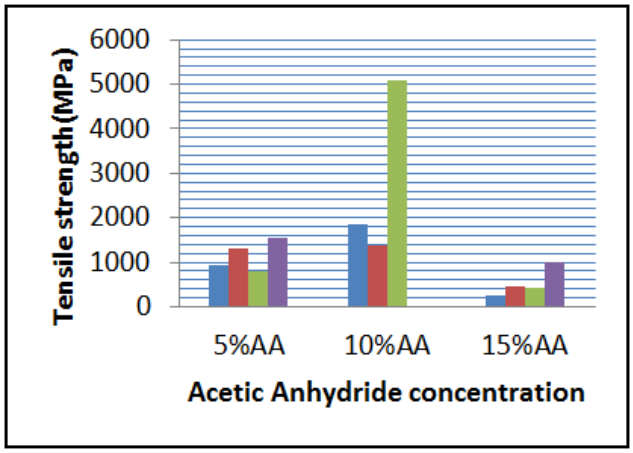

Fig-12: Strengths at varying Acetic Anhydride concentrations at acetylation temperature 50^0C

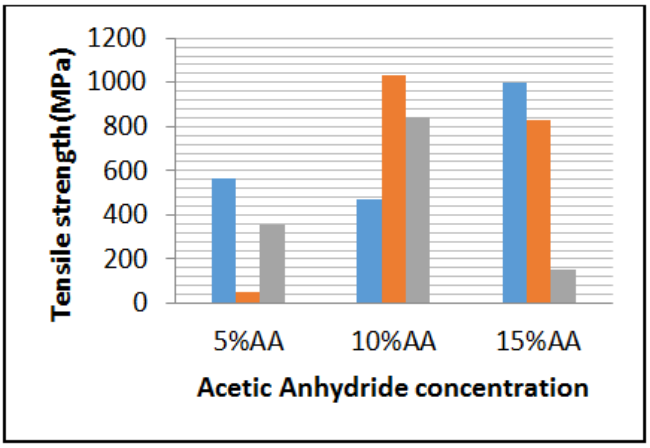

Fig-13: Strengths at varying Acetic Anhydride concentration at acetylation temperature 30^0C

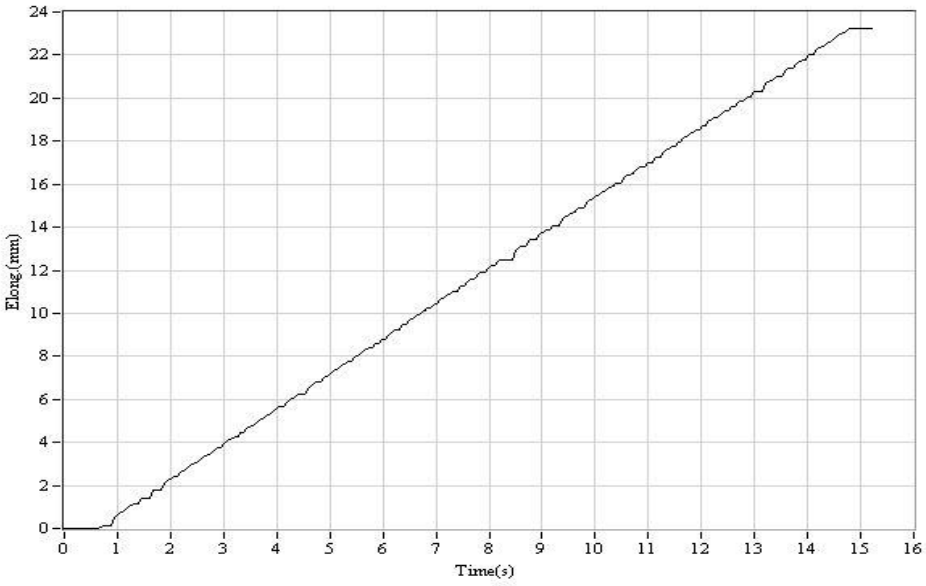

Fig-14: Elongation Vs Time Plot at Optimum strength of 10% concentration at 50^0C

