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Structural Modification of Cellulosic Fabric via Esterification Using *Balanites aegyptiaca* Seed Oil

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Authors' contributions

This work was carried out in collaboration between all authors. Author FIO designed the study, performed the research and carried out the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors KAB, JOA, HMA and DEAB supervised and managed the analysis of the study. Author IYC managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Cellulose fabrics were esterified by using varying volume of *Balanites aegyptiaca* seed oil. X-ray diffraction analysis was carried on the esterified fabrics and unesterified fabric. The crystallographs of all esterified fabrics gave almost the same profile different from that of control fabric. The control fabric gave a sharp single peak at 24.033º diffractometer angle. Esterified fabrics gave a slightly broad split peaks at lower diffractometer angles which ranged from 20.080 – 22.690º, suggesting that there was structural modification of cellulose. The inter-atomic distance (d-spacing) for control fabric was 3.7027 Å. There was increase in d-spacing which ranged from 3.9192 – 4.4216 Å for esterified fabric. The peak width increased from 1.5º (control) to range of 1.6 – 2.8º (esterified) fabrics. The peaks intensity increased from 5489 (control) to highest value (7798) for 30 cm^3 of oil esterified fabric. The crystallite size reduced from 9.9 nm (control) to a maximum value (9.2 nm) for

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40 cm^3 and to minimum value (5.3 nm) for 10 cm^3 oil esterified fabrics. The percentage crystallinity reduced from 65% (control) to a range of 62–63% for esterified fabrics. From the results obtained, it can be concluded that reduction in crystallite size, increase in d-spacing, intensity and peak width were due to the presence of a bulky ester group in the cellulose chain; this may also have led to the observed decrease in percentage crystallinity of the esterified fabrics. This structural modification is expected to have direct consequences on the physical and mechanical properties as well as the dyeing properties of the esterified fabrics.

Keywords: Modification; cellulosic fabric; esterification; Balanites aegyptiaca; x-ray diffraction.

1. INTRODUCTION

Cellulose is the component of fibers of vegetable origin [1,2]. It is linear, consisting of long, continuous, covalently bonded atoms [3]. Cellulose is cheaply available in many plant sources and its application in textile is vast. X-ray diffraction photographs [4] showed that cellulose is both crystalline and amorphous in nature [5,6]. The problem is that the amorphous region is the reason for creasing and other forms of distortion when cellulose fabric is under stress or in use. Crease recovery depends on fabric construction, yarn twist, pressure and time [7]. The 3-OH groups in the cellulose molecule provide a characteristic ability of cellulose to produce numerous cellulose derivatives, excellent moisture and dye absorption [8]. In the past, urea and melamine formaldehyde resins have been applied to improve the physical, mechanical and dye fastness properties of cellulosic fabric [9-12].

The problem is that these resins are not entirely satisfactory for easy-care finishes because apart from the very high cost of melamine and urea; formaldehyde is very toxic to man and the environment. Also the resins are not sufficiently resistant to repeated washing and they contain secondary amino groups which react with hypochlorite to form chloramines [13]. The chloramines decompose on storage with the accompanying appearance of a yellow discoloration and under the influence of ironing they give rise to hydrochloric acid which causes loss of strength to the fabric [13,14].

Globally, increasing attention has been focused on green chemistry due to environmental degradation and economic challenges. Therefore, it has become imperative to source for locally available materials that are biodegradable, cheaper and could be used for easy-care finish in the textile industry. In this study, esterification of cellulosic fabric with fatty acid from seed oil of *Balanites aegyptiaca* was attempted as a replacement to amino resins. The new properties of the fabric was investigated and compared with the unesterified fabric.

Balanites aegyptiaca is an oil seed yielding plant belonging to the family Zygophyllaceae [15]. It is called desert date in English and is one of the most common, but neglected wild plant species of dry land areas of Africa and South Asia [16]. It is mainly cultivated for fibre and oil although many parts of the plant are used as famine foods in Africa and some developing countries [17]. It is also used for herbal medicine [18]. The oil is known for its good physical and chemical properties [19].

