

EFFECT OF VARIATION IN UREA CONCENTRATION USED IN RETTING, ON THE PHYSICO- CHEMICAL AND MECHANICAL PROPERTIES OF KENAF FIBRES.

Abstract

AIMS: (i) To compare the effect of 1% and 2% urea concentration on the proximate & chemical compositions (alpha-cellulose ,lignin and hemi cellulose content) and physico-mechanical properties of kenaf fibre

(ii).To evaluate the effect of variation in urea concentration on the enzyme and microbial activities during fibre retting.

STUDY DESIGN: Data obtained from each treatment were subjected to analysis of variance (ANOVA) using Duncan multiple range test. Significant difference was accepted at $P < 0.05$.

PLACE AND DURATION: The retting experiment was carried out in the Institute of Agricultural Research and Training, Obafemi Awolowo University. From April, 2015 to January, 2016.

METHODOLOGY: Two kenaf stems of each of the four varieties were tied (bundled) together was replicated three times and soaked in an improvised retting tank containing urea solutions at 1% and 2% concentrations and control (without urea). Fibres retted in 1%, 2% urea and control (without treatment) were subjected to proximate, microbiological, chemical and physico-mechanical analyses.

RESULTS: The results showed slight increase in alpha-cellulose and lignin content of kenaf fibres across the four varieties. Hemi-cellulose content of kenaf fibres retted in 1% urea was significantly lower than others. The protein and iron content of the kenaf fibres were highest in 2% urea followed by 1% urea and least in control. The tensile strength of 2% urea retted fibres was significantly higher than others.

CONCLUSION: From the results, It can be inferred that 2% urea had produced fibres with promising physico-chemical and mechanical properties.

Keywords: alpha-cellulose, hemi-cellulose, Kenaf fibre, lignin, tensile strength

1. INTRODUCTION:

Presently, natural fibres form an interesting option for the most widely applied fibre in the composite technology, pulp and paper industries, and other ligno-cellulose conversion technologies[1-2]. Traditionally, kenaf bast fibres were usually ret in streams, rivers and ponds, and this practice was environmentally

unfriendly as it deteriorates the water body and predisposes the users to pathogenic micro-organisms. Recent research had proved that chemical retting using mild reagents like sodium hydroxide, potassium hydroxide or urea is a good substitute for whole stalk stream retting [3]. Kenaf (*Hibiscus cannabinus* L.) is a natural fibre crop used for rope, twine and coarse sacking materials. Kenaf is a multi-purpose biodegradable and environmentally friendly crop and has been found to be an important source of raw materials for other industrial applications [1].

The importance of treatments in raw plant materials goes a long way to determine the quality of the finished products. It was observed that the majority of variation in burst and tensile strength in hardwood pulp sheets could be accounted for by fiber length and cell wall thickness [4-5]. Updegraff *et al* [6] has categorically stated the significance of fibre dimensions in predicting wood pulp mechanical properties. However, there is little or few documentation on the effect variation in concentration of chemical reagents (e.g. urea) used in retting on the properties of kenaf fibre. Hence, the objectives of this study were to:

- i. Compare the effect of 1% and 2% urea concentration on the alpha-cellulose content and hemi cellulose content of kenaf fibre
- ii. Compare the effect 1% and 2% urea concentration on the lignin and tensile strength of kenaf fibre.
- iii. Assess the effect 1% and 2% urea concentration on the proximate compositions of kenaf fibre
- iv. Evaluate the effect of variation in urea concentration on the enzyme and microbial activities during retting

2.0: MATERIALS AND METHODS:

2.1: Kenaf samples preparation

The four varieties of Kenaf namely: Cuba-108, Tianung-2, Ifeken-Di-400 and Ifeken 400 were collected from the Kenaf and Jute Improvement Programme, Institute of Agricultural Research and Training, Ibadan, Nigeria .

Traditional whole stalk retting was carried out using water and urea. Two kenaf stems of each of the four varieties were tied (bundled) together was replicated three times and soaked in an improvised retting tank containing urea solutions at 1% and 2% concentrations and control (without urea). Each treatment was replicated three times. Textures of the fibres were tested on daily basis to ascertain exact time taken to attain complete retting. Each completed retted fibre was removed, washed thoroughly with water and sundried.

2.2: Measurement procedure

The fibre samples were analysed for α -cellulose, hemi-cellulose and acid insoluble lignin; α -cellulose was determined using a colorimetric method with the anthrone reagent. 0.3g (dry weight) ground (0.5mm) samples were treated and boiled (at 100°C) with a mixture of nitric/acetic acid (1:8, v/v) for 1h to remove lignin and

hemicelluloses after successive centrifugations, and diluted with 67% H_2SO_4 (v/v). Cellulose was then determined at 620nm using cold anthrone reagent [7]. Protein, ash and iron content of the fibres were determined according to the procedure described by AOAC [8].

2.3: Mechanical properties of the fibres

Mechanical testing of the fibres was carried out using fibre testing machine model-Instron 3369. This was according to the standard of American Society for testing and materials, ASTM D [9]. Tensile strength and tensile (Young's) modulus were calculated from this test.

