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RESEARCH ARTICLE

EFFECT OF ESTERIFICATION USING *JATROPHA CURCAS* SEED OILS ON SOME PHYSICAL AND MECHANICAL PROPERTIES OF CELLULOSIC FABRIC

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ABSTRACT

For many decades cellulose fabrics has been subjected to different chemical treatments in order to improve resistance against shrinkage, creasing, flexibility, tensile strength and absorbency. Such chemicals include urea and melamine formaldehyde resins. These chemicals are expensive and pose danger to the environment. Polycarboxylic and nitric acids have also been used on cellulose base fabrics for easy care finish. Recently ionic cross linking, polyurethane with nanomaterials as catalyst or co-catalysts has been considered. In order to go more environmentally green and less expensive, this paper presents the results of the physical and mechanical properties of cellulosic fabric esterified with 50cm³ of oils extracted from the seed of *Jatropha curcas*. The oil was extracted with hexane under reflux with yields of 47.25% and moisture of 0.56%. The fabric was subjected to pretreatments of scouring, bleaching and mercerization to remove impurities and to enhance fibre consolidation before esterification. The results showed that yarn crimp was 31% warp direction while the grey fabric (control) gave the lowest crimp value (5%) along the same direction. The linear density (46 Tex) along warp direction was recorded for *Jatropha* esterified fabrics compared to 37 Tex for the grey fabric. The fabric sett increased from 24th/cm for grey fabric to 34th/cm for *Jatropha* fabric along warp direction. The reduction in shrinkage was obvious after esterification using the oil. The tensile parameters were remarkable after esterification 280.78N and 170.40N with extension of 18.07mm and 15.88mm along warp and weft directions respectively for the esterified fabric. That of the grey fabric was 223.87N and 109.39N with extensions of 3.64mm and 3.56mm in warp and weft directions respectively. There was a remarkable improvement in the dry and wet crease recovery angles after esterification (128° dry and 86° wet) along warp direction. The grey fabric gave the lowest crease recovery (50° dry and 37° wet) in the same direction. The observed short fall in strength of the cellulose ester may be attributed to the formation of ester bond. However the general improvement in the investigated properties due to dimensional stability, flexibility and fineness is commendable. This research has contributed immensely to knowledge because this is the first time that biodegradable organic seed oil like *Jatropha curcas* is used to modify the physical and mechanical properties of cellulosic fabric through esterification. Therefore, the oil is recommended for replacement of the present day toxic chemicals used in textile finishing of cellulosic fabrics.

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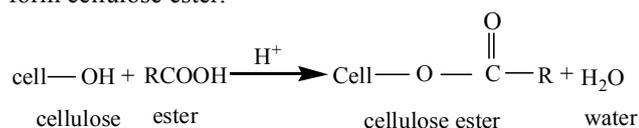
INTRODUCTION

Chemical finishing such as scouring, bleaching, mercerization and resination of cellulosic fabric with the aim of improving the physical and mechanical properties has been established. In recent years, application of urea and melamine formaldehyde in particular in the form of precondensate on cellulosic fabric has been the focus. However, there has been some noticeable problems with the amino precondensate such as the toxicity to man, environmental degradation, formation of yellow

discoloration on cellulosic fabric during storage, the release of hydrochloric acid during ironing leading to loss of strength in such fabric (Timar-Balazsy and Eastop, 2011). These resins are equally very expensive therefore is believed to be the reason for the high cost of the finished products. The reason for structural modification of cellulosic fabric is based on the inherent propensity of the fabric to crease or deform during processing or in use Ajayi et al. (2005). Structurally, cellulose consists of both amorphous and crystalline regions and is the basis of all natural and man-made fibres which constitute cellulose fabrics

(Morton and Hearle, 1975; Billmeyer, 1984; Harder, 2004; Pandey *et al.*, 2015). The inherent strength of cellulosic fabric is attributed to the presence of the crystalline region where as noticeable flaws like dimensional instability and creasing are attributed to the amorphous region (Klemm *et al.*, 2005; Carraher, 2010). This is why cellulose has been chemically modified in order to establish orientation of molecules in the amorphous region, improve the intermolecular attraction and enhance the fibre modulus (Morton and Hearle, 1975).

