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Combined Effect of Water Retting and Sodium Hydroxide Concentration on Properties of Luffa Cylindrica Fibres

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ABSTRACT: *Luffa cylindrica is an annual climbing plant whose fruit has a vascular network of fibres. These fibres are held together by pectins which degrade during water retting to release the fibres. In this paper, the extraction of luffa cylindrica fibres together with the combined effect of water retting and alkali treatment on its tensile properties are reported. The fibres were extracted from mature fruits of Luffa cylindrica through aerobic water retting process over a duration of 2 weeks, 4 weeks and 8 weeks. The extracted fibres were oven dried to a moisture content of 9.74±1.1% and tested in tension at gauge lengths of 10 mm. The fibres were characterized in terms of moisture regain, lignin content, hemicellulose content, and cellulose content. The combined effect of water retting and sodium hydroxide (NaOH) concentration on the fibre's breaking load, elongation, tenacity and linear density was also investigated. The luffa cylindrica fibres had a moisture regain of 10.81%, lignin content of 12.03%, cellulose content of 65.69%, and hemicellulose content of 19%. The linear density of the fibres retted for 2-8 weeks was 572–470 dTex .The determined breaking load of the fibres retted for 2-8 weeks was 1444.19-417.04 cN and 997.81-298.05 cN after treatments with 0% -16% NaOH. The fibre elongation was 4.0-24% after 2-8 weeks of retting and 4.3-14.5 % after treatments with 0% -16% NaOH. Tenacity of luffa cylindrica fibres was 6.0-25.25 cN/Tex after 2-8 weeks of retting and 5.9-20.22 cN/Tex after treatments with 0% -16% NaOH. From the study, an increase in aerobic water retting and concentration of NaOH had a positive effect on the tensile properties of the luffa cylindrica fibres.*

Keywords: *Characterization, Luffa Cylindrica, Mechanical Properties, Sodium Hydroxide, Water Retting*

I. INTRODUCTION

Luffa cylindrica is an annual climbing vine, which produces a fruit (gourd) containing a fibrous vascular system (Aluyor, 2009). When separated from the skin, flesh and seeds, the fibre network can be used as a bathroom sponge (Akankwasa & Patrick, 2014). Because luffa has a compact network of close fibres, its resiliency makes it useful for many products like filters, slipper soles, baskets, packaging materials and other crafts (Marcos et al, 2012, Elemo et al, 2011). Luffa is environmentally safe, biodegradable and a renewable resource (Adie, 2013). To obtain the fibres, it is necessary to subject the gourds to a retting process that separate the fibres from the extra pectin. When subjected to Alkali treatment, cellulosic fibre properties are affected due to the removal of natural and artificial impurities (Ebisike et al, 2013). A study by Valcineide et al, 2005 concluded that the treatment of *luffa cylindrica* fibres with sodium hydroxide for a duration ranging from 10 to 90 minutes adequately modifies the fibre's surface characteristics (Valcineide et at , 2014; Valcineide et al, 2005) but they did not explore the additional effect of water retting on mechanical properties of the fibres. Similar study conducted by López–Vásquez involved the treatment of *luffa cylindrica* fibres with 0.1 M sodium hydroxide for 20 minutes in order to improve fibre hydrophilicity (López–Vásquez, 2012). Surface modification of areca and hemp fibres with sodium hydroxide has also been found to improve absorbency and mechanical properties of the resultant products (Sampathkumar, 2012; Mehta, 2005). A similar study involving the treatment of cotton with sodium hydroxide concluded that treated fabrics have better interfacial bonding properties. (Achukwu, 2015). In another study, treatment of flax fibres with 5% NaOH for one hour at room temperature resulted in improved performance of the fibres with epoxy resins (Zhu, 2015). The improvement of properties as result of sodium hydroxide treatment stems from the ability to form a charged intermediate species between Sodium Hydroxide and the fibre, which allows the faster nucleophilic addition of the matrix materials (Cicala, 2010).