2.4: Organic tests

The properties of retting liquor were assessed at two days interval. And subsequently the organic tests for biochemical oxygen demand (BOD), chemical oxygen demand (COD) and dissolved oxygen (DO) were conducted to determine of the concentration (in mg/L) of carbon-based Compounds aimed at establishing the relative strength of retting water [2].

2.5: Microbiological Procedure

2ml of retting water (liquor) were taken from each treatment at day 1, 3, 5 and 8 using syringe. Aliquot of each liquor was serially diluted in 10 folds. The highest of the three dilutions were considered for microbial count analysis. Each liquor was inoculated on nutrient agar and potato dextrose agar at 37°C and 25°C respectively. They were observed for 3 to 5 days and the different colonies isolated were identified using slides culture technique [8].

2.6: Statistical analysis

Data obtained from each treatment were subjected to analysis of variance (ANOVA) using Duncan multiple range test. Significant difference was accepted at $P < 0.05$

3.0: RESULTS AND DISCUSSION

3.1 Alpha –cellulose content

The alpha-cellulose content of kenaf fibres for the four varieties ranged from 59.91-60.41% for Control; 60.38-61.30% for 1% urea and 61.58-62.17% for 2% urea (Figure 1). Urea treatment significantly improved the alpha-cellulose content of the fibres across the four kenaf varieties (Figure. 1). Alpha-cellulose content of the fibres increased gradually with an increase in concentration of the urea solution from 1% urea to 2% urea. (Fig 1). Cellulose fibre with high tensile strength can lead to composite with high specific properties due to their low densities. The urea treatment retains cellulose and removes hemi-cellulose from the kenaf fibres. The values for alpha cellulose content in urea treatments were far above those (40.2- 43.8%) reported by Ververis *et al* [10]. Tianug-2 retted in 2% urea had the highest α -cellulose content followed by Ifeken 400 of the same treatment. The alpha-cellulose content in this range makes kenaf a suitable raw material for the paper and pulp industry. Plant materials with cellulose content of 40% and above have been characterized as promising for pulp and paper production [5 ;11]

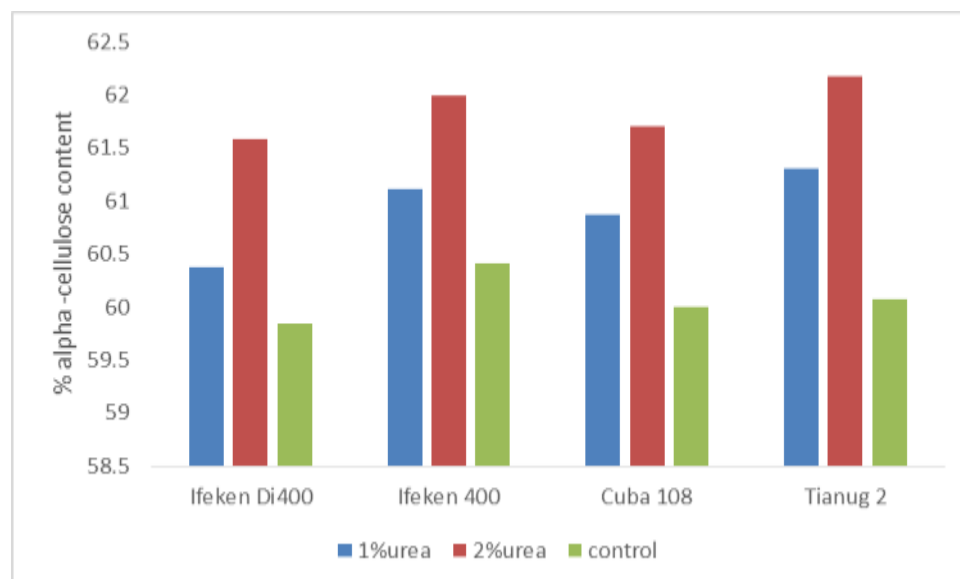


Figure 1: Alpha-cellulose content of retted fibre varieties.

3.2:Lignin content

Urea treatments had slightly increased the lignin content in the retted kenaf fibres when compared with control but the percentage range of lignin in all the treatments was significantly lower than the one reported for softwoods and hardwoods [10].Cuba-108 fibre retted in 2% urea had the highest lignin content followed by Tianug-2 of the same treatment.