Recently, attention is focused on green chemistry because of the need to improve on the environment. It is possible to employ green chemistry to modify the structure of cellulose (Omizegba *et al.*, 2017). Hence, this research is focused on esterification of cellulose with free fatty acid from vegetable seed oil to form cellulose ester and investigation of its effect on the physical and mechanical properties of the cellulosic fabric. Esters are chemical compounds consisting of a carbonyl adjacent to an ether linkage derived by reaction of an oxoacid with a hydroxyl compound such as alcohol or phenol (Nic *et al.*, 2006). Carboxylic acids whether organic or inorganic react with alcohols to form esters (Niak *et al.*, 2006). The formation of natural esters cross-linkages in cellulosic materials during drying and heating is of interest to give cotton materials improved characteristics. Conversion of cellulose to its esters affords materials that are processible into various useful forms and solutions to be used for coating and casting of films and membranes (Tosh, 2011). Carboxylic acid such as butane tetracarboxylic acid or citric acid has been used to improve both the durable press of cotton and the performance of paper. Interestingly, Pantze (2006) stated that several carboxylic acids can be tested for their ability to form ester linkage with hydroxyl groups in cellulose. The choice of *Jatropha curcas* is based on the low utilization in this part of the world. They are mostly discarded as waste in bushes and surrounding mountains. Where the trees are planted in homes; the aim is to provide shades and to keep grazing animals away. A few research to utilize the oils for biodiesel production is ongoing (Ibanga *et al.*, 2004; Gutti *et al.*, 2011). The seeds contained about 47.25% oil, 0.65% moisture Omizegba *et al.* (2017) *Jatropha curcas* is a drought resistant perennial plant growing well in marginally poor soil and lives producing seeds for 50years (Centre for *Jatropha* Promotion (CJP) and Biodiesel (2013). The oil of the plant is biodegradable and have many medicinal importance (Achetan *et al.*, 2008; Wilson *et al.*, 2009; Khalid *et al.*, 2010). This research investigated the effect of *Jatropha curcas* seed oil by esterification on crease recovery, tensile property, linear density, fabric sett, yarn crimp and shrinkage of cellulosic material after pretreatments of scouring bleaching and mercerization. Equation (1) represents the reaction between the acids in the oil and hydroxyl in cellulose to form cellulose ester.



Where, R is the triglyceride chain.

MATERIALS AND METHODS

Materials: The following materials were employed in this study; Oven (memmert 854 Schwabach and Gallenkamp size one Bs)

- Weighing Balance (Digital) (Sauter RC 8021)

- Analytical Balance (Model NJ07932 florham Park U.S. A.)
- Heating Mantle (Clifton)
- Shirley Crease Recovery Tester (Model No. 308)
- Viscometer (Prolabo No. 4)
- Hook's Travelling Microscope (Serial No. 901879)
- Tensile Strength Tester (Model No. RS-232)
- Shirley Crease Loading Device (Model No. 308)
- Stop Clock (Raffin), Hook's Travelling Microscope (Serial No, 101879),

Sodium hydroxide, acetic acid, sodium silicate, magnesium sulphate, hydrogen peroxide, sulphuric acid, sodium carbonate, methanol. All chemicals were analytical grades supplied by BDH Chemicals Ltd. Poole, England.

METHODS

Extraction of oils: Extraction of the oils was according to Pearson (1991). The fruits were decoated, soaked in water for about 6 hours to dissolve the sticky pulp then sun dried. The seeds were removed from the hard shells through cracking. The kernels obtained were air dried and then ground to fine powder ready for extraction. The ground seeds (50.0 g) was placed in a pre-weighed thimble and then placed in the barrel of the Soxhlet Apparatus. Hexane (200 ml) was poured into the flask and the apparatus set for extraction and allowed to run for 6 hours.