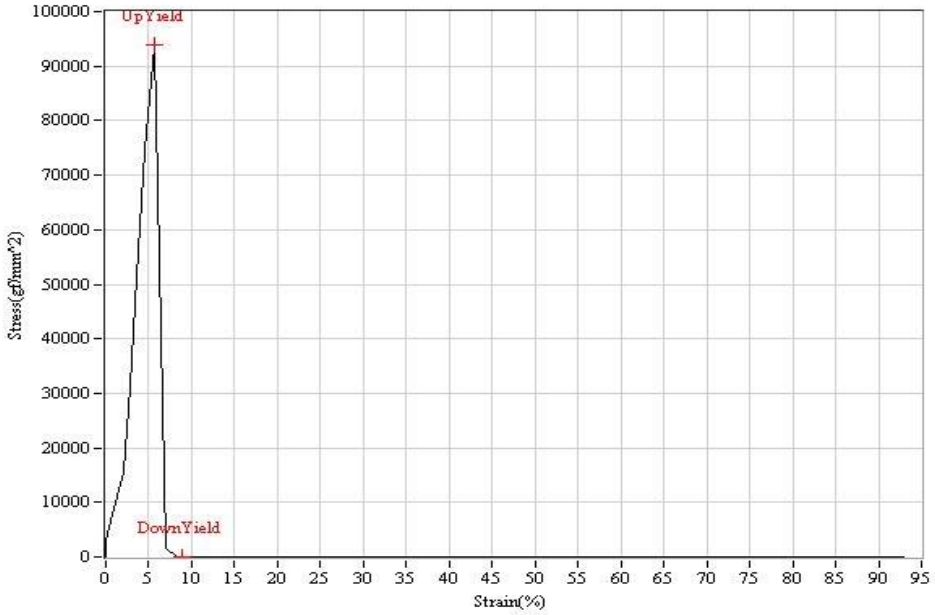

Fig-15: Stress Vs Strain Plot at Optimum Strength of 10% Concentration at 50^0C

The test for strength conducted on the acetylated fibres indicates an optimum tensile strength of an average of 2783.660MPa for those fibres acetylated at 50^0C and at 10% acetic anhydride concentration. The results also show further increase in strength of about 272% when the fibre surface is further modified with acetic anhydride. Meanwhile, a treatment combination of 2% NaOH, 15% acetic anhydride at 50^0C and oven drying temperature of 80^0C is now the benchmark for the modification of the material development. At this juncture, the developed plantain pseudo-stem fibre has a Young's modulus of 98,067 MPa (98.07 GPa) which was calculated from the stress-strain graph of Fig. 14.

However, besides the tensile property, other properties of this developed plantain pseudo stem fibre along with some other natural fibres are compared in Table 2, this enables us understand the good and trade-off properties of the developed fibre material.

Table-2: Compared Properties of Fibres with E-Glass Fibre

Fibre	Density (g/cm^3)	Tensile strength (MPa)	Specific tensile strength (MPa)	Elastic modulus (GPa)	Specific elastic modulus (GPa)
E-glass	2.5-2.55	2000-3500	800-1373	70-73	28 - 28.62
Flax	1.4	800-1500	571.43-1071.42	60-80	42.86
Hemp	1.48	500-900	337.83-608.11	70	47.30
Jute	1.46	400-800	273.97-547.96	10-30	6.85-20.55
Ramie	1.5	500	333.33	44	29.33
Coir	1.25	220	176	6	4.8
Sisal	1.33	600-700	451.13-526.32	38	28.57
Abaca	1.5	980	653.33	12	8
Cotton	1.4-1.51	400	285.71-264.90	12	8.51-7.95
Kenaf	1.45	930	641.38	53	36.55
Unmodified Plantain stem	1.358	70-240	51.55 - 97.20	3 - 9.6	2.22 - 7.06
Modified Plantain stem	0.132	2800	7757.58 - 21212.12	40-98	303.03 - 545.46

CONCLUSION

Kumar [5] stated that investigating the tensile strength of fibres by the application of single fibre strand is very time consuming, while that of fibre bundle adopted by most industries and researchers is pretty easy but result is always average. On this premise, and on the findings of this research, it better to follow the harder right, instead of the easier wrong, and the following conclusions are drawn from this paper:

- Those plantain fibres can be finely extracted after 28 to 34 days of retting at temperature between 30^0C to 36^0C.
- These extracted fibres has its optimum strength when treated at 2% (0.5 mole) NaOH concentration, and at 10% acetic anhydride concentration acetylated at 50^0C, with these fibres being dried at 80^0C oven temperature.
- This paper estimated the tensile strength of plantain pseudo-stem fibre studied under strand of fibres (fibre bundle) as 340.983 MPa at 2% (0.5M) concentration. Subsequently at the same 2% alkaline concentration the uptimum tensile strength of 1024.988 MPa was recorded for single srand fibre, hence single alone was adopted for strength test at acetylation treatment.
- Increase in tensile strength was observed at 10% concentration acetic anhydride treatment recorded as 2783.660 MPa to 5099.586 MPa.
- Comparing some physical properties of this developed fibre and other natural fibres and e-glass fibre clearly indicated a fibre with very good and unique properties.
- Conclusively, the research has proved that the best route to establishing the tensile strength of fibres is through single strand.

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