This paper presents the results of powder x-ray diffraction obtained from reaction between OH of cellulose and the –COO- of triglyceride in the oil via esterification. A cellulose monoester was obtained with modified structural properties and increased weight. The impact of this structural modification on the physical and mechanical properties of the fabric will be the main focus of next research.

2. MATERIALS AND METHODS

Balanites aegyptiaca fruits were obtained in Bauchi metropolis. Its identification was established by Alexander Sindama of biological Sciences department, Federal University, Wukari, Taraba State. A loom state 100% cotton fibric of commercial quality. Chemicals of commercial grade, Oven, Soxhlet apparatus, Weighing balance, Beakers, Reflux condenser, Bruker D8 Advance x-ray Machine and accessories.

2.1 Fabric Identification and Purification

Standard methods according to ASTM [20] were employed for fabric analysis. Threads were subjected to solubility and flame test to ascertain the origin. The fabric was scoured, bleached and mercerized using 2% sodium hydroxide, 5%

hydrogen peroxide and 20% sodium hydroxide solutions respectively.

2.2 Extraction of Oil

Extraction and characterization of the oil was based on the standard method by Pearson [21]. 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 ground to fine powder.

The powder (50.0 g) was placed in a pre-weighed thimble and then placed in the barrel of the Soxhlet apparatus. 200 cm^3 of n-hexane was poured into the flask and the apparatus set for extraction and allowed to run for 6 hours. Percentage yield was calculated using equation 1;

$$Yield\ \% = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \quad (1)$$

Esterification of fabric was carried out using 100 cm^3 of methanol mixed with 10, 20, 30, 40, 50 and 60 cm^3 of the oil respectively. 0.5 cm^3 of concentrated H_2SO_4 was added as catalyst and refluxed for 1 hour at 60ºC. The mercerized samples were weighed and then immersed into each flask and refluxed for 3 hours at 60ºC with occasional shaking. The fabrics were removed and neutralized in 2% solution of Na_2CO_3 in order to destroy any acid residue that remained in the samples. The residual oil was removed by immersing the fabrics in a very dilute detergent solution. The samples were rinsed in distilled water and dried in the oven at 60ºC for 20 minutes, then weighed again.

2.3 X-ray Diffraction Analysis

This analysis was carried out in the University of Kwazulu Natal, West Ville Campus 4000 Durban, South Africa, using Bruker D8 Advance.

The control fabric measuring 1 cm x 0.5 cm was ground into fine powder. The ground sample was placed in a glass tube and was mounted on the Bruker D8 Advance x-ray machine. The sample was positioned in the centre of the copper source monochromatic radiation (ʎ = 1.5418 Å). The diffraction pattern was generated by a radiation counter and was plotted automatically. The procedure was repeated for other fabrics esterified under varying volume of the oil. The inter-atomic distance (d-spacing) was calculated according to Bragg's law in equation 2.

$$n\lambda = 2d\sin\Theta \quad (2)$$

The crystallite size was obtained from the scherrer equation with the method based on the width of the diffraction pattern.

$$D = \frac{k\lambda}{B\cos\theta} \quad (3)$$

Where D is the size of crystallite (nm), k is the scherrer constant (0.94), ʎ is the x-ray wavelength and B is the full-width at half-maximum of the peaks measured in 2Θ angle [22].

The percentage crystallinity was calculated according to equation 4.

$$Crystallinity\ (\%) = \frac{I_c}{\sum I_a + I_c} \times 100 \quad (4)$$

Where I_c is intensity of crystalline peak, I_a is intensity of amorphous peak.

3. RESULTS AND DISCUSSION

3.1 Fabric Identification and Purification

The origin of the fabric was confirmed to be 100% cellulose. The burning test showed that the fiber burned rapidly with a yellow flame, giving a powdery ash residue. The solubility test showed that the material was soluble in concentrated sulphuric acid but insoluble in concentrated solution of sodium hydroxide.

The effects of purification were very significant. The scoured fabric became more absorbent, cleaner and softer to handle. This may be due to hydrolysis of oil and fat in the presence of hot alkali, emulsification of unsaponifiable fats and waxes, degradation of proteins, pectins, lignins, nitrogenous compounds into soluble salts [9]. The outstanding whiteness of the bleached material may be due to lose of coloring matter. There was swelling of the mercerized fabric, dimensional shrinkage and increased luster.

