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EFFECT OF ESTERIFICATION BY VARYING THE VOLUME OF *BALANITES AEGYPTIACA* SEED OIL ON SOME PHYSICOCHEMICAL PROPERTIES OF CELLULOSIC FABRIC

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ABSTRACT

This research investigated the effect of varying the volume of *Balanites aegyptiaca* seed oil via esterification on the physicochemical properties of cellulosic fabric. The oil was soxhlet extracted with hexane. The oil yield was 40% and the moisture content was 0.22%. The fabrics (10cm x 10cm and 21 cm × 2.5 cm) were pretreated by scouring, bleaching and mercerization then esterified with $10cm^3$ through $60cm^3$ of oil. The effects of these treatments gave improvement in the dry and wet crease recovery angles and yarn twist. The highest values of dry crease (127° warp and 118° weft), wet crease (71° warp and 61° weft) and yarn twist (24 TPI warp and 22 TPI weft) were obtained with $50cm^3$ of oil. The values of these properties for the control (Unesterified) fabric were; dry crease (50° warp 45° weft), wet crease (37° warp and 35° weft) and yarn twist (14TPI warp and 12TPI weft). These revealed that the improvements were generally better in warp direction than weft direction due to difference in fabric construction. The bending properties increased but did not optimize within the volume of oil used. There was reduction in air permeability and water imbibition due to the presence of a more hydrophobic ester bond. Hence this fabric will be comfortable for clothing during the spring season and for drape making. It will have better resistance to microbial attack during storage.

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INTRODUCTION

Chemical modification of cellulose by esterification has the potential to improve cellulose properties. For instance, most research on esterification of wood cellulose in the recent times is aimed at improvement of dimensional stability and reistance to fungal attack and decay (Rowell, 1983; Larson-Brelid, 1998; Iwomoto and Itoh, 2005) and with improving capacity in polyethylene/wood-flour composite systems (Oksman *et al.,* 1998). Another quality of esterified wood is improved light stability (Chang and Chang, 2001). During esterification, the hydrophilic hydroxyl groups are replaced by an ester link and a more hydrophobic methyl group ($-COOCH_3$).

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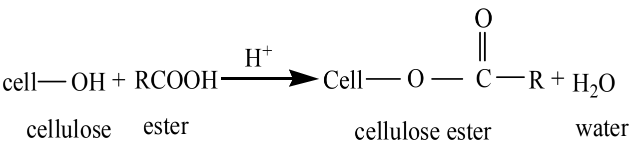
Hence the cellulosic material is expected to become water resistant, with improved dimensional stability and decay resistance. The formation of natural covalent bond ester cross-linkages in cellulose or lignocellolosic composites materials during drying and eating is of interest for production of paper and wood composite boards and in giving cotton materials improved characteristics (Pantze, 2006). Cellulose and linocellulosic materials contain considerable number of hydroxyl groups. Therefore, under suitable reaction conditions such as heating and drying, esterificaiton reactions can probably occur between hydroxyl and carboxylic groups within the material (Pantze, 2006). This has been achieved by reacting OH groups in cellulose with the free fatty acid of *Balanites aegyptiaca* seed oil. Results of the x-ray diffraction analysis revealed evidence of structural modification which resulted to the formation of cellulose mono-ester (Omizegba *et al.,* 2017). Structural modification in cellulosic fabric became necessary because of the natural tendency of cellulose textile materials to crease or deform during processing or in use (Ajayi *et al.,* 2005). The deformation is basically attributed to the presence of amorphous regions. By improving the intermolecular attraction in this region and enhancing the fibre modulus through esterification, cellulose can become processible into various useful forms and solutions to be used for coating and casting of films and membranes (Tosh, 2011). Esters are structurally flexible functional groups because of rotation about the C-O-C bonds which has a low barrier (Eugenio, 2014) The flexibility and low polarity is manifested in their physical properties; they tend to be less rigid due to lower melting point and more volatile as a result of lower boiling point than their corresponding amides (March, 1992). *Balanites aegyptiaca* is chosen for this study because it contains a good amount of oil (40% - 60%); the seed cake remaining after extraction is commonly used to treat tumors and wounds in some part of Africa (Khalid *et al.,* 2010).

