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Comparing the Effect of Mercerisation, Acetylation and Oxidation on the Tensile Properties of *Luffa Cylindrica* Fibers

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Abstract

Treatment of natural fibers is an important way of improving their properties for specific applications. *Luffa cylindrica* is an excellent low-cost fiber for reinforced composite applications. This study aimed to investigate the influence of mercerisation, acetylation and oxidation pre-treatment on the tensile properties of *Luffa* fibres. It was observed that the stress-strain relationship for *Luffa* fiber is linear in the elastic region, with the untreated fiber withstanding the highest value of force in this phase. This proportional relationship was also consistently observed at the elastic region for all treatment types employed. The tensile strength of the untreated fibers was 7.083 MPa. There was an improvement in the tensile strength with acetylation (7.541 MPa) and a reduction due to oxidation (5.11 MPa) and mercerisation (5.517 MPa). We observed that the stress and strain within and outside the elastic region differed across treatment types and elastic regimes. Therefore, the study highlights the importance of considering the specific application when selecting the appropriate *Luffa* fibre treatment method.

Paper type: Research paper

Keywords: Acetylation, *Luffa* cylindrica fibers, tensile properties, Mercerisation, oxidation.

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Introduction

Natural fibers are readily available and abundant in nature. Due to their low cost and renewability, they serve as a viable alternative in polymer reinforcement compared to synthetic fibers which are expensive and non-renewable (Adeniyi *et al*., 2019; Adeniyi *et al*., 2019). Sponge gourd (*Luffa cylindrica*), commonly referred to as vegetable sponge or *Luffa* cloth, is obtained from the matured dried fruit of *Luffa* (See **Figure 1(a-b)**) (Chen *et al*., 2017). It has different vernacular names like ridge gourd, angled *Luffa*, Chinese okra, dish-cloth gourd, ribbed *Luffa*, Silk gourd, Sinkwa towel sponge, Sinqua melon and vegetable sponge (Panicker *et al*., 2019). The fibrous vascular system of the dried sponge gourd fruits forms a natural mat that can deflect cracks and increase the toughness of resins (D'Almeida *et al*., 2006). Their biodegradability can contribute to a healthier ecosystem and their low cost and reasonable performance fulfil the economic interests of various industries (Tanobe *et al*., 2005). Moreover, many environmentally conscious consumers appreciate that *Luffa* products are biodegradable, natural and renewable resources. The tough fibers can promise as being processed into industrial products such as filters, insulation and packing materials (Demir *et al*., 2008). *Luffa* is a sub-tropical plant, which requires warm summer temperatures and a long frost-free growing season when grown in temperate regions (Oboh and Aluyor, 2009). In principle, the advantages of using natural fibres such as *Luffa* in composites include low density, high flexural strength, flexibility and high elastic modulus (Alshaaer *et al*., 2017).

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Tanobe *et al*. (2005) conducted chemical treatments of Luffa fibers using 2% NaOH 2% and (1–3%) methacrylamide. Methacrylamide treatment severely damaged the fibers. NaOH on the other hand, showed the same beneficial effect regarding enhancement of surface area and thermal stability together with similar levels of lignin and hemicellulose extraction, without causing exaggerated harm to fiber integrity. Ghali *et al*., (2009) treated the *Luffa* fibers with two methods: alkali treatment and mixed treatment (sodium hydroxide and hydrogen peroxide). Both treatments resulted in the removal of lignin, pectin and hemicellulose substances, and change the characteristics of the surface morphology. Mohanta and Acharya (2016) studied the effect of fiber surface using 5% NaOH, benzoyl chloride, and 0.05% potassium permanganate at room temperature. It was observed that the chemically treated fibers improved the final mechanical properties of the *Luffa*-reinforced epoxy composites. A similar observation of improvement of the mechanical properties of the final *Luffa*-reinforced polyester composites due to NaOH and acetic acid treatment of the fibers was observed by Ghali *et al*., (2011).

Chen *et al*., (2019) investigated the effects of three alkali-based softening treatments on the properties of high-density *Luffa* fiber bundles. They observed that the treatment with 5% NaOH/5% H_2O_2 significantly lowered the compressive strength and plateau stress of the fibers. However, the treatment using 10% NaOH/20% CH_3COOH was considered to be the best. Zhang *et al*., (2019) studied alkali-hydrogen peroxide, alkali-acetic acid and alkali-urea treatments of *Luffa*. The treatments were observed to greatly improve the compressive strength of the fibers. Premalatha *et al*., (2019) studied the use of NaOH, potassium permanganate, benzoyl peroxide and stearic acid to reduce the hydrophobic nature of *Luffa* fibers. It was noticed that all modified *Luffa* fibers improved crystallinity and thermal stability. Though the stearic acid treatment was superior for the thermal stability property.

(a) **(b)**

Fig. 1 The *Luffa* pods (a) and its fibre (b) (Adeyanju *et al*., 2021).