3.2 Oil Extraction and Percentage Yield

The seeds of *Balanites aegyptiaca* contain a high amount of oil (40%) which could be highly economical in chemical industries. The oil is light yellow in colour, odorless, tasteless and liquid at room temperature. Hence, this plant could be fully harnessed for oil production.

3.3 Fabric Esterification

Esterification of cellulosic fabric was carried out using varying volume of *Balanites aegyptiaca* seed oil ranging from 10–60 cm^3, while the time of esterification was maintained at 4 hours. The effect of esterification was observed in the weight of the fabric which was measured before and after esterification as shown in Table 1.

Table 1. Effect of esterification and volumes of oils on weight of fabrics

Volume of oil (cm^3)	Weight of fabric before esterification (g)	Weight of fabric after esterification (g)
10	1.0375	1.0661
20	0.9907	1.0230
30	0.9768	1.0197
40	1.0432	1.0833
50	0.9861	1.0072
60	1.0071	1.0352

The increase in weight after esterification is indication that a reaction occurred between the oil and the cellulosic fabrics. It also implies that a bulky ester group has been introduced into the structure of cellulose. The percentage increase in weight of the esterified fabrics was calculated and presented in Fig. 1.

The result showed that the highest weight (4.39%) was obtained with using 30 cm^3 of oil followed by 40 cm^3 indicating that there is increase in thickness of these fabrics than others. This structural modification may also limit the permeability of air; being a very important parameter for assessing the comfort of clothing materials.

3.4 X – ray Crystallography

X – ray diffraction analysis of untreated (control) and esterified fabrics using *Balanites aegyptiaca* seed oil was carried out and presented in Figs. 2 – 8. This investigation was necessary to further ascertain the impact of esterification on the structure of the cellulosic fabric.

With respect to Fig. 2, the diffraction pattern of the unesterified cellulose showed a sharp single peak. The diffraction pattern of all esterified cellulose as depicted in Figs. 3 – 8 gave almost the same profile, accompanied by splitting. This suggests that structural modification has taken place. Structurally, cellulose molecule contains 3-main atoms; carbon, hydrogen and oxygen. However, x-ray diffraction hardly picks up hydrogen atom because of its very small size [23]. Therefore, the single peak of the unesterified fabric in Fig. 2 is an indication that ester bond was not formed. While the resulted double peaks of esterified samples in Figs. 3–8 are evidence of ester formation. The first peak having lower intensity accounts for the oxygen atom that links the cellulose while the second peak with higher intensity account for the carbonyl group of the ester.