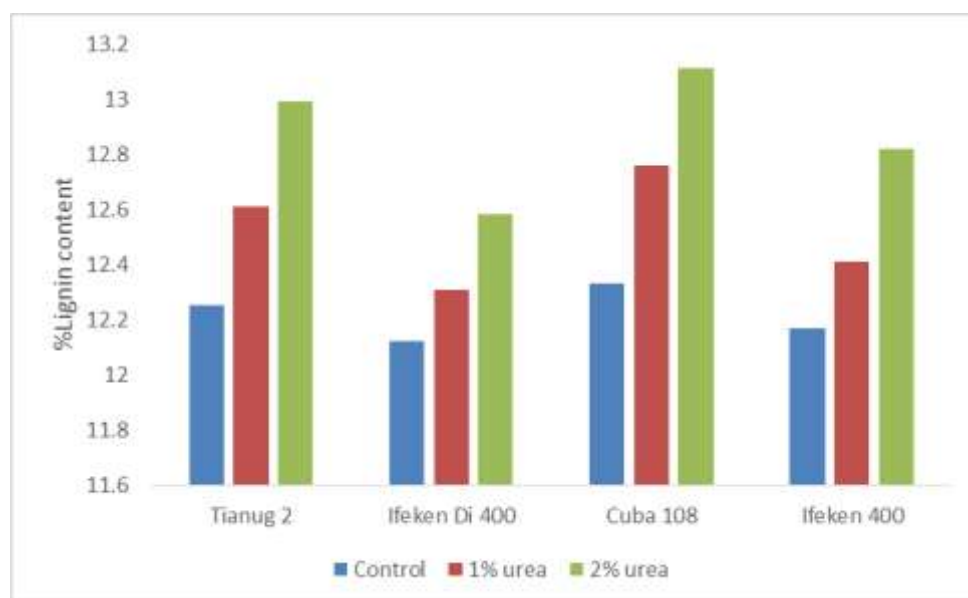


Figure 2: Percentage lignin content of retted fibre varieties.

3.3:Hemi-cellulose content

There was a significant reduction of hemi-cellulose content of kenaf fibres retted in 1% urea solution (Fig. 3). Ifeken Di-400 fibre retted in 1% urea had the least hemi-cellulose content.

The reduced amount of hemicellulose will minimize the undesirable water absorption capacity of natural fibre reinforced composites [11].

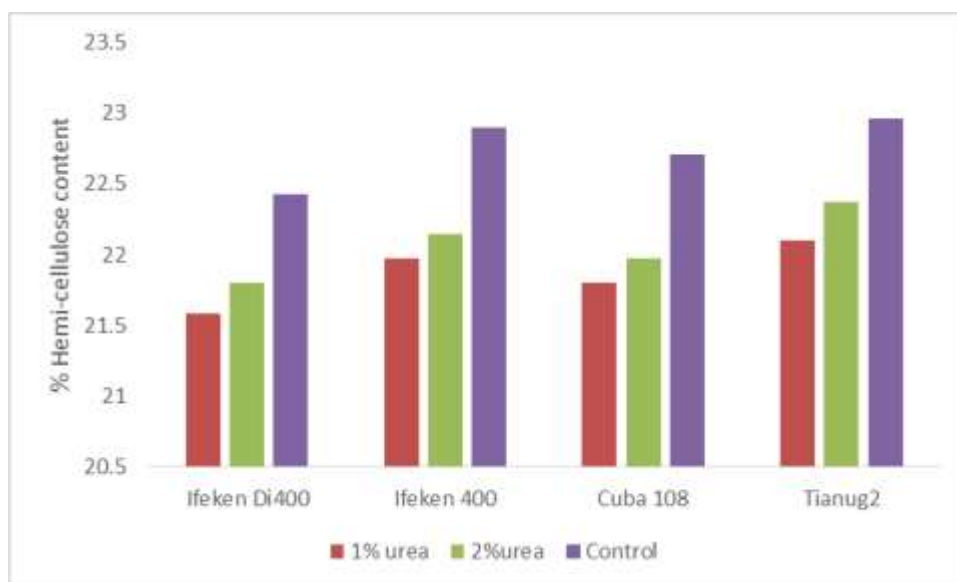


Figure 3: Hemi-cellulose content of retted fibre varieties.

3.4: Proximate composition of the retted fibres

The ash contents of all the treatments was generally low, this quality is good because higher ash content is undesirable and it can affect the pulp mechanical properties. 2% urea retted fibres had the highest ash content (0.8- 1.05%) followed by 1% urea (0.71-0.96%) and least in control (0.61-0.85%), this may be due to the presence of some metallic salts like iron, calcium, etc. (figure 4). Ifeken 400 fibre retted in 2% urea had the highest (1.05%) ash content while Tianug-2 fibre of control had the least (0.61%). The values recorded in this work were significantly lower than 3.6-4.1% ash content of the part of kenaf stalk reported by Ververis et al[10]. Urea treatments had slightly increased the percentage protein content of all the fibre varieties except the Tianug-2 fibre with 1.91% protein for control, 1.43% protein for 2% urea and 1.2% protein for 1% urea. This was in agreement the report by Neto *et al* [11-12]. The protein content of the kenaf fibres was highest in 2% urea followed by 1% urea and least in control (figure 4).