Percentage yield: Ground sample (50 g) was placed into the pre-weighed empty thimble (w_1). Weight of sample plus that of thimble was recorded (w_2). The thimble was removed after extraction and dried in an oven to a constant weight (w_3). The percentage yield was calculated using;

$$\text{Percentage yield} = \frac{w_2 - w_3}{w_2 - w_1} \times 100$$

Moisture content: A 3 g of oil sample weighed into an empty crucible (w_1), so that the weight of the crucible and oil sample were recorded as (w_2). The crucible and its content were then placed in an oven at 105°C for 4 hours after which it was removed, cooled in a dessicator and reweighed (w_3). The process of heating and cooling was repeated until a constant weight was obtained (Pearson,1991). Percentage of moisture content is calculated as:

$$\text{Percentage moisture content} = \frac{W_2 - W_3}{W_2 - W_1} \times 100$$

Purification of fabric: Standard method based on the (British Standard Handbook 11 (1974) and American Society for Testing Materials ASTM (1994 American Society for Testing Materials ASTM D6774 2010) was employed.

Scouring of grey fabric: 10 cm × 10 cm of the grey fabric was immersed in 2% NaOH solution and boiled for 1 hour. It was rinsed severally in overflowing water followed by washing in detergent solution, after which it was neutralized with 5% acetic acid then rinsed with water and dried at room temperature.

Bleaching of scoured fabric: The scoured sample was boiled for 45 minutes in a bleaching liquor containing 5% of H₂O₂, 0.1 g NaSiO₃, 10 ml of 1% NaOH solution and 0.5 g MgSO₄, then

rinsed severally in tap water for 10 minutes and neutralized with 5% acetic acid and dried at room temperature.

Mergerization of bleached fabric: The bleached fabric was immersed in 20% solution of NaOH at 5°C with occasional turning with a glass rod for 20 minutes, after which it was washed in detergent solution for 10 minutes, rinsed with tap water for 5 minutes, neutralized with 5% acetic acid, rinsed with distilled water and dried at room temperature.

Esterification of mercerized fabric: Methanol (100 ml) and the oil (50 ml), respectively were mixed; 0.5 ml of concentrated H₂SO₄ was added and refluxed for 1 hour at 60°C. The mercerized sample was weighed (1 g) and then immersed into the flask and refluxed for 3 hours at 60°C with occasional shaking. The fabric was removed and neutralized in 2% solution of Na₂CO₃ in order to destroy any acid residue that remained in the sample, while the residual oil was removed by immersing the fabric in a very dilute detergent solution. The sample was rinsed in distilled water and dried in the oven at 60°C for 20 minutes then weighed again.

Determination of physical and mechanical properties: Standard methods according to BS11 (1974) was employed.

Yarn crimp: The original length of the sample was taken as L_o, and the unraveled yarn was subsequently straightened to remove the wavy curves on the thread. The length of the straightened yarn recorded as L_c. the entire procedure was carried out in both weft and warp directions for the grey, scoured, bleached, mercerized and esterified fabrics and the yarn crimp calculated using;

$$\text{Yarn Crimp \%} = \frac{L_c - L_o}{L_o} \times 100$$

Yarn linear density (Tex): The mean weight of 10 threads was taken in g for both weft and warp directions. The length of the sample is 10 cm × 10 threads which gives 1 m. The Yarn Linear Density is calculated using;

$$\text{Yarn Linear Density(Tex)} = \frac{\text{Weight in gram}}{\text{Length in meter}} \times 1000$$

This procedure was carried out for the grey, scoured, bleached, mercerized and esterified samples.