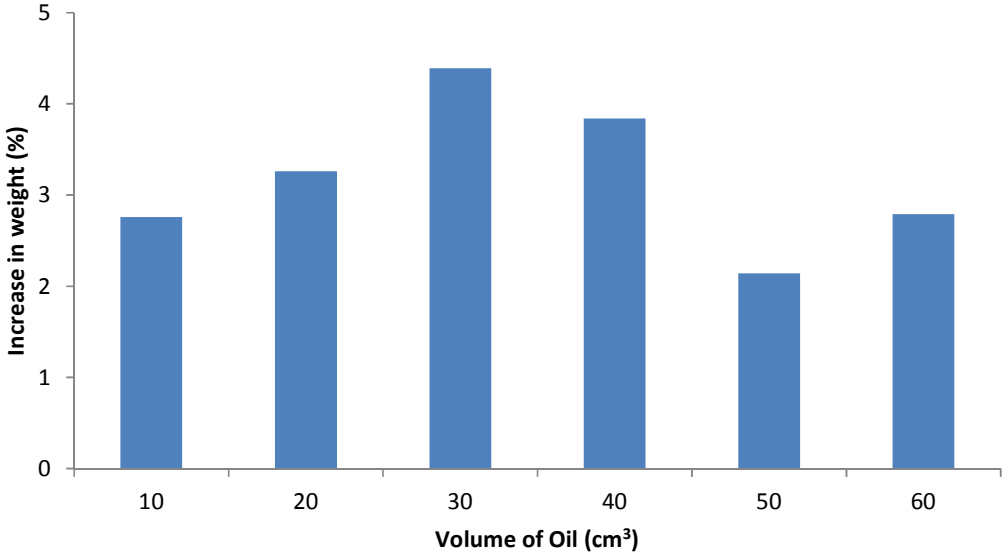


Fig. 1. Effect of volume of oil on percentage increase in weight of fabrics

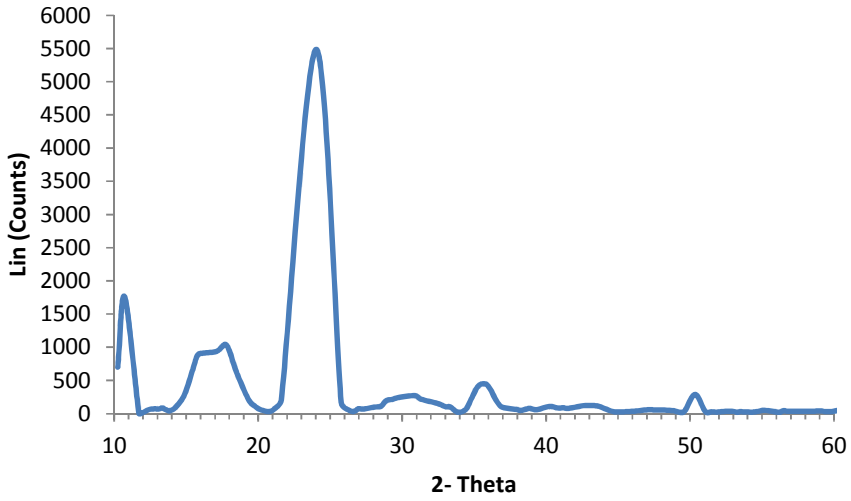


Fig. 2. X-ray crystallograph of unesterified fabric (Control)

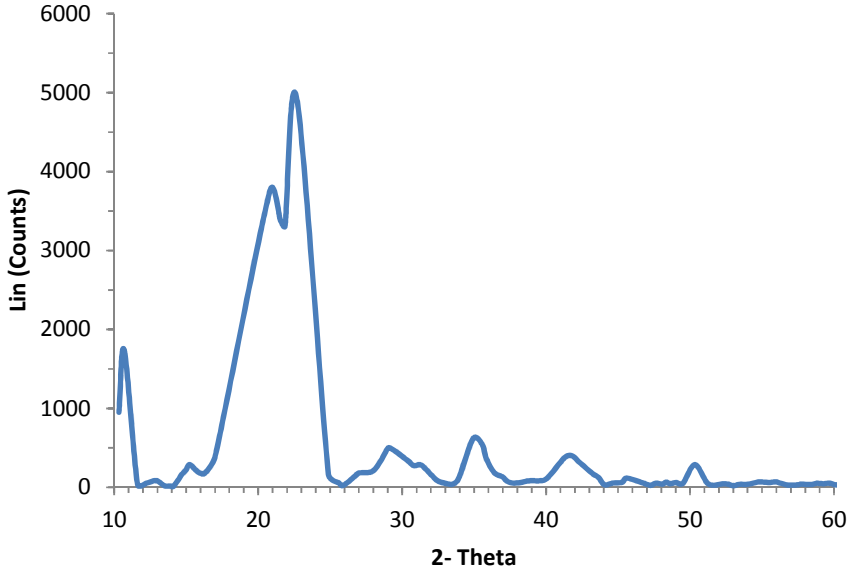


Fig. 3. X-ray crystallograph of fabric esterified with 10 ml *Balanites aegyptiaca* oil

3.5 X-ray Diffraction Analysis

The first step in the analysis of the x-ray pattern obtained in this research was to determine the values of d for the particular type of radiation using the Braggs equation ($n\lambda = 2d\sin\Theta$). The analysis was carried out based on the strongest peaks in the diffraction pattern while every other peak represented a characteristic of the untreated cellulose material [24]. The results of these analyses are presented in Tables 2 and 3.