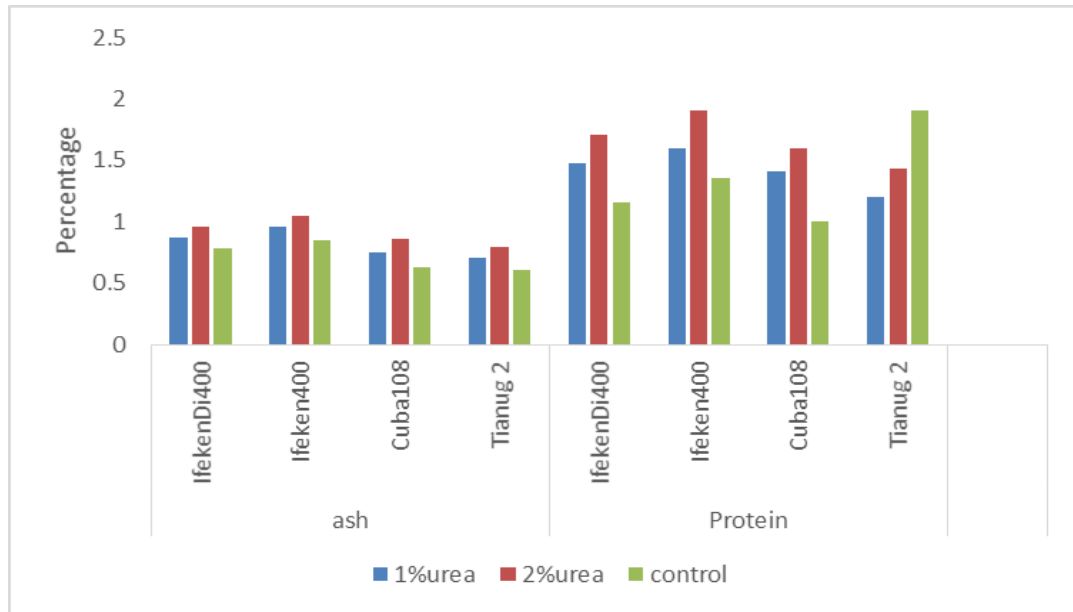


Figure 4: Percentage ash and protein content of retted fibres.

3.5: Mineral contents of retted fibres

The presence of inorganic substance like iron, carbonates, calcium, and potassium had significant effect on the ash content of kenaf fibres. Urea treatments had significantly increased the level of iron in all the varieties of kenaf fibres. Ifeken 400 fibre retted in 2% urea had the highest (95.35mg/kg) iron content followed by Ifeken Di-400 (94.00mg/kg) of 2% urea and least in Tianug-2 (86.80mg/kg) of control (figure 5).

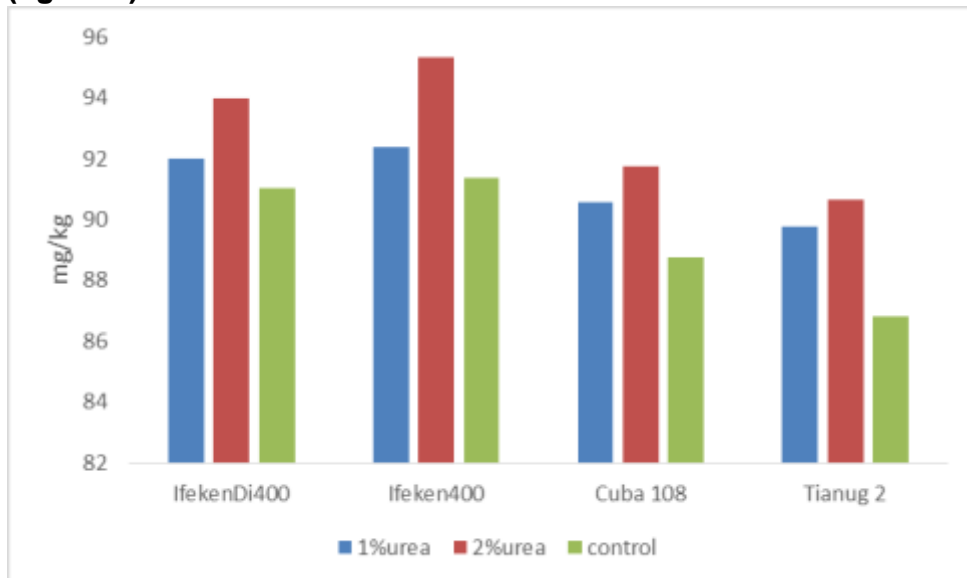


Figure 5: Iron content of retted fibres.

3.6: Tensile strength of retted fibres

The strength and durability of kenaf composites are a function of the physical and mechanical properties of its fibres. The tensile strength of 2% urea retted fibres was significantly higher than that of 1% urea and control for all the tested varieties of kenaf fibres (figure 6)

The increased tensile strength of 2% urea retted fibres may be linked with the proportionate increase in its alpha-cellulose content. Ifeken 400 fibre had remarkable tensile strengths in control and 1% urea except for 2% urea treatment where it was significantly lower than others. The data obtained in this findings strongly agrees with the report by Mohd *et al.* [4] that the tensile strength and modulus of kenaf composites increase when the concentration of sodium hydroxide solution (NaOH) was increased,