Yarn sett or thread count: The Hook's Travelling Microscope instrument (Serial No. 901879) was used to determine the threads per cm of the fabric samples. This was done for the grey, scoured, bleached, mercerized and esterified samples.

Fabric shrinkage: The dimension of the fabric was measured for weft and warp directions as L_o. After scouring, bleaching, mercerizing and esterifying, the new dimensions was measured in each case for both weft and warp directions as L_s.

The fabric shrinkage is determined using;

$$\text{Fabric Shrinkage\%} = \frac{L_o - L_s}{L_o} \times 100$$

Standard methods according to BS11 (1974) was employed.

Fabric Tensile Strength: The sample dimension 10 cm × 5 cm was mounted on the Tensile Strength Tester. The gauge length of the Tester adjusted to accommodate the sample then the

instrument operated at the speed of 350 mm/min and the breaking load/extension characteristics of the sample recorded automatically while the load cell was 5000 N. This was carried out on the grey, mercerized and esterified samples in both weft and warp directions.

Crease Recovery

Dry Crease Recovery: The Shirley Crease Recovery Tester was calibrated by adjusting the knobs to face 0° mark. The fabric sample was cut using the template dimensions along the warp and weft directions for grey, scoured, bleached, mercerized and esterified fabrics. The samples were folded end to end and placed on the Shirley loading device for 5 minutes. The load was removed and the samples allowed recovering for another 5 minutes, after which they were transferred to the Crease Recovery Tester to measure the angle of crease recovery.

Wet crease recovery: The samples were immersed in water and the excess water on the samples drained with filter paper without pressing. The test procedure was repeated as in dry crease recovery angle measurement.

RESULTS AND DISCUSSION

Percentage yield of oil: After extraction with hexane, the percentage yield of the oil was 47.25% (Omizegba *et al.*, 2017). The oil has neither taste nor odour but is light yellow in colour. It remained liquid at room temperature which is indication that the oil sample contained predominantly unsaturated acids of low molecular weight (Gubitz *et al.*, 1999). Therefore, the oil is suitable for cellulose esterification because of easy penetration into the fabric.

Moisture content: The seed of *Jatropha curcas* contained 0.65% moisture and this value is above 0.55% moisture content for edible oil as recommended by ASTM (2010), this implies that the oil is a non-edible oil and will not compete for food. Therefore it will be solely useful industrially.

Fabric Purification

Scouring of grey fabric: The scouring process allowed hydrolysis of the fatty and waxy substances, degradation of protein and simpler nitrogenous compounds into water soluble salts, conversion of pectins and lignins into soluble salts while mechanically adhering dirt are loosened and held in suspension as stated by Omizegba *et al.* (2017). Hence the solution changed from colourless before the treatment to a very dark yellow-brown solution after the treatment. The fabric becomes softer, more absorbent and cleaner.

Bleaching of scoured fabric: The whiteness of the bleached material was very significant. The bleaching action of the perhydroxyl ion leads to the removal of colouring matter as stated by Timar-Balazsy and Eastop, 2011. The stabilizing action of the sodium silicate in the liquor also enhanced the bleaching process.

Mergerization of bleached fabric: The effects of mergerization are evident in the physical appearance of the mercerized fabric. There was a clear difference between the mercerized and the unmercerized fabrics due to the formation of alkali cellulose or cellulose alcoholate which appeared lustrous, shrank longitudinally, swelled laterally, more absorbent and creased very badly Timar-Balazsy and Eastop, 2011; Gutti *et al.*, 2011.