3.6 Effect of Esterification on the Diffractometer Angle (2-theta angle)

There was a shift of diffractometer angle from 24.033º (control fabric) at 100% count to the lowest (20.080º) at 85% count for –O- atom after esterification with 20 cm^3 of oil. The highest values of diffractometer angle (20.827º) at 76.5% count and (22.690º) at 100% count were obtained at 10 cm^3 optimum volume of oil for –O- and C=O respectively. These shifts in

diffractometer angles may be attributed to the broadening of the diffraction peaks [25] and it is indication that certainly modification has taken place in the structure of the cellulose as a result of chemical reaction with the oil.

3.7 Effect of Esterification on Inter-atomic Distance (d-spacing)

The result showed that d-spacing for the control fabric was 3.7027 Å. However, the effect of esterification led to an increase in values of d-spacing as the volume of oil increased. The values ranged from 4.2650–4.4216 Å at 76.5% and 85.0% counts respectively for –O- and 3.9192–4.0563 Å at 100% count for C=O. the maximum values (4.4216 and 4.0563 Å) for –O- and C=O respectively were obtained at 20 cm^3 optimum volume of oil. Increase in d-spacing for esterified samples suggests that there was modification in the structure of cellulose which may consequently lead to a decrease in crystallinity of the esterified fabrics [26].

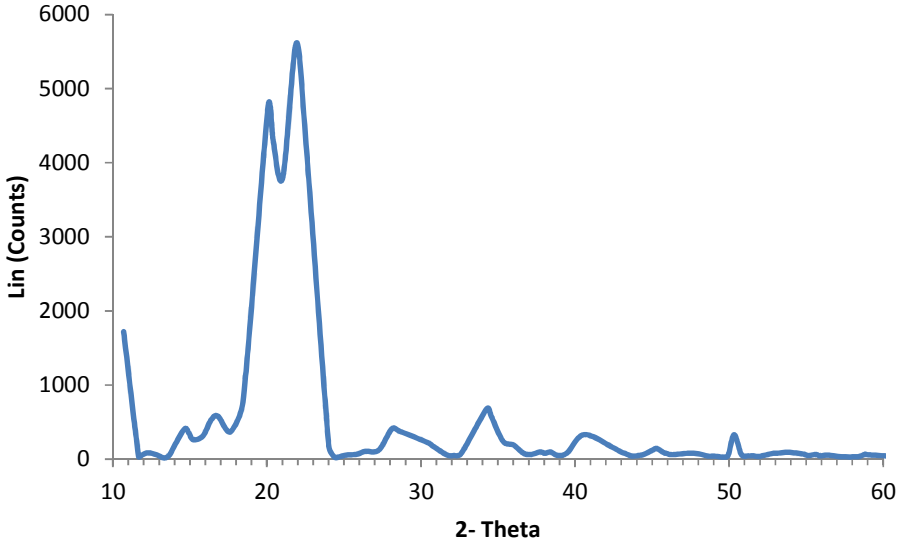


Fig. 4. X-ray crystallograph of fabric esterified with 20 ml *Balanites aegyptiaca* oil