Besides surface modification, caustic treatment can result in the modification of the fibre's internal structure of cellulosic fibres, causing the fibres to have more voids and rough surface and hence reduced tensile properties (Mwaikambo, 2009). Ultrasonic and microwave surface modification techniques have also been studied on *luffa cylindrica* fibres (Şahinbaşkan, 2015; Merdan, 2012). Sanna et al (2015), established that the surface modification of foam formed cellulose fibres with plasma resulted in a positive adhesion effect at 8kW (Sanna et al, 2015). However, no study explored the combined effect of water retting and Sodium Hydroxide treatment on the mechanical properties of *luffa cylindrica* fibres. This study intends to further explore the effect of water retting and alkali treatment on the mechanical properties of the luffa cylindrica fibre.

II. MATERIALS AND METHODS

2.1 Materials

The *luffa cylindrica* fibres were obtained from 1500 mature gourds. The gourds were subjected to aerobic water retting using mineral water of known composition for a duration of 2 weeks, 4 weeks and 8 weeks.

2.2 Physical and Chemical Characterization

2.2.1 Moisture Regain

It is defined as the amount of water present in a specimen expressed as a percentage of its dry mass. The fibre sample was conditioned for 24h at 22±3 ^{o}C and the weight was taken (x). The conditioned fibre sample was dried in oven at 105 ^{o}C for 4h and the weight was taken (y). Moisture regain percentage of the given sample was calculated according to the formula (Nawaz, 2002). Twelve tests were conducted and the average was taken as moisture regain from equation 1.

Moisture regain (%) $= \frac{x-y}{y} X\ 100\%$ Equation 1 *(Nawaz, 2002)*

2.2.2 Lignin Content

1g of *luffa cylindrica* fibres (Mi) was treated with 15 ml of 72% H_2SO_4 for 2 hours. The material mixture was then diluted to 3% H_2SO_4 to a volume of 575 ml with distilled water and then boiled for 4 hours. These two steps are intended to dissolve the carbohydrates leaving lignin to float in the acid as a black substance. The lignin was filtered into a pre-weighed crucible, washed with distilled water to remove any remaining acid, dried to a constant weight (Mf) and weighed to determine the amount of lignin is determined as a percentage of the original Mass of material (Klason Procedure) (Ronald & Romualdo, 2005; Fagerstedt, 2015). Seven tests were conducted and the average was taken to give the lignin content from equation 2.

Lignin% $= \frac{Mi}{Mf} X\ 100$ Equation 2 (*Fagerstedt, 2015)*

2.2.3 Cellulose Content

The cellulose determination was made according to the Kurschner and Hoffer method: 1g of *luffa cylindrica fibres* (M1) were treated with 100 ml of a 1:4 (v/v 72% nitric acid: 96% ethanol) mixture (Brauns, 2013), and then raised to boiling for 1 hour. Nitric acid hydrolyses, oxidises and dissolves lignin and hemicellulose while cellulose is protected by ethanol. (Chen, 2015) The solution was filtered and the insoluble residue was retreated twice again using the previous process. After 3 treatments, the solid was washed with distilled water and hot ethanol to neutral. The solid residue was oven dried at 105 ^{o}C to constant weight (M2) to determine the percentage of cellulose (Ouensanga, 1989; Bremer, 2013). Seven tests were conducted and the average was taken as cellulose content from equation 3.

Cellulose content% $= \frac{M2}{M1} X\ 100$ Equation 3 *(Bremer, 2013)*

2.2.4 Hemicellulose Content

1g of sample (W1) was weighted and 24% KOH was added to the sample and allowed to stand for 2hours. The mixtures were filtered and washed with additional 24% KOH solution and the filtrate was further precipitated by the addition of ethanol. The precipitated hemicellulose was filtered with Whitman filter paper and the residue was washed with ethanol before oven drying to constant mass at 105^{o}C. After the treatment, the hemicellulose was transferred into desiccators and allowed to cool for 30minutes after which the weight was taken (W2) (Cox & Webster, 1960; Oladele, 2010). Seven tests were conducted and the average was taken as the hemicellulose content from equation 4.