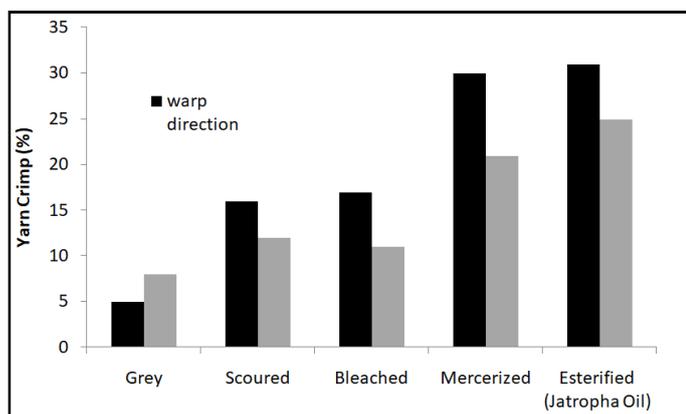


Figure 1. Effect of treatments on yarn crimp (Warp and Weft directions)

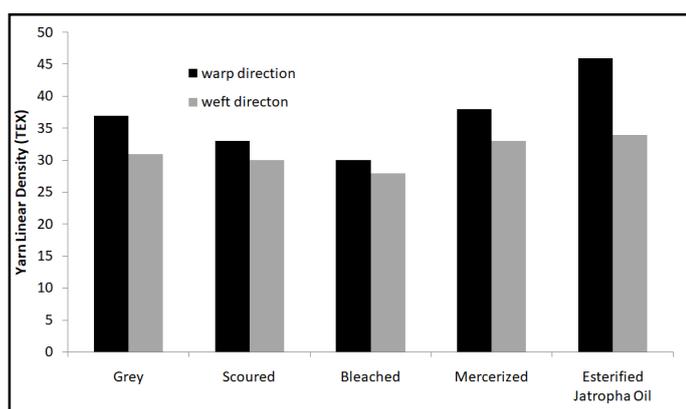


Figure 2. Effect of Treatments on yarn linear density (Warp and Weft Directions)

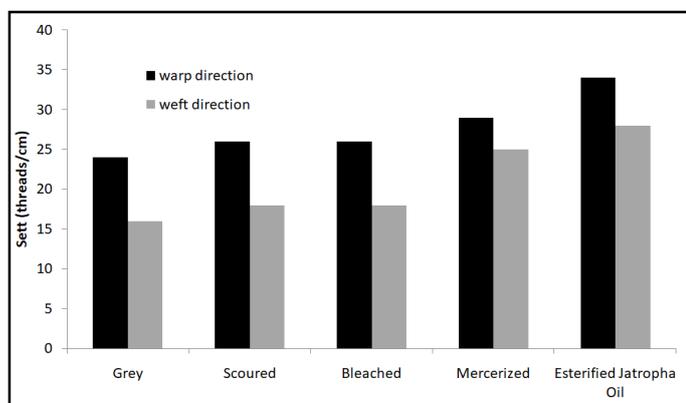


Figure 3. Effect of treatments on fabric sett (Warp and Weft Directions)

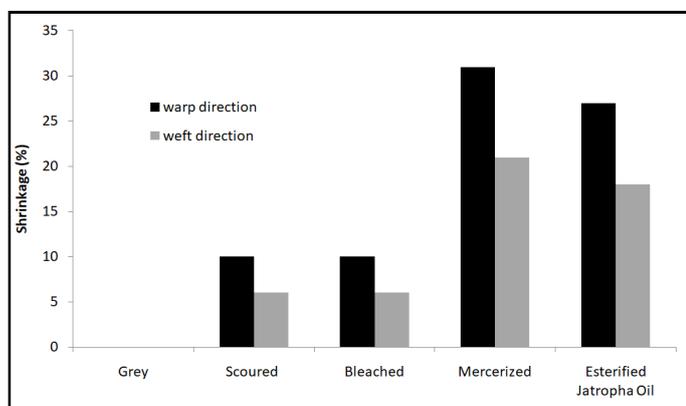


Figure 4. Effect of treatments on fabric shrinkage (Warp and Weft Directions)

Esterification of mercerized fabrics: Esterification of the fabric with 50ml of *Jatropha curcas* seed oil revealed that there was a

reaction between the OH of cellulose and the COOH of the oil to give a new cellulose monoester with structural modification in defractometer angle, inter-atomic distance, peak intensity, peak width, crystallite size, percentage crystallinity and water imbibition. This was revealed by the x-ray diffraction analysis (Omizegba *et al.*, 2017). The esterified fabric recorded increase in weight and is less hydrophilic due to the presence of a more bulky and hydrophobic ester group in the cellulose chain (Omizegba *et al.*, 2017). This structural modification might be responsible for the improvements in the properties investigated in this research.