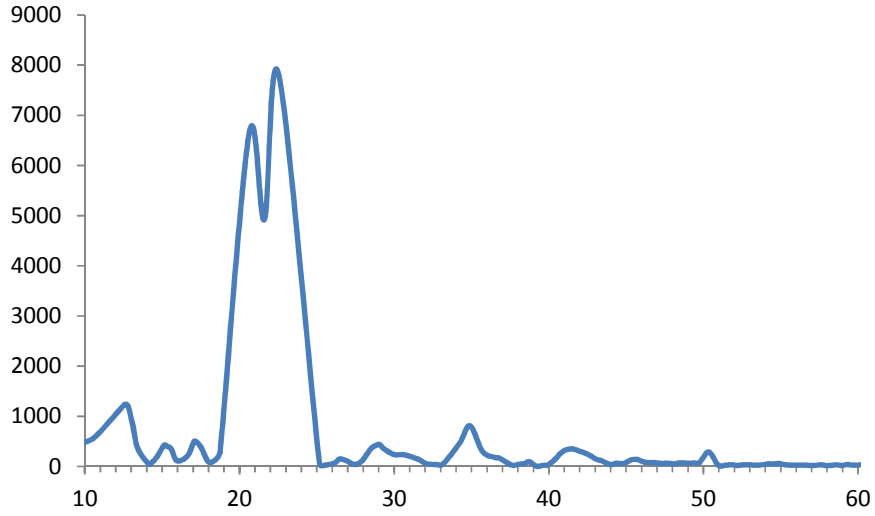


Fig. 5. X-ray crystallograph of fabric esterified with 30 ml *Balanites aegyptiaca* oil

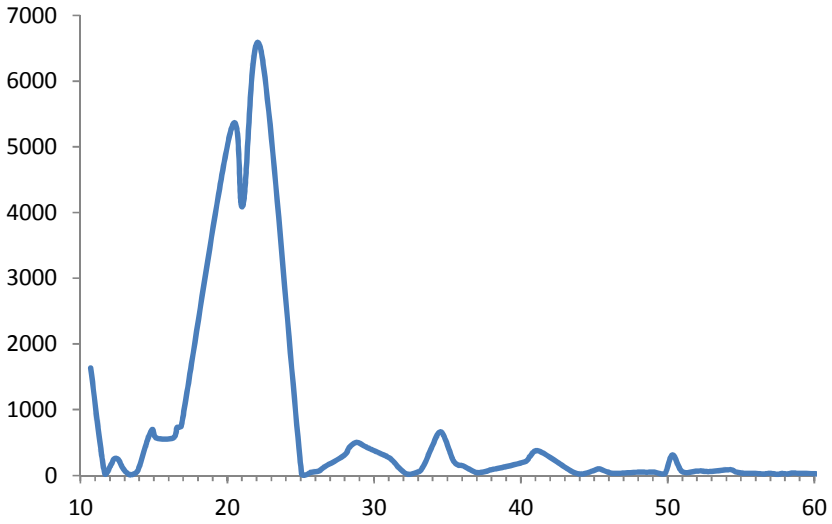


Fig. 6. X-ray crystallograph of fabric esterified with 40 ml *Balanites aegyptiaca* oil

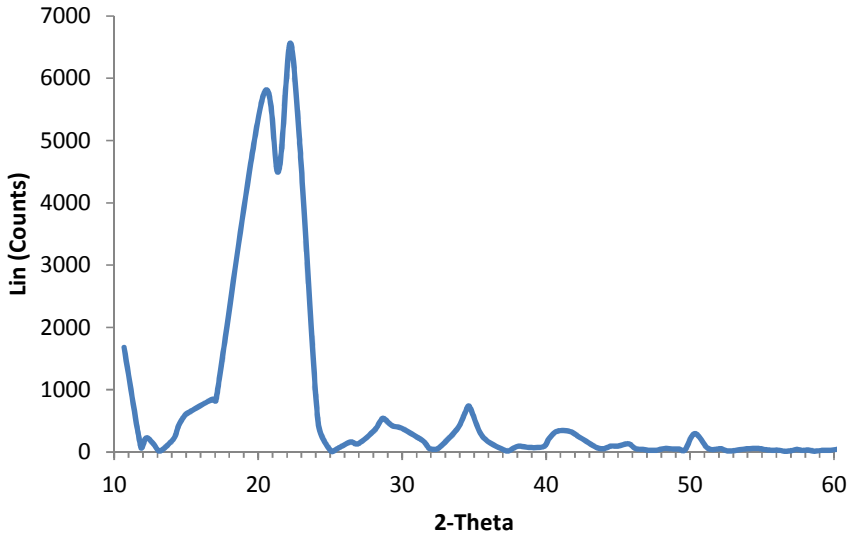


Fig. 7. X-ray crystallograph of fabric esterified with 50 ml *Balanites aegyptiaca* oil

3.8 Effect of Esterification on Peaks Intensity

The control fabric recorded intensity of 5489 at 100% count. After esterification there was increase in intensity as the volume of oil increased except for fabric treated with 10 cm^3 of oil. The increased values ranged from 5617 – 7798 for C=O. This may be attributed to the ester linkage in the cellulose chain that has given rise to higher molecular mass [25]. The maximum values (6651 and 7798) for –O- and C=O at 85.3% and 100% count respectively were obtained with 30 cm^3 optimum volume of oil.