It is mainly cultivated for fibre and oil (Wilson *et al.,* 2009) although several parts of the plant have been utilized also in India for various ethnobotanical purposes (Chothani and Vaghasiya, 2011). The leaves and flowers are cooked for food, the fruits are fermented for alcoholic beverages (Guinand and Lamessa, 2000). The oil could also be a good raw material for biodiesel production (Gutti *et al.,* 2011). Even though every part of *Balanites aegyptiaca* plant has been used for various purposes as stated above, never has it been used in textile processing. Hence, this research investigated the effect of varying the volume of *Balanites aegyptiaca* seed by esterification on crease recovery, Bending properties, Yarn twist, Air permeability and water imbibitions after pretreatments of scouring, bleaching and mercerization. Equation 1 represents the reaction of the OH group in cellulose and COOH of the acid in the oil while Fig. 1 shows the structure of the cellulose monoester obtained from x-ray diffraction analysis (Omizegba *et al.,* 2017).



Where R is the triglyceride chain

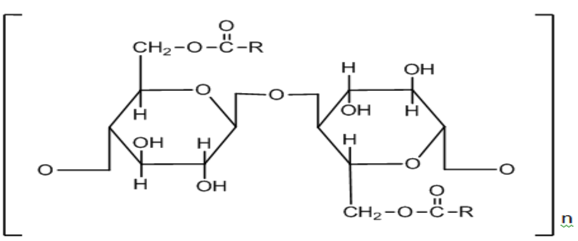


Fig. 1. Structure of Cellulose Monoester

MATERIALS AND METHODS

Materials: All chemicals were analytical grades supplied by BDH Chemical Ltd. Poole, England. They are: sodium hydroxide, acetic acid, sodium silicate, magnesium sulphate, hydrogen peroxide, sulphuric acid, sodium carbonate, methanol. The equipments include: Oven (Memmert 854 Schwabach and Gallenkamp size on Bs), Digital weighing Balance (Santer RC 8021), Analytical Balance (Model UJ07932 Florham Park USA), Heating Mantle (Cliffon), Shirley Crease Recovery Tester (Model No. 308), Shirley crease loading device (Model 308), Stop Clock (Raffin), Hook's Travelling Microscope (Serial No. 901879).

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 (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;

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

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 was recorded (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:

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

Purification of Fabric: Standard method based on the BS 11 (1974), ASTM (1994) and ASTM (2010) was employed.

Scouring of Grey Fabric: 10 cm × 10 cm and 21 cm × 2.5 cm of the grey fabrics were 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 H_2O_2, 0.1 g $NaSiO_3$, 10 ml of 1% NaOH solution and 0.5 g $MgSO_4$, then rinsed severally in tap water for 10 minutes and neutralized with 5% acetic acid and dried at room temperature.

Mercerization 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 (10, 20, 30, 40, 50 and 60 ml) respectively were mixed; 0.5 ml of concentrated H_2SO_4 was added and refluxed for 1 hour at 60^{o}C. The mercerized sample was weighed (1 g) and then immersed into the flask and refluxed for 3 hours at 60^{o}C with occasional shaking. The fabric was removed and neutralized in 2% solution of Na_2CO_3 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^{o}C for 20 minutes then weighed again.

Crease Recovery

Dry Crease Recovery: The Shirley Crease Recovery Tester was calibrated by adjusting the knobs to face 0^o mark. The fabric samples were cut using the template dimensions along the warp and weft directions for fabrics esterified with varying volumes of oil (10, 20, 30, 40, 50 and 60 ml) respectively. The esterified 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 it was transferred to the Crease Recovery Tester to measure the angle of crease recovery. The procedure was repeated for the control sample.

Wet Crease Recovery: The esterified samples were immersed in water and the excess water on the samples drained with filter paper without pressing, the same was carried out for the control sample. The test procedure was repeated as in dry crease recovery angle measurement.