There is no consensus on the best modification technique for *Luffa* in the domain of tensile properties. In this work, we investigate the influence of chemical treatment on the tensile properties of *Luffa* fibres. Three chemical compounds were used based on the pre-experiments to modify the *Luffa* fiber, which includes NaOH, CH_3COOH, and $KMnO_4$. The reagents were used for the mercerisation, acetylation and oxidation of *Luffa* fibers respectively. The effects of the three treatment methods on the tensile properties of *Luffa* fiber bundles were investigated by tensile strength test.

1 Materials and Methods

1.1 Materials

The *Luffa* fibers were sourced from Ilorin town, Nigeria. The outer bark of the already dried pods was removed leaving behind a neat fiber for study. The chemicals (analytical grade) used in the study were sodium hydroxide (NaOH), glacial acetic acid (CH_3COOH), potassium permanganate ($KMnO_4$) and distilled water (H_2O).

1.2 Fiber modification

For the fiber modification, 0.25 M sodium hydroxide solution was used for mercerisation, 30vol% acetic acid solution was used for acetylation whilst 0.25M potassium permanganate solution was used for oxidation. The seeds within the dry fiber framework were carefully removed and the fibers were immersed in the modification agent for 24 hour at room temperature 25±^{o}C. The fibers were then removed and sundried for two days before further tests and characterisation was carried out. The tests were designated as test 1-oxidation (potassium permanganate), test 2- untreated (raw), test 3-acetylation (acetic acid) and test 4-mercerisation (NaOH).

1.3 Tensile tests

Before testing, the cutting of fibre network was performed along the longitudinal length of the *Luffa* bunch for tensile characterisation. The measurement and analysis were carried out using UTM 500AT equipped with machine response analysis software. The tensile tests were conducted at room temperature (25ºC), according to ASTM Standards. Tensile loads were applied till the failure of the sample and stress–strain curves were obtained for all samples. Three specimens were used for all the tests and the final results represent the average. The major tensile properties of the fibres in different treatment methods were also obtained.

2 Results and Discussion

2.1 Stress-strain behaviour of the fibers

Luffa is a natural woven fibre with a net-like fibrous vascular system. When they are treated and dried, the fibrous network structure becomes readily open like cell foam material (Shen *et al*., 2012). This makes the stress-strain relationship to be linear in the elastic region as observed in **Figure 2**. The maximum load for treatments 1, 2, 3 and 4, in the elastic regime, are 80.30N, 113.00N, 122.20N, and 86.70N respectively. This confirmed the untreated fibre withstood the highest value of force in the elastic phase. Beyond the elastic regime, the further applied forces were no longer proportional to the elongation produced, hence inelasticity resumes. The array of peak forces for each treatment (1-4) was found to be 64.70N, 104.90N, 118.50N and 86.70N respectively. It should be noted that the proportional relationship is observed at the elastic region for all the treatment types employed. The extent of stress and strain in each case confirmed the specific application. This is unlike other non-naturally woven fibre like coir and celery fibres with non-linear behaviour in the elastic region (Bakri and Eichhorn, 2010). Also of importance in the stress–strain behaviour of treated *Luffa* is the two-stage elastic elongation for the untreated and acetylated fibers. This is possible due to treatment effects which have modified the properties of the fibre involved and other tensile properties of *Luffa* fibres. Moreover, the mechanical performance of the natural fibres depends on their chemical structure, cellulose content, microfibrillar angle etc. The untreated and acetylated fibers could be adjudged to have decreased the microfibrillar angle and increased the cellulose content of the *Luffa* and thereby enhancing the mechanical properties of the fibre. Similar observations were made in the report of Mannan and Talukder (1997) for jute fibre.

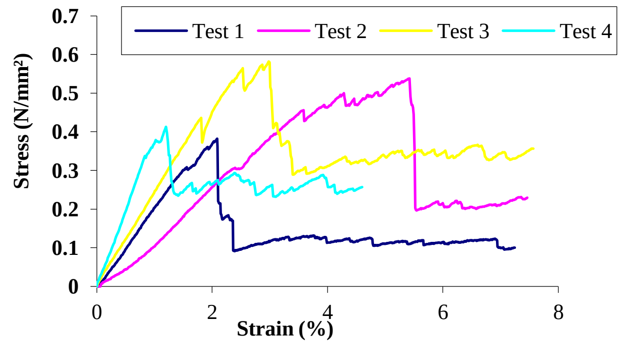

Fig. 2 Stress-strain curves for test 1- oxidation (potassium permanganate), test 2- untreated (raw), test 3- acetylation (acetic acid) and test 4- mercerisation (NaOH).