Effect of treatments on yarn crimp: Yarn crimp is the waviness of a yarn owing to interlacing in the fabric during yarn construction (Francis, 2008). It plays an important role in yarn extensibility, compressibility fabric extensibility and improves smoothness, fullness and softness (Arthur, 2009; Francis, 2008). The percentage yarn crimp along warp and weft directions for grey, (Control), Scoured, bleached, mercerized *Jatropha curcas* esterified samples are 5, 16, 17, 30, 31 and 8, 12, 11, 21, 25 for warp and weft direction respectively as outlined in Fig. 1. It can be seen that the grey fabric had the lowest crimp suggesting that the fabric will have the lowest dimensional stability and very poor drapping quality. Crimp was significantly enhanced by the different treatments. However, esterification with *Jatropha curcas* has mostly improved crimp more along the warp direction. This suggest that *Jatropha curcas* oil was able to improve the crimp property of the fabric hence, the esterified fabric may have a better spinning performance due to better dimensional stability.

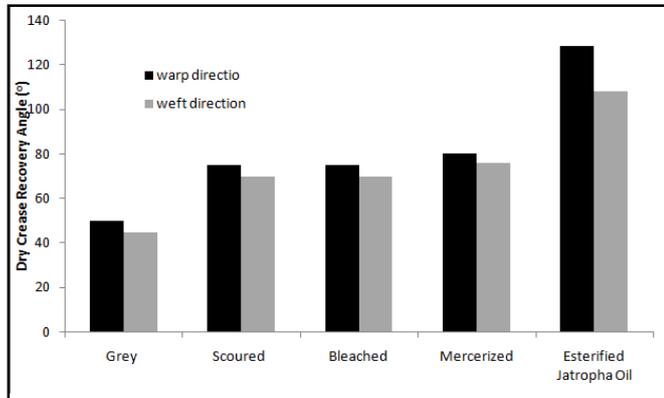
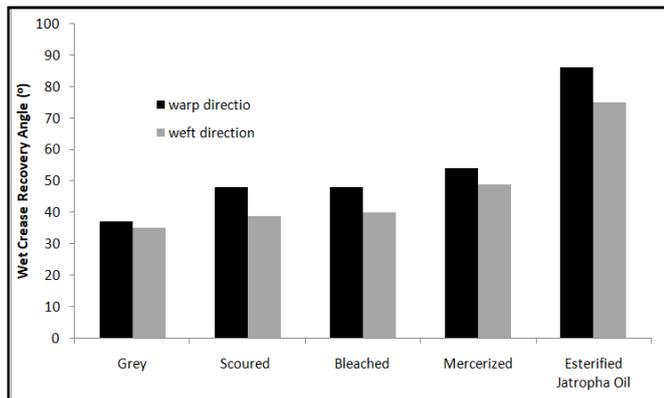
Effect of treatments on linear density: Linear density (Tex) is the weight in grams per unit length of 1000m of yarns (Morton and Hearle, 1975; Ajayi *et al.*, 2005; Boryo, 1999; Bradow, 2000). It is a parameter for assessing the fineness or coarseness of a textile fabric. The yarn linear density of grey, scoured, bleached, mercerized, *Jatropha* esterified fabrics are 37, 33, 30, 38, 46 along warp direction and 31, 30, 28, 33 and 34 along weft direction, respectively as depicted in Fig. 2. The esterified fabric gave the highest tex value. This may be attributed to fibre consolidation due to increase in weight of fabric after esterification, suggesting the presence of a more bulky -O-CO- group in the cellulose chain (Omizegba *et al.*, 2017). Linear density is lower in scoured and bleached fabrics due to removal of waxes, pectins and coloring matter during the purification. Hence the esterified fabrics appeared finer and smoother to handle.