Bending Length: This test was carried out using a Cantiller Stiffness Tester FF 20 No. 78005. The esterified samples were cut into a dimension of 21 cm x 2.5 cm. The rectangle strip of fabric was mounted on the horizontal platform of the Tester in a way that it hung like a cantilever and bent down-wards. The bending lengths and their corresponding angles were recorded from the testing device for all the esterified fabric and for the control fabric.

Yarn Twist: A single yarn was unraveled from the fabric along the warp direction. The yarn was clamped between the two jaws of the Twist Tester at a pre-determined length that is, an inch length, under constant tension. The revolution counter was set to zero while the handle was rotated in the direction that untwisted the yarn. The number of turns of twist formed when a pre-determined length of yarn was formed into a loop as well as the direction of twist was read off the counter. The procedure was repeated for the weft yarn for all the esterified samples and that of the control fabric.

Air Permeability: A sample measuring 20 cm x 2.5 cm was used for this test. Air was drawn through the specimen from the Air Permeability Tester. The air flow was maintained under a suitable water pressure while the rate of air flow was read off the appropriate calibrated capillary for all the esterified samples and that of the control sample.

Water Imbibition: The esterified fabrics were weighed and soaked in 250 cm^3 of distilled water in a beaker for 5 minutes. They were removed and mopped with filter paper gently to remove excess water and then reweighed again immediately. This was followed by progressive drying at 80^{o}C in an oven for 5, 10, 15, 20, 25 and 30 minutes. At each of these intervals, the weight of the samples were recorded using analytical balance. The temperature of the laboratory was maintained at 25^{o}C during the experiment. The procedure was repeated three times for each sample and the average weight was calculated using varying volumes of oil (10, 20, 30, 40, 50 and 60 ml). The experiment was repeated for the control sample.

$$Water\ imbibition\ (regain)\% = \frac{mass\ of\ water\ retained}{mass\ of\ dry\ sample} \times 100$$

RESULTS AND DISCUSSION

Percentage Yield of Oil: After extraction with hexane, the percentage yield of the oil was 40% (Omizegba *et al.,* 2017). The oil has neither taste nor odor 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 *Balanites aegyptiaca* contained 0.22% moisture and this value is with below 0.55% moisture content and within the range for edible oil as recommended by ASTM (2010). This implies that the low moisture content of the oil will not interfere with esterification.

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 Trotman (1970), and Timar-Balazsy and Eastop (2011). The stabilizing action of the sodium silicate in the liquor also enhanced the bleaching process.

Mercerization of Bleached Fabric: The effects of mercerization are evident in the physical appearance of the mercerized fabric. There was a clear difference between the mercerized and the unmecerized fabrics due to the formation of alkali cellulose or cellulose alcoholate which appeared lustrous, shrank longitudinally, swelled laterally, more absorbent and creased very badly (Omizegba *et al.,* 2017; Sadov *et al.,* 1973).

Esterification of Mercerized Fabrics: The fabric was esterified by varying the volume of oil. The x-ray diffraction revealed structural modification leading to changes in the internal structure of the new cellulose monoester (Omizegba *et al.,* 2017) compared to the unesterified fabric. There was a noticeable increase in the weight and a decrease in water absorbency of the esterified fabrics due to the presence of a more bulky and hydrophobic ester group in the cellulose chain (Omizegba *et al.,* 2017). Therefore it is believed that the structural modification might be the reason for the observed improvements in the physicochemical properties of the fabrics.

Effect of Varying Volume of Balanites aegyptiaca Oil on Dry Crease Recovery Angle of Esterified Cellulosic Fabric: Crease recovery is a factor that depends on the elastic behavior of the fibre, yarn and fabric geometry and the ability of the fabric to recover from deformation in use or during laundry that would otherwise distort the aesthetic appeal of the fabric (Fashola and Alonge, 2002). With respect to Fig. 2 as the volume of oil increased, the dry crease recovery angle increased, for the esterified fabrics than that of the control fabric ($0cm^3$). The recovery in warp direction ranged between 80^o to 127^o optimum at $50cm^3$ oil, while the recovery in weft direction ranged between 75^o to 118^o. The dry crease recovery for the control fabric is 50^o warp and 45^o weft. The increase in crease recovery may be attributed to the presence of a more flexible O-CO- bond in the cellulose ester (Eugenio, 2014; Omizegba *et al*., 2017), hence reduction in rigidity of the esterified fabric. The crease recovery is more in the warp way than in the weft way because warp yarns are well in quality, strength and are kept in more tension during weaving (Adanur, 2001; Muthu, 2017).