Hemicellulose % $= \frac{W2}{W1} X\ 100$ Equation 4 *(Oladele, 2010)*

2.3 Alkali Treatment

Treatment with Sodium Hydroxide at concentrations from 0% (w/v) to 16% (w/v) was conducted with the help of the temperature generated in-situ for a duration of 45 minutes after which the fibres were neutralized with mild acetic acid and rinsed thoroughly with distilled water.

2.4 Mechanical Testing

Tensile tests on the fibres were conducted on a Universal Tensile Tester machine in accordance with ISO 5079:1995(E) standard. The tests were performed in displacement controlled mode at a constant rate of 100 mm/min crosshead speed using a gauge lengths of 20 mm. The load and the extension (difference between final and initial lengths) at the point of fibre rupture were recorded as the breaking load and breaking extension, respectively. Linear density was determined using the gravimetric method described by ISO 1973:1995 (E) by determining the total weight and total length of a bundle of 100 fibres. The tenacity was computed by dividing the breaking load with the linear density of the unstrained fiber. The tests were conducted at 65%RH and 21°C.

III. RESULTS AND DISCUSSION

3.1 Physical and Chemical Composition of the *Luffa Cylindrica* Fibres

Table 1 shows the results of the chemical composition of ***luffa cylindrica* fibres**. It is clear the fibres contains larger proportion of cellulose and hemicellulose indicating their potential as textile fibres. The results are comparable with previous studies on ***luffa cylindrica* fibres** [Parida 2013, Macuja 2015, Laidini 2012). Typically, bark and leaf materials have higher cellulose content in the range of 50-70% which is consistent with the current results.

Table 1: Chemical composition of luffa cylindrica fibres

Composition	Values
Cellulose	65.69%±3.77%.
Hemicellulose	19%±3%.
Lignin	12.03%± 2.34%.
Moisture regain	10.81% ±1.34%.
Moisture content	9.74%±1.1%.

3.2 Effect of Water Retting and Caustic Treatment on Breaking Load

Figure 1 shows the combined effect of Sodium Hydroxide and water retting on the breaking load of *luffa cylindrica* fibres

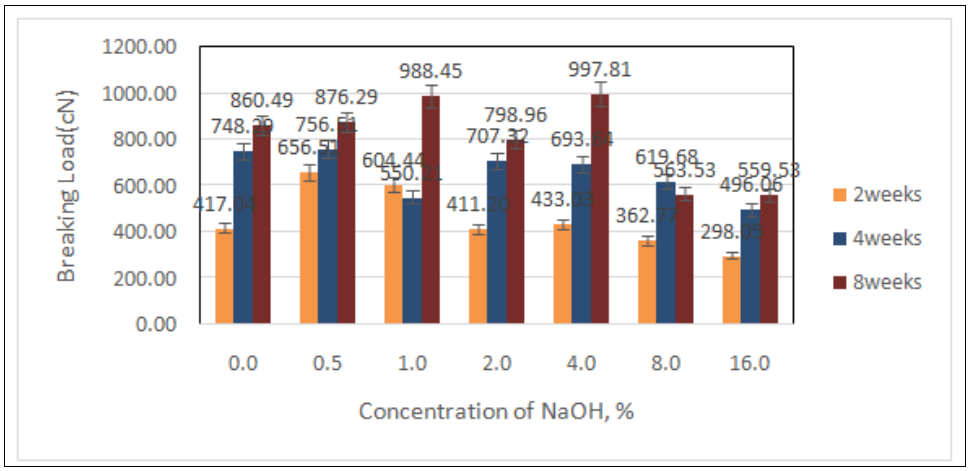

Figure 1: Effect of Sodium Hydroxide and water retting on the breaking load of *luffa cylindrica* fibres