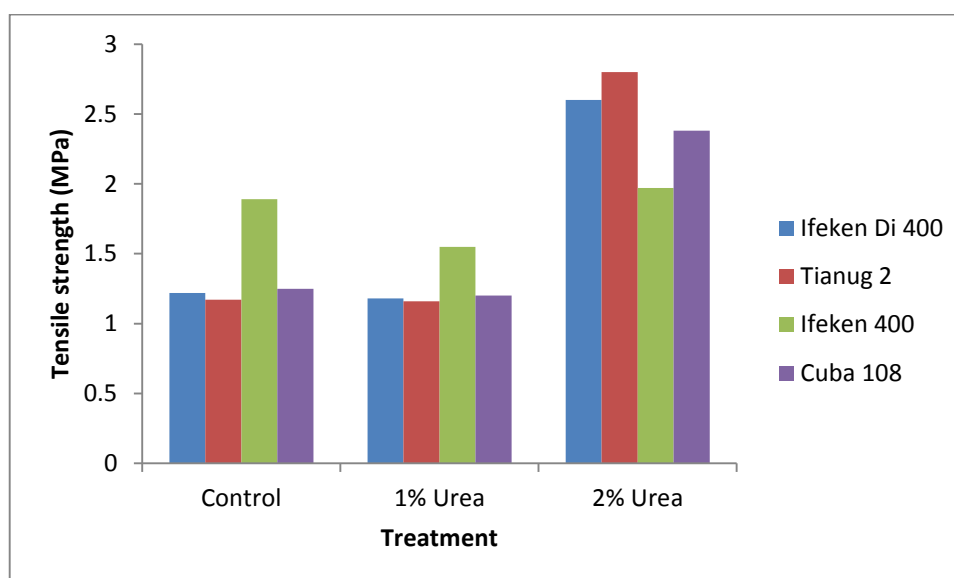


Figure 6: Tensile strength of the retted fibre varieties.

3.7: Enzyme activities in the liquor during fibre retting

There was partial removal of non-fibrous materials like hemi-cellulose and lignin which would make kenaf fibre considerably softer for fibre spinning. Lignase and hemi-cellulase enzymes allowed the selective removal of lignin and hemi-cellulose respectively without affecting the strength of the cellulosic fibre itself. The cellulase enzyme was significantly more active in 2% urea at day 1, 3 and 5 compared with 1% urea and control. The activities of the lignase and hemi-cellulase enzymes during retting were significantly higher in urea treatments than control for the first five days after which there was a decline (figure 7). The action of these enzymes during fibre retting does not show any damage on the tenacity of the fibres.

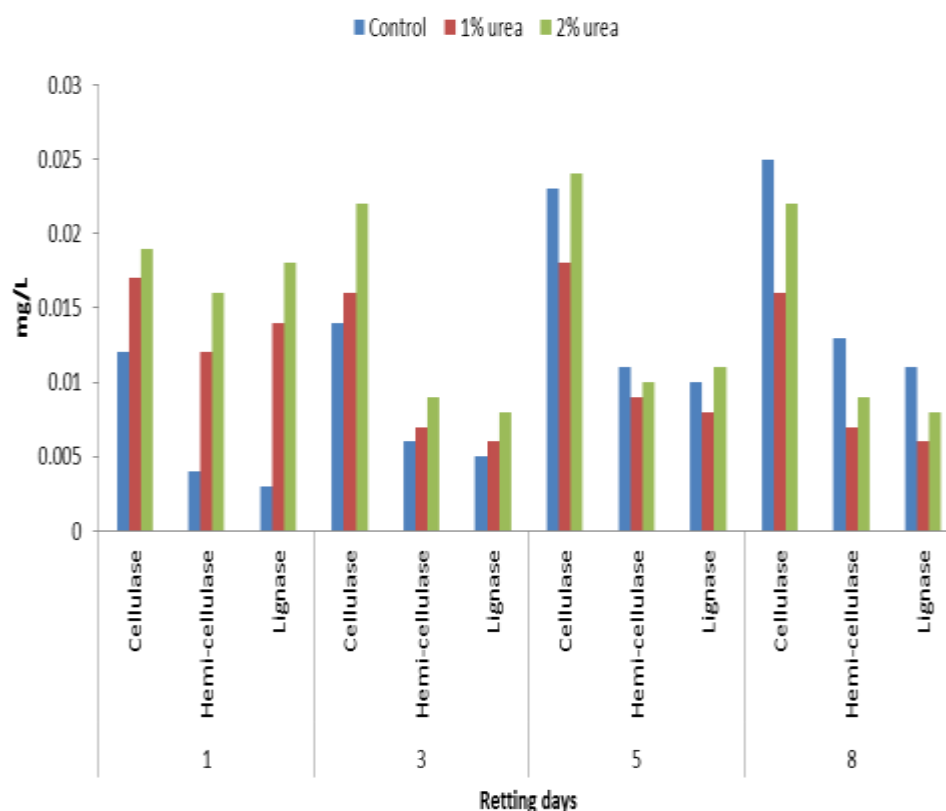


Figure 7: Enzyme activities at different retting days.

3.8: Physico-chemical parameters of water before, during and after retting *Acidity /alkalinity of the retting media*

In all the treatments, the pre-retting pH of the liquor (6.80 for control, 4.80 for 1% urea and 4.51 for 2% urea) was significantly higher than those recorded during and after retting (from 4.8 - 4.05 for 1% urea; 4.51- 3.80 for 2% urea and 6.80 - 6.50 for control). The decrease in pH value led to more acidity of retting water (table 1).The pH values of all the urea treated liquors were significantly lower than that of control.