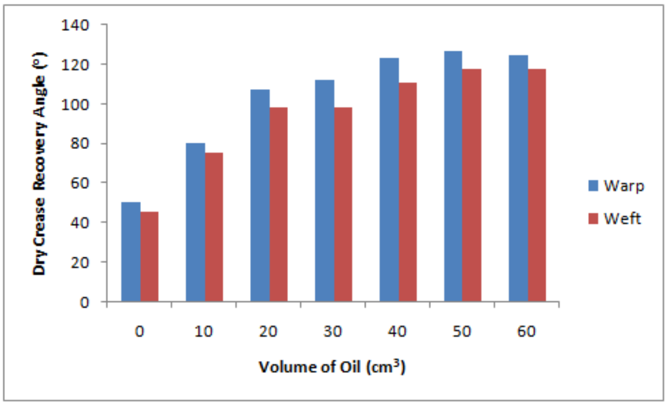


Fig. 2. Dry Crease Recovery Angle of Cellulosic Fabric Esterified with Varying Volumes of *Balanites aegyptiaca* Seed Oil (Warp and Weft Directions)

Effects of Varying Volume of Balanites aegyptiaca on Wet Crease Recovery of Esterified Cellulose Fabric: Wet crease recovery is the ability of a material to bounce back from deformation during wet treatment. It is a measure of the strength of a fabric during wetness (Steele, 2016). In Fig. 3. The wet crease recovery angle recorded a remarkable improvement. The values ranged from 61^o to 71^o in warp direction and 53^o to 61^o in weft direction, and optimized at $50cm^3$ of the oil. The control fabric recorded the lowest wet crease recovery angle of 37^o and 35^o for warp and weft directions respectively. It is observed that the wet crease recovery angle is lower than the dry crease recovery angle. This may be attributed to the shift in the position of the hydrogen bonds in water by the cellulose structure; hence recovery from deformation is less compared to when the fabric is dry (Klemn *et al.,* 2005). This implies that the inter-fibre hydrogen bond is weakened in the presence of water but this has been improved by esterification. By this, it is an indication that the esterified fabric will not crease as much as the unesterified fabric after laundry.

Effects of Varying Volume of Balanites aegyptiaca Oil on Bending Properties of Esterified Cellulosic Fabric: A stiff material is characterized by poor draping quality. Bending properties of fabric govern much of their performance such as hang and drip and are essential parts of complex fabric deformation analysis (Gonca *et al.,* 2012). Bending in polymer for instance, fabric is related to fibre fineness and flexibility (Bello, 2001). It is true that as fineness decreases, resistance to bending increases more rapidly than does fibre weight per unit length (Amutha, 2016). The bending property of fabric esterified with varying volumes of *Balanites aegyptiaca* oil, for warp and weft directions are given in Fig. 4 and 5 respectively.

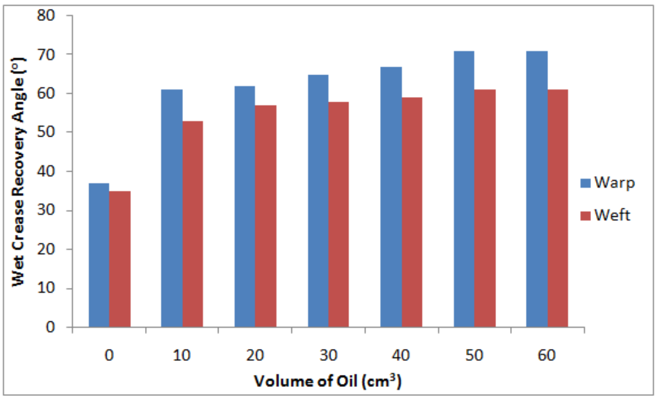


Fig. 3. Wet Crease Recovery Angle of Cellulosic Fabric Esterified with Varying Volumes of *Balanites aegyptiaca* Seed Oil (Warp and Weft Directions)