It is evident from the results that the fibre strength exhibited similar trends in three sections i.e. between NaOH concentrations of 0-0.5%, 1.0-4.0% and 8-16%. The initial increase in fibre strength can be attributed to the increase in packing density and molecular orientation as the cementing materials are removed by sodium hydroxide (Siregar et al, 2010). However, further increase of sodium hydroxide concentration from 8% to 16% resulted in the extra drop in strength which can be associated with the hydrolysis of some cellulose components (Hashim et al, 2013) of the *luffa cylindrica* fibres. Considering a fibre at 8 weeks water retting duration and initial strength at 0% NaOH as control level, the effect of NaOH treatment resulted in 2% increase in breaking load at 0.5% NaOH, 15% increase at 1.0%NaOH, 7% reduction at 2%NaOH, 16% increase at

4%NaOH, 35% reduction at 8%NaOH and 16% NaOH. These results were similar to those obtained by Paschal (2015) in the study to establish the effect of Sodium Hydroxide treatment on the tensile properties of *luffa cylindrica* composites. A regression analysis using Minitab 17 software for the maximisation of breaking load showed that the optimal duration of water retting was 8 weeks and concentration of sodium hydroxide of 0%. This implies that the breaking load had a positive dependence on water retting and a negative dependence on concentrations of caustic treatment as shown in the linear regression model equation 5. The relation between combined effect of water retting and caustic treatment on breaking load shown in equation 5 was statistically significant with a p-value less than 0.1 and R^2 Value of 0.63.

$B_L = 578.6 + 62.49W - 24.22C$ equation 5.

Where B_L – breaking load, W – weeks of water retting, and C – percentage concentration of NaOH.

3.3 Effect Of Water Retting And Caustic Treatment On Elongation

Figure 2 shows the combined Effect of Sodium Hydroxide and water retting on the percentage elongation of *luffa cylindrica* fibres

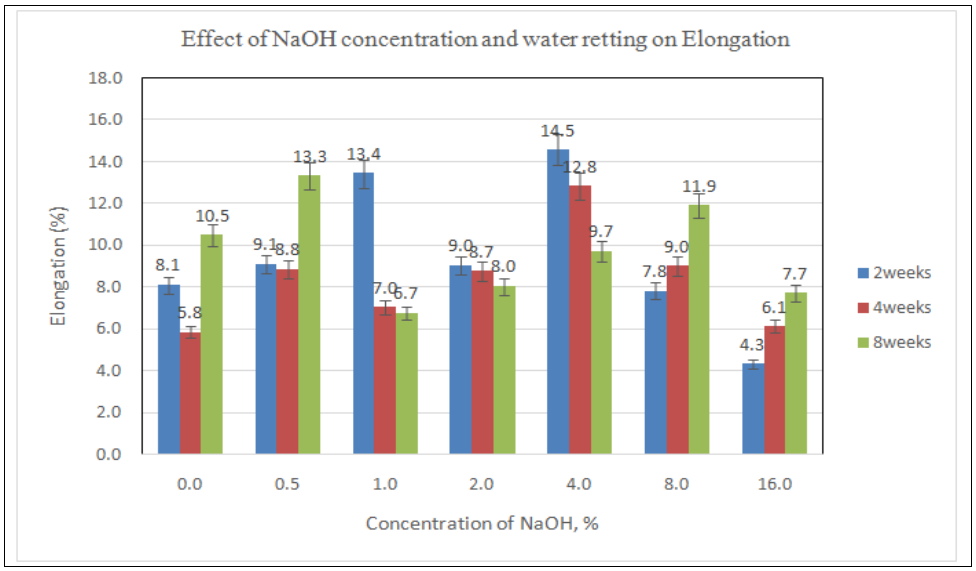

Figure 2: Effect of Sodium Hydroxide and water retting on the percentage elongation of *luffa cylindrica* fibres