The lowering of pH values during retting may be due to the release of organic acids and other gummy materials into the retting liquors.

Table 1: The pH of retting water (liquor) at different days.

Trt	Day 1	Day 3	Day 5	Day 8
Control	6.80a	6.65a	6.50a	6.50a
1% urea	4.80b	4.3b	4.53b	4.05b
2% urea	4.51c	4.43c	4.25c	3.80c

Means with the same letter in a column are not significantly different at $P < 0.05$, Trt = treatment.

3.9: The degree of hardness (carbonate salts) of the retting water

Total hardness of the retting water increased significantly in all the urea treatments (38.41-46.65mg/L for 1% urea and 45.60 - 47.75mg/L for 2% urea) with the corresponding increase in retting period. Conversely, control had a decreasing trend of total hardness with the increase in retting time. Hardness is caused by the bicarbonate salts of calcium and magnesium.

Table 2: The total hardness of retting water at different days (mg/L).

Trt	Day 1	Day 3	Day 5	Day 8
Control	36.65c	36.50c	35.50c	35.25c
1% urea	38.41b	43.65b	44.65b	46.55b
2% urea	45.60a	45.60a	46.80a	47.75a

Means with the same letter in a column are not significantly different at $P < 0.05$

3.9.2: Degradation of water retting

The level of water deterioration was revealed by the amount of dissolved oxygen (DO) in each liquor.

The liquor from 1% and 2% urea had significantly higher DO than control at day 1, 3 and 5 but at day 8, control had higher DO than the treated liquors (figure 8a).

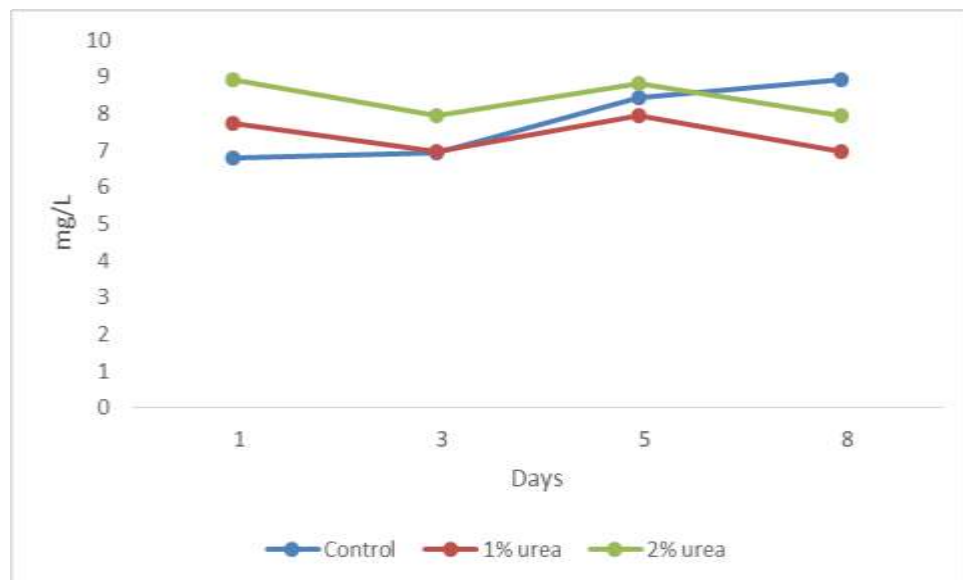


Figure 8a: Dissolved oxygen (DO) at different retting days.

Biochemical oxygen demand of the treated liquors were significantly higher than that of control at day 1,3 and 5 (figure 8b).

A high BOD indicates a high content of easily degradable, organic material in the liquor, while a low BOD indicates a low volume of organic materials, substances which are difficult to break down