Generally, there was a progressive increase in bending angle and bending length of the esterified fabrics along warp and weft directions as the volume of oil was increased. The fabric esterified with $60cm^3$ of oil gave the highest bending angles (62^o warp, 89^o weft). It implies that there is an increase in fibre fineness, free rotation and flexibility along the oxygen linking the carbonyl group of the ester and the cellulose chain. The result showed that the control fabric had the lowest bending angle (42^o warp and 74^o weft). This again may be attributed to the presence of strong hydrogen bonding between the chains in cellulose thereby holding the chain in a fixed position (Omizegba *et al.,* 2015). By implication, this fabric is less flexible or stiffer than the esterified fabrics. Therefore, it is expected that the esterified fabrics will have better crease recovery angles as revealed.

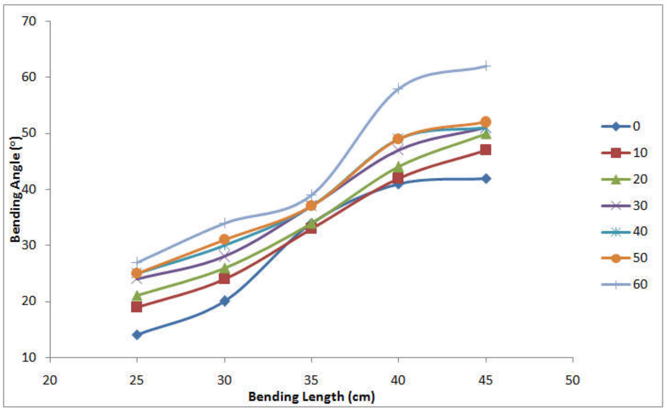


Fig. 4. Bending Properties of Fabric Esterified with Varying Volumes of *Balanites aegyptiaca* Seed Oil (Warp Direction)

Effect of Varying Volumes of Balanites aegyptiaca Oil on Yarn Twist Property of Esterified Cellulose Fabric: In the manufacture of stable fibre yarns, twist is inserted into the fine strand of fibres to hold the fibres together and impart the desired properties to the twisted yarns. Without twist, the fine strand of fibres will be very weak and of little practical use. A change in the level of twist also changes many yarn properties such as strength and softness (Yousuf, 2015). In the case of moisture absorption, high twist holds the fibres tight thus restricting water to enter. The result of this investigation is depicted in Fig. 6. There was a progressive increase in yarn twist as the volume of oil increased. The highest twist (24TPI warp and 22TPI weft) was obtained at 50cm^3 of oil while the control fabric had the lowest twist (14TPI warp and 12 TPI weft). The warp twist appeared to be greater than the weft twist; this may be the reason for the observed decrease in bending angle (less flexibility) along warp direction shown in Fig.4. It also implies that the warp yarn is stronger and stiffer than the weft yarn (Abdullahi, 2011) increase in yarn twist also indicates that the porosity of the fabric will be reduced and this may mean that the esterified fabric will have reduced capacity to retain moisture. Increased yarn twist may also be the reason for the improvement in crease recovery angles as observed in Fig. 2 and 3.