Effects of treatment on fabric sett: Sett or thread count reveals the number of treads/cm (Omizegba *et al.*, 2015). In weaving clothes, the warp is the sett of length wise yarns while the yarn that is inserted over and under the warp is the weft (Gordon, 2006). The fabric sett for grey, scoured, bleached, mercerized, *Jatropha* oil esterified sample is 24, 26, 26, 29, 34 for warp direction and 16, 18, 18, 25, 28 for weft direction respectively as outlined in Fig. 3. It was observed that fabric sett increased as the grey material was subjected to the various treatments especially along the warp direction.

Jatropha oil esterified fabric recorded the highest sett. According to Fashola and Alonge (2002), the higher the fabric sett, the more the waviness (crimp) of the fabric; hence improvement in the elastic properties and flexibility of the fabric. It implies that the esterified fabric is more flexible and elastic (Fashola and Alonge, 2002).

Table 1. Effect of Treatment on Breaking Load/Extension of Fabrics

Sample	Warp		Weft	
	Load (N)	Extension (mm)	Load (N)	Extension (mm)
Grey	223.87	3.64	109.39	3.56
Mercerized	282.88	20.20	176.10	17.74
Esterified (Jatropha oil)	280.78	18.07	170.40	15.88

**Figure 5. Effect of treatments on dry crease recovery angle (Warp and Weft Directions)****Figure 6. Effect of treatments on wet crease recovery angle (Warp and Weft Directions)**

This may be attributed to the exceptional flexibility and free rotation about the $-O-CO-$ ester linkage (Bello, 2001) in the cellulose formed (Omizegba *et al.*, 2017). It is an indication that *Jatropha curcas* is acting as a lubricating agent contrary to resination with urea and melamine formaldehyde which resulted to a stiffer and coarse fabric (Omizegba *et al.*, 2016). Again the esterified fabric is finer, smoother and is expected to have a better crease recovery.

Effect of treatments on fabric shrinkage: Shrinkage is the contraction in the dimensions of fabrics as a result of chemical interaction with the cellulose molecules or moisture absorption that could result to creasing (Morton and Hearle, 1975). The result of the fabric shrinkage in percent is represented in Fig. 4. The values for grey, scoured, bleached, mercerized and Jatropha oil esterified samples are 0, 10, 10, 31, 27 along warp direction and 0, 6, 6, 21, 18, along weft direction respectively. The grey fabric recorded 0 shrinkage because it did not undergo any treatment. Generally, there is no difference in physical and mechanical properties of scoured and bleached fabrics apart from colour change; any difference is very minimal. This may be because scouring and bleaching are aimed at removal of impurities and to impart a pure and permanent white effect (Cai, 2000). The mercerized fabric had the highest percentage shrinkage in the warp and weft directions. This may be due to

the effect of the 20% concentration of sodium hydroxide used for mercerization leading to the formation of alkali cellulose having a better fibre alignment and fabric consolidation (Ajayi *et al.*, 2005; Omizegba *et al.*, 2015). Economically, shrinkage in fabric is sometimes not desired; hence it was minimized by esterification probably because the alkali cellulose was replaced by the formation of cellulose ester which has a better dimensional stability (Deguchi *et al.*, 2006; Pantze, 2006).