3.2 Tensile characteristics of the fibers pre and post-elastic regions

For an exhaustive analysis of the results, the tensile characteristics of pre and post-elastic regions are presented in **Table 1**. It is important to evaluate the effect of the treatment method on the transitory behaviour of the *Luffa* fibres for proper and specific applications. A sudden drop in load-carrying capacity was observed from 80.30N to 64.70N across the referenced regimes. This confirms sudden elastic deformation. Decreased load bearing with higher elongation parameters are equally observed with treatments 2 and 3 across the regimes but less suddenly; these are: 113N to 104.9 N and 122.2N to 118.50N respectively. In summary, treatments 2 and 3 produced greater ductility compared to 1, while the untreated remain stiffened. The values of strain within the elastic region also differ with the treatment types and across elastic regimes. Strain values are from 2.053% to 1.525%, 5.358% to 4.218%, 2.976% to 2.528% and 1.199% to 1.199% for treatments 1, 2, 3 and 4 respectively. Untreated fibers treatment is confirmed to have the highest strain within and outside the elastic region. This further established the initial position that the untreated *Luffa* fibre (test 2) comes out as the most ductile followed by the acetylated fibre (test 3). The oxidised (test 1) and mercerised (test 4) are at two opposing poles in properties, while the mercerised fibre retains its natural stiffness, the oxidised sample is the entire opposite and shows more flexibility. The sundry tensile properties value changes with different fibre treatments employed, and across elastic and non-elastic regions upon load applied. The decrease in the tensile modulus is most prominent with oxidation treatment across all tensile characteristics, followed by mercerisation. The tensile characteristics of the *Luffa* fibre are of considerable improvement for the untreated fiber and the acetylated fiber. The tensile modulus of the mercerised fibre increased due to zero elongation achieved beyond the elastic regime. The elongation at the break of the fibres increased considerably in the acetylated and untreated fiber by 9.07 and 8.89mm respectively.

2.3 Comparing the tensile strengths

Knowing the values of the force at peak and that the average cross-sectional area of our *Luffa* fibers is (20µm), we can obtain the strength of the fibers. The tensile strength of the untreated fibers was 7.083MPa. There was an improvement in the tensile strength with acetylation to a value of 7.541MPa. Oxidation and mercerisation led to tensile strengths of 5.11MPa and 5.517MPa respectively. Other studies have reported the tensile properties of *Luffa* fibers as 6.7MPa (Chethan *et al*., 2019), 9.4 MPa (Dharmalingam *et al*., 2020), 17.4MPa (Lai *et al*., 2016), and 68.1MPa (Siqueira *et al*., 2010). In a recent review, it was shown that these values can be widely varying due to factors such as cultivation region, plant duration, the nature of the plant, source of the fibre, location, nature of the soil and climate condition (Adeyanju *et al*., 2021). In conclusion, we see that oxidation and mercerisation do not improve the tensile properties of the fibers. Treatments such as oxidation and mercerisation are usually targeted at other further applications like composite production where the interfacial adhesion between the fibers and the resins is of utmost importance (Cruz and Fangueiro, 2016). These treatments are used to improve composite properties (Ighalo *et al*., 2020). Hence, the choice of treatment would determine the domain of application. However, in terms of tensile properties, only acetylation is shown here to improve the quality of the fibers.

Table 1 Summary of tensile characteristics of the *Luffa* fibers. Test 1- oxidation (potassium permanganate), test 2- untreated (raw), test 3- acetylation (acetic acid) and test 4- mercerisation (NaOH).

	Test 1	Test 2	Test 3	Test 4
Elong-Break (mm)	8.653	8.884	9.077	5.520
Elong-Yield (mm)	1.831	5.065	3.034	1.439
Elong-Peak (mm)	2.464	6.434	3.572	1.439
Strain-Upper Yield (%)	1.525	4.218	2.528	1.199
Strain-Peak (%)	2.053	5.358	2.976	1.199
Strain-Yield (%)	1.525	4.218	2.528	1.199
Stress-Break (N/mm^2)	0.100	0.230	0.357	0.257
Stress-Peak (N/mm^2)	0.382	0.538	0.582	0.413
Stress-Upper Yield (N/mm^2)	0.308	0.500	0.564	0.413
Force-Peak (N)	80.30	113.0	122.2	86.70
Force-Upper Yield (N)	64.70	104.9	118.5	86.70
Force-Yield (N)	64.70	104.9	118.5	86.70

Conclusions

In this investigation, we set out to examine three popular fiber treatment techniques and which would be suitable for improving the tensile properties of *Luffa* fibers. The stress-strain relationship for *Luffa* fiber is linear in the elastic region, with the untreated fiber withstanding the highest value of force in this phase. This proportional relationship is observed at the elastic region for all treatment types employed. The results showed that the untreated fibre was the most ductile, while the mercerised fibre was the stiffest. The tensile properties of the *Luffa* fibre improved significantly for the untreated and acetylated fibres. The oxidation treatment resulted in the most prominent decrease in tensile modulus, followed by mercerisation. The elongation at break increased considerably in the acetylated and untreated fibres. The values of stress and strain within and outside the elastic region differed across treatment types and elastic regimes

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