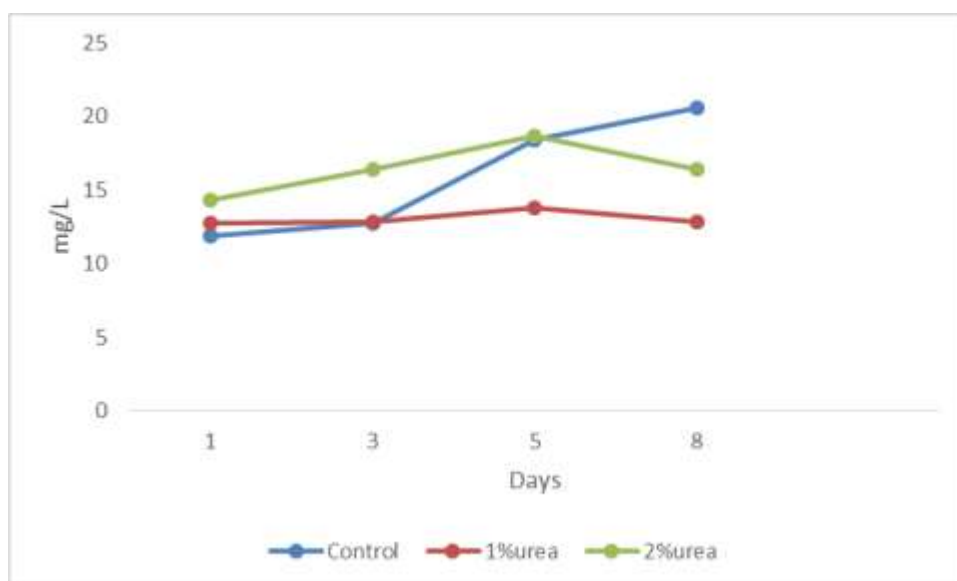


Figure 8b: Biochemical oxygen demand at different retting days

The chemical oxygen demand (COD) of the pre-retting liquors were 37.95mg/L for 1% urea, 41.33mg/L for 2% urea and 32.62mg/L for control. These values increased progressively with the retting in all the treatments (figure 8c). Urea treatments had higher amount of COD than control at the first three days of retting. There was rapid increase in COD of control at day 5 and 8. Chemical oxygen demand (COD) shows the oxygen equivalent of the organic matter content of each liquor that is prone to oxidation which can lead to water deterioration.

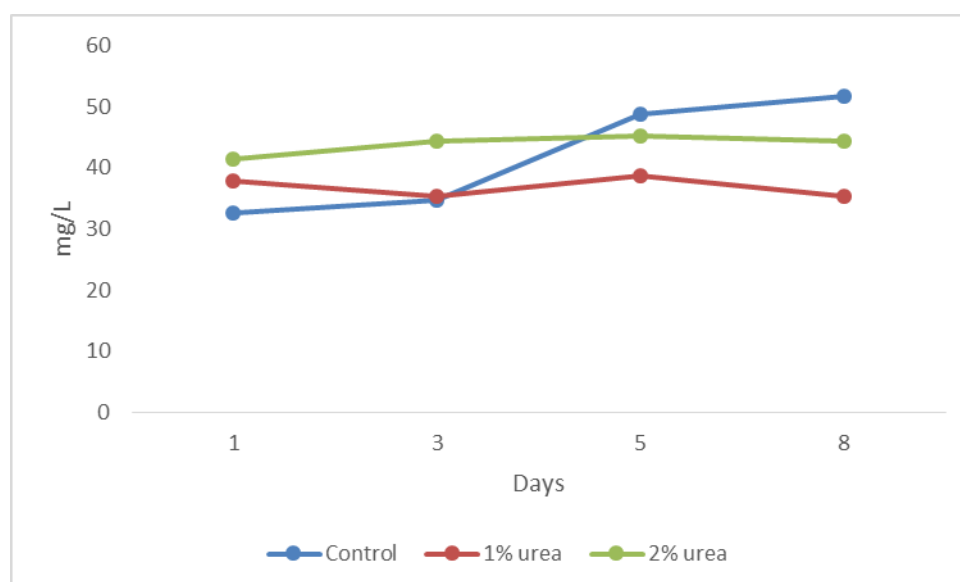


Figure 8c: Chemical oxygen demand (COD) at different retting days.

Table 3: Microbial counts at different interval (Cfuml⁻¹).

Day 1	Control	1% urea	2% urea
Total bacteria count	3.1x10^{6c}	3.85x10^{6b}	4.25x10^{6a}
Total coliform count	0.3x10^{6c}	0.55x10^{6b}	0.8x10^{6a}
<i>E.coli</i>	0.2x10^{6b}	0.4x10^{6a}	0.4x10^{6a}
<i>S. aureus</i> count	0.45x10^{6c}	0.6x10^{6b}	1.2x10^{6a}
Total fungi count	0.2x10^{6b}	0.2x10^{6b}	0.25x10^{6a}
Day 3			
Total bacteria count	3.55x10^{6c}	4.35x10^{6b}	4.6x10^{6a}
Total coliform count	0.5x10^{6c}	0.7x10^{6b}	1.05x10^{6a}
<i>E.coli</i>	0.35x10^{6c}	0.5x10^{6b}	0.65 x 10^{6a}
<i>S. aureus</i>	0.65x10^{6c}	0.75x10^{6b}	1.6x10^{6a}
Total fungi count	0.35x10^{6c}	0.4x10^{6b}	0.5x10^{6a}
Day 5			
Total bacteria count	3.75x10^{6c}	5.0x10^{6b}	5.1x10^{6a}
Total coliform	0.7x10^{6c}	0.95x10^{6b}	1.0x10^{6a}
<i>E. coli</i>	0.5x10^{6c}	0.7x10^{6b}	0.75x10^{6a}
<i>S. aureus</i>	0.8x10^{6c}	1.0x10^{6b}	1.15x10^{6a}
Total fungi count	0.6x10^{6c}	0.7x10^{6b}	0.8x10^{6a}
Day 8			
Total bacteria	3.55x10^{6c}	4.6x10^{6b}	6.65x10^{6a}
Total coliform	0.5x10^{6c}	0.7x10^{6b}	1.7x10^{6a}
<i>E. coli</i>	0.35x10^{6c}	0.5x10^{6b}	1.1x10^{6a}
<i>S. aureus</i>	0.65x10^{6c}	0.75x10^{6b}	1.35x10^{6a}
Total fungi count	0.4x10^{6c}	0.5x10^{6b}	0.95x10^{6a}

Means with the same superscripts in a row are not significantly different (P< 0.05).Cfuml⁻¹ represents colony forming unit per millilitre.