3.9 Effects of Esterification on Peaks Width

The peak widths were obtained from the plots shown in Figs. 2-8 by measuring the full-width at half maximum in 2-theta angle for each peak. The smallest value (1.5º) was obtained for control fabric. This is indication that this fabric may be more crystalline than the esterified fabrics. This is because there was increase in peak widths due to esterification which ranged from 1.6 to 2.8º as the volume of oil increased. The widest peak (2.8º) and narrowest peak (1.6º) for esterified fabrics were observed for C=O and

–O- from using 10 cm^3 and 40 cm^3 of oil respectively with 10 cm^3 being the optimum volume of oil. The observed increase in peak width may be due to the presence of ester bond, it may also be as a result of increase in d-spacing and diffractometer angle [25]. Consequently, this may lead to decrease in crystallinity of the esterified fabrics.

3.10 Effect of Esterification on Crystallite Size

The crystallite size of unesterified fabric (9.9 nm) was obtained in this research. Clearly, there was a decrease in crystallite size due to esterification which was 9.2 nm for –O- at 40 cm^3 optimum volume of oil. The observed decrease in crystallite size may be attributed to the presence of short chain ester group and this may result to loss of alignment in the polymer chains. This molecular irregularity is an indication that crystallinity was reduced [25].

3.11 Effect of Esterification on Percentage Crystallinity

Crystallinity determines the strength [11] and other very important mechanical properties such as the ability to recover from deformation [4], it is also used to determine the end use of any product.

With respect to Table 3, the percentage crystallinity of the control cellulose was 65%. However, after esterification a decrease in percentage crystallinity was observed which ranged from 62 - 63% at 20 cm^3 optimum volume of oil. This decrease may be attributed to factors like increase in d-spacing, decrease in crystallite size and the broadening of peak width [25,24, 27]. The presence of bulky ester side groups and loss of alignment [28] may also be responsible for the decrease in crystallinity. The extent of esterification according to the x-ray pattern in Figs. 2–8 showed that a cellulose mono-ester was produced and the bulky ester groups is attached to the primary carbon (sixth position) atom on the cellulose chain as shown in the Scheme 1. The reduction in crystallinity also means that the fabric has become more porous which could be an advantage for easy dyeing and good dye absorption. However, the reduction in crystallinity may lead to decrease in tensile modulus. Tensile strength is the property of a fiber defined as the ability to resist stress and it has a direct influence upon the strength of the finished product, whether yarn or fabric [29].

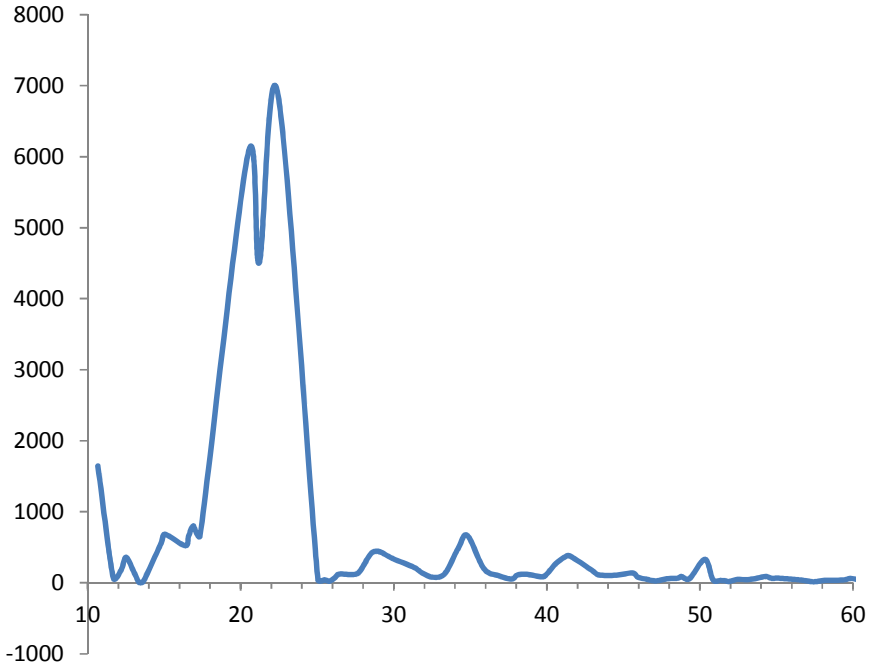
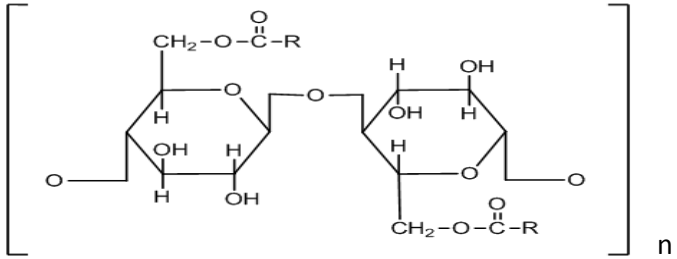