For fibres retted for 8 weeks, their percentage elongation increases with concentration of Sodium Hydroxide up to 8% when it drastically drops at 16% Sodium Hydroxide. From 0%NaOH to 0.5%NaOH the increment was 37%, 1% NaOH resulted in a decrease of 19% of the original control value at 0%NaOH. At 2%NaOH, the elongation reduced by 21% before increasing again at 4%NaOH by 27%. Further increase of Sodium Hydroxide concentration at 8% resulted in a decrease of 30% and a further decrease of 10% at 16%NaOH. Therefore, a treatment with 0.5% Sodium Hydroxide is sufficient for higher observable elongation while a treatment at 8% Sodium Hydroxide showed the highest percentage decrease in elongation. The regression analysis of the combined effect of water retting and caustic treatment for maximisation of percentage elongation as shown in equation 6 was not statistically significant with a p-value of 0.258 and R^2 value of 0.044.

$E = 10.53 + 0.272\ C - 0.0279\ C^2$ equation 6.

Where E – percentage elongation, C – percentage concentration of NaOH

3.4 Effect of Water Retting and Caustic Treatment on Linear Density

Figure 3 shows the combined Effect of Sodium Hydroxide and water retting on the linear density of *luffa cylindrica* fibres

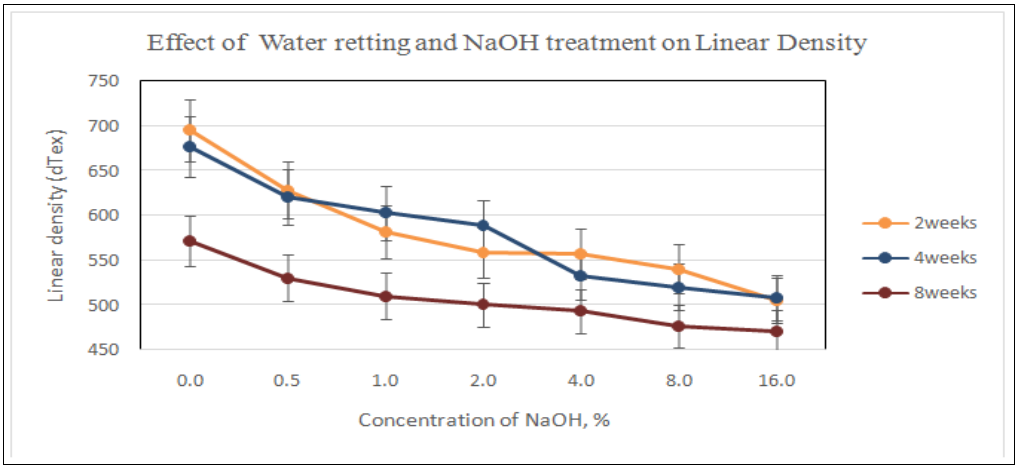

Figure 3: Effect of Sodium Hydroxide and water retting on the linear density of *luffa cylindrica* fibres

There was a general reduction in linear density with the increase in duration of water retting weeks and increase in Sodium Hydroxide concentration as shown in Figure 3. This can be attributed to the fact that at 8 weeks of water retting, the fibres are more eroded by water and hence cleaner with less surface impurities that resulted in a decrease in both diameter and linear density than those retted for 2 to 4 weeks for the same alkali treatment condition (Hashim M, 2013). The results also implies that the linear density of *luffa cylindrica* fibres are highly dependent on the level of surface erosion treatments. The apparent similarity in linear density irrespective of retting period for fibres treated with alkali concentration above 4% may be attributed to uniformity in fibre surface modification by the strong alkali solution. A regression analysis using Minitab 17 software for the minimisation of linear density showed that the optimal duration of water retting was 8 weeks and concentration of sodium hydroxide of 10.5%. This implies that the linear density of luffa cylindrica fibres was dependent on both water retting than concentrations of caustic treatment with a quadratic relationship shown in equation 7. The relation between combined effect of water retting and caustic treatment on linear density shown in equation 7 was a statistically significant quadratic regression model with a p-value less than 0.1 and R. sq. value of 0.85.

$$L_d = 626.1 + 13.57W - 24.85C - 2.888W^2 + 0.927C^2 + 0.678W*C \qquad \text{equation 7}$$

Where L_d – linear density, W – weeks of water retting, and C – percentage concentration of NaOH.