3.9.2:Microbial activities

The activities of some microbes during retting were on the increase and their presence had hasten the retting process thereby reducing its duration. The result in table 3 showed that the highest microbial load was recorded at day 5.This range

of values were not significantly different from those obtained at day 8 for both urea treatments. It implies therefore that 1% and 2% urea treatments induced the similar microbial load. It is pertinent to note that pathogenic microbes (*Aspergillus* spp, *Staphylococcus* spp and *Escherichia* spp) emanated during retting and this calls for high level of environmental sanitation and proper sewage (effluent) disposal or management. In line with Bello *et al* [13], washing of Kenaf fibre with clean water of potable quality could reduce the microbial load. Proper sensitization on the involvement of pathogenic bacteria in Kenaf retting should be carried out and adequate precaution must be taken while carrying out this farm-gate processing to prevent spread of the pathogenic micro-organisms to Kenaf farmers.

4.0: Conclusion

The synergistic enzymatic action with urea solution had produced fibres with promising physico-chemical and mechanical properties. Urea treatments produced fibres with satisfactory levels (< 30%) of lignin and (> 40%) of alpha-cellulose content. The quality (length, fineness) of the fibres retted in urea treatments was considerably higher than control. Urea retting at 2% concentration had better tensile strength, improved alpha-cellulose content and shorter retting days. Complete and effective retting lasted for 7-8 days for all the urea treatments while control lasted for 14 days.

References

- [1] Kaldor, FA. The logistics and feasibility of utilizing kenaf fibers for commercial scale processing in existing chemical pulp mills. In: TAPPI Proceedings, NA Nonwood Fiber Symposium, Atlanta, GA, USA, 1998, pp. 161–173.
- [2] Chow, P.; Lambert, RJ; Bowers, C.T. ; McKenzie, N. Physical and mechanical properties of composite panels made from kenaf plant fibers and plastics, The 2000 International Kenaf Symposium, Hiroshima.
- [3] Omenna,EC.,Adeniyi,ON., Ejigbo, EA,Oduwaye,OF.Ezaka ,E. Comparative effect of chemical and steam retting on Kenaf fibre quality. *Agric. Biol. J. N. Am.*, 2016, 7(5): 275-283.

- [4] Mohd Yuhazri, Y., Phongsakorn, P.T., Haeryip Sihombing, Jeefferie A.R., Puvanasvaran Perumal, Kamarul, A.M., and Kannan Rassiah, 2011. Mechanical Properties of Kenaf/Polyster Composites. *International Journal of Engineering & Technology IJET-IJENS*. 2011, Vol: 11. Pp 106 -110.
- [5] Shatalov, AA; Pereira, H. Influence of stem morphology on pulp and paper properties of *Arundo donax* L. reed. *Ind. Crops Prod*. 2002, 15, 77–83.
- [6] Updegraff, DM. Semi-micro determination of cellulose in biological materials. *Anal. Biochem*. 1969, 32, 420–424.
- [7] Aguiar, CL, 2001. Biodegradation of the cellulose from sugarcane bagasse by fungal cellulase. *Cienc. Tecnol. Aliment*. 3 (2), 117–121.
- [8] Association of Official Analytical Chemists, Official Methods of Analysis (18th Edn.). International, Maryland, USA. 2005, 2341p.
- [9] Standard of American Society for testing and materials .New York. 2010.
- [10] Ververis, C.; Georghiou, K.; Christodourakis, N.; Santas, P.; Santas, R. Fiber dimension, lignin and cellulose content of various plant materials and suitability for paper production. *ELSEVIER, Int'l J.* 2004, 245-254.
- [11] Neto, P, Fradinho, D, Coimbra, MA; Domingues, F; Evtuguin, D; Silvestre, A., Cavaleiro, JAS., Chemical composition and structural features of the macromolecular components of *Hibiscus cannabinus* L. grown in Portugal. *Ind. Crops Prod*. 1996, 5, 189–196
- [12] Neto, P, Nunes, A, Coimbra, MA, Domingues, F, Evtuguin, D.; Silvestre, A.; Cavaleiro, J.A.S. Variations in chemical composition and structure of macromolecular components in different morphological regions of *Arundo donax* L. *Ind. Crops Prod*. 1997, 6, 51–58.
- [13] Bello, OB, OLawuyi, OJ; Adebisi, OS; Azeez, AH, Oladimeji, DT. Genetic pathogenic microbes associated with deterioration of sweet orange (*Citrus sinensis* L) fruit in Nigeria. *Sci. Agric* 2016, 13, 19-22.