Effect of treatment on fabric strength parameters: Tensile strength is the property of a fibre, defined as the ability to resist stress and it has a direct influence upon the strength of the finished product whether yarn or fabric (Morton and Hearle, 1975). In tensile testing, the breaking force of fabric is calculated to be the maximum force applied to a material carried to rupture. This breaking force is the tensile strength of the fabric (Ashraf, 2015). The breaking load/extension for grey, mercerized, Jatropha esterified samples are outlined in Table 1. The mercerized samples showed the highest strength with a breaking load of 282.88N and 176.10N as well as extension of 20.20mm and 17.74mm along warp and weft directions, respectively. This implies that mercerization impacts strength to the fabrics due to realignment of the fibre along the axis even though the coarseness and much creasing as observed on the fabrics are undesirable. Most importantly, the effect of esterification on the fabric strength is apparent compared to that of the grey sample. The tensile properties of Jatropha esterified fabric with breaking load of 280.78N and 170.40N, extension of 18.07mm and 15.88mm along warp and weft directions, respectively competes favorably with the mercerized samples. The slight decrease in strength between the mercerized and esterified samples agrees with investigation by Yuping *et al.* (2007) and Omizegba *et al.* (2017) who stated that esterification reduces the crystallinity of cellulosic fabric. The tensile parameters for grey fabric was the lowest 223.87N and 109.39N with extension of 3.64mm and 3.56mm in warp and weft directions respectively.

Effects of treatments on dry crease recovery: The hydrogen bonds crosslink the cellulose polymers in cotton and these cross linked hydrogen bonds keep in place the cellulose polymers in cellulose fabrics. The disadvantage is that the hydrogen bonds are relatively weak and easily broken by moisture. Once this happens, the fibres can shift place and realign themselves and crease is born in cellulose based fabric thereby creating an ugly and undesirable sight. The ability of a fabric to go back to its original position after creasing is termed crease recovery (Muthu, 2017) and is expressed quantitatively in terms of crease recovery angle. If the angle is 0° then there is no recovery and if 180° , then recovery is full (Omizegba *et al.*, 2015). Crease recovery gives information on the flexibility of the material and how easy it is for the material to recover from deformation. The dry crease recovery angle for the grey, scoured bleached, mercerized, Jatropha oil esterified samples are: 50° , 75° , 80° , 128° , for warp direction, then 45° , 70° , 70° , 76° , 108° , for the weft direction respectively are represented in Fig. 5. Clearly, it can be seen that Jatropha oil esterified sample gave the highest recovery in both warp and weft directions. This suggests that the esterified fabrics are more flexible and elastic than other samples which may be due to the O-CO- ester bond. The grey fabric recorded the lowest crease recovery due to the presence of the 3-OH bonds in the cellulose chain capable of holding the molecule in a fixed position because of hydrogen bonding; hence this fabric is very rigid.

Effect of treatments on wet crease recovery: Wet crease recovery is the ability of a material to bounce back from deformation during wet treatment and is a measure of the strength of a fabric during wetness (Steele, 2016). The wet crease recovery angles for grey, scoured, bleached, mercerized and *Jatropha* oil esterified samples are 37°, 48°, 48°, 54°, 86° along warp direction and 35°, 39°, 40°, 49°, 75° in weft direction respectively are outlined in Fig. 6. It was observed that the wet crease recovery angle is lower than the dry crease recovery angle in Fig. 6. This may be attributed to the shift in the position of the hydrogen bonds after the fabrics were immersed in water (Klemm *et al.*, 2005).

Conclusion

This research investigated the effect of esterification using *Jatropha curcas* seed oil on the physical and mechanical properties of cellulosic fabric. The results showed that generally there was improvement in the properties due to the various treatments and even more due to esterification. However, there was a slight reduction in tensile properties of esterified fabrics compared to the mercerized fabrics. The improvement in crease recoveries after esterification was remarkable. Based on these findings *Jatropha curcas* seed which has been shown to contain a good amount of oil could be fully harnessed. The oil is recommended for easy-care finish in the textile industry because it is non-toxic, biodegradable and locally available than amino resins, hence would reduce the cost of the finished products.

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