3.5 Effect of Water Retting and Caustic Treatment on Tenacity

Figure 3 shows the combined effect of Sodium Hydroxide and water retting on the tenacity of *luffa cylindrica* fibres.

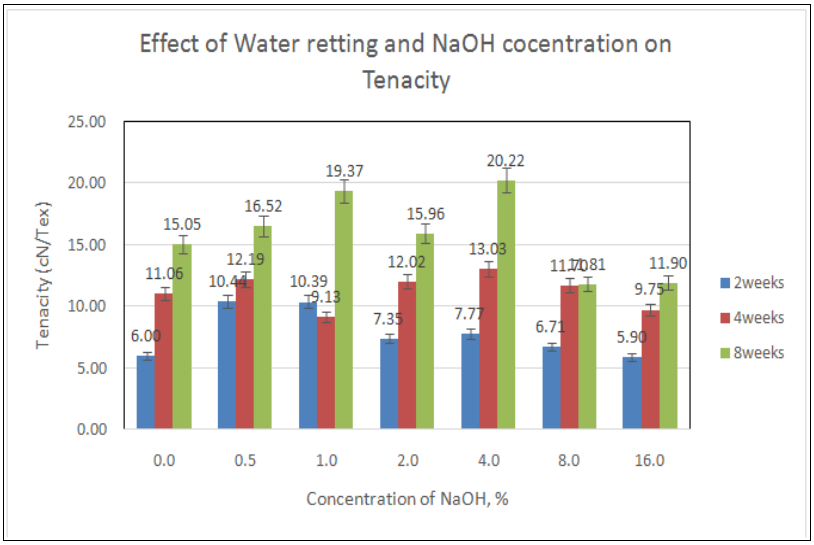

Figure 4: Effect of Sodium Hydroxide and water retting on the tenacity of *luffa cylindrica* fibres

Considering retting of 4 weeks, the tenacity appears to be less sensitive to Sodium Hydroxide treatment (0% to 16%) with a loss of 46% as compared to 48% at 2 weeks and 53% at 8 weeks. A regression analysis using Minitab 17 software for the maximisation of tenacity showed that the optimal duration of water retting was 8 weeks and concentration of sodium hydroxide of 0%. This implies that the tenacity of luffa cylindrica fibres had a positive dependence on water retting and a negative dependence on concentrations of caustic treatment as shown by a linear relationship in equation 8. The relation between combined effect of water retting and caustic treatment on tenacity shown in Equation 8 was a statistically significant linear regression model with a p-value less than 0.1 and R^2 value of 0.79

$$T = 7.275 + 1.689W - 0.096C - 0.0430W*C \quad \text{Equation 8}$$

Where T – tenacity, W – weeks of water retting, and C – percentage concentration of NaOH.

IV. CONCLUSION

The combined effect of water retting and sodium hydroxide concentration on properties of luffa cylindrica fibres revealed that:-

- The tensile strength of luffa cylindrica fibres was highest at 1% NaOH after 8 weeks of retting. It was also observed that 8 weeks of retting produced better performance in tensile strength which can be attributed to improved packing density and molecular orientation of the fibres.
- The elongation at break of luffa cylindrica fibres was maximum at 2 weeks of water retting and 4% concentration of NaOH. This can be due to internal breakdown of some binders like lignin and hemicellulose thereby allowing the fibres to extend more as compared to other pre-treatments.
- The linear density of luffa cylindrica fibres showed a consistent decline. This can be associated with the more removal of surface impurities and improved packing density as the treatments increased in strength.
- A concentration of 4% NaOH at 8 weeks of water retting resulted in the highest tenacity. And overall, 8 weeks of water retting produced the highest tenacity at all levels of caustic treatment.

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