Fig. 8. X-ray crystallograph of fabric esterified with 60 ml *Balanites aegyptiaca* oil

Table 2. X-ray diffraction analysis of fabric esterified with varying volume of *Balanites aegyptiaca* oil

Properties	10 cm^3		20 cm^3		30 cm^3		40 cm^3		50 cm^3		60 cm^3		Control
Functional groups	**- O -**	**C = O**	**- O -**	**C = O**	**- O -**	**C =O**	**- O -**	**C = O**	**- O -**	**C = O**	**- O -**	**C = O**	**O – H**
2-theta Angle (^{o})	20.827	22.690	20.080	21.912	20.642	22.537	20.261	22.272	20.326	22.215	20.471	22.423	24.033
d-spacing (Å)	4.2650	3.9192	4.4216	4.0563	4.3031	3.9452	4.3826	3.9912	4.3689	4.0016	4.3382	3.9645	3.7027
Intensity	3761	4913	4773	5617	6651	7798	5265	6485	5703	6563	6038	6886	5489
Count (%)	76.5	100.00	85.0	100.0	85.3	100.0	81.2	100.0	86.9	100.0	87.7	100.0	100.0
Peak Width (^{o})	2.0	2.8	1.9	2.6	2.1	2.4	1.6	2.1	2.1	2.2	2.0	2.2	1.5
Crystallite Size (nm)	7.4	5.3	7.8	5.7	7.0	6.2	9.2	7.0	7.0	6.7	7.4	6.7	9.9



Scheme 1. Cellulose mono-ester
R is alkyl group in the oil

Table 3. Percentage crystallinity

Volume of oil (cm^3)	Balanites oil esterified fabric crystallinity %
0	65
10	62
20	63
30	63
40	63
50	62
60	62

0 cm^3 is for unesterified sample

4. CONCLUSION

In this research, cellulose fabric was esterified by varying the volume of *Balanites aegyptiaca* seed oil. X-ray diffraction of the esterified samples proved that there was structural modification that led to the formation of cellulose mono-ester. The observed increase in weight and decrease in crystallinity are evidences of the presence of bulky ester group in the cellulose molecule. Increase in d-spacing, peak width, fabric weight and reduction in crystallite size of the cellulosic material are all evidences of structural modification. The effects of this structural modification on the physical and mechanical properties of the cellulose ester will be the main focus of next research. Base on the present findings it is recommended that oil from *Balanites aegyptiaca* be used to replace amino resins for improvement of easy-care finish in the textile industry because it is cheaper, non-toxic and biodegradable. The cellulose ester produced is expected to have a better dimensional stability and resistance to microorganisms such as bacteria and fungi due to hydrophobic property of esters. The new fabric will be comfortable for clothing during the low temperature season of the year due to the observed increase in thickness. Hence, this new product will be suitable for shirt making and drape.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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