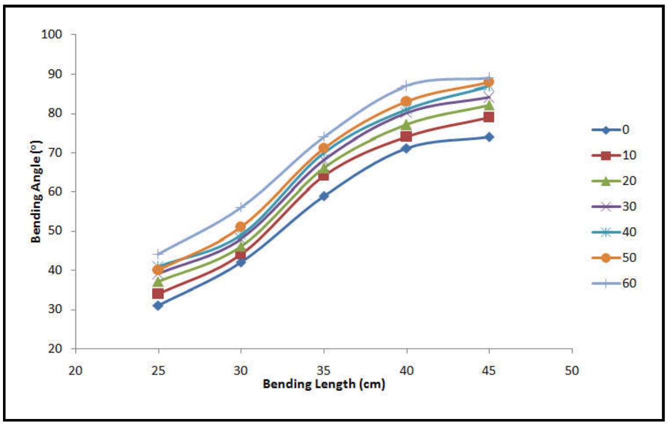


Fig. 5. Bending Properties of Fabric Esterified with Varying Volumes of *Balanites aegyptiaca* Seed Oil (Weft Direction)

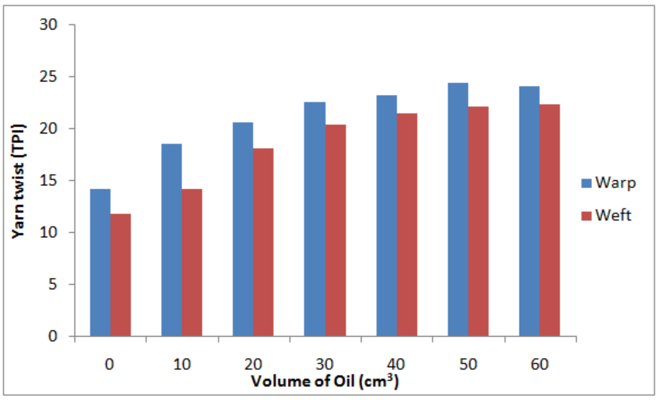


Fig. 6. Amount of Yarn Twist (Z-Direction) Property of Cellulosic Fabric Esterified with Varying Volumes of *Balanites aegyptiaca* Seed Oil (Warp and Weft Directions)

Effects of Varying Volumes of Balanites aegyptiaca Oil on Air Permeability of Esterified Cellulose Fabric: Air permeability is a very important factor in the performance of some textile materials. It is an important parameter for accessing comfortability and fibre fineness. Ogulata (2006) stated that air permeability is mainly dependent upon the fabric's weight and construction (thickness and porosity). Other factors that influence air permeability are fabric structure, the number of warp and weft yarns per centimeter or inch and the amount of twist in yarns (Ogulata, 2006). With respect to Fig. 7, there was observed decrease in air permeability as the volume of oil increased. Clearly, the control fabric recorded the highest air permeability, indicating that this material is less dense and more porous than the esterified materials. This is expected because by esterification, the oil has increased the weight of the fabrics due to a bulky ester group. Again, the porosity is reduced because of the increase in yarn twist as observed in Fig. 6. Hence, 60cm^3 of oil esterified fabric recorded the lowest air permeability, which implies that esterified materials would be more suitable and comfortable during the spring season of the year when temperature is slightly low due to rainfall.

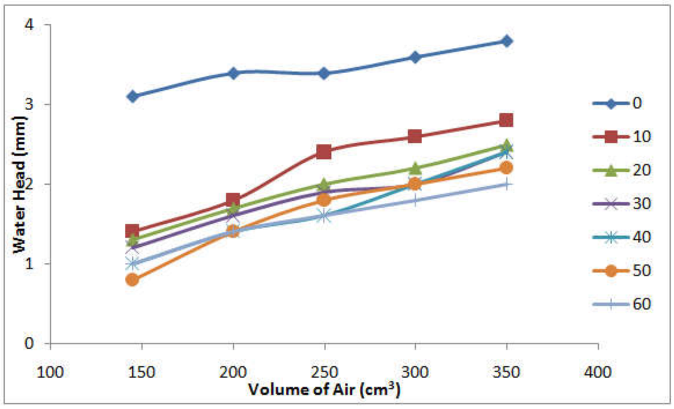


Fig. 7. Air Permeability Property of Cellulosic Fabric Esterified with Varying Volumes of *Balanites aegyptiaca* Seed Oil

Effect of Varying Volumes of *Balanites aegyptiaca* Oil on Water Imbibitions of Esterified Cellulose Fabric: The uptake or absorption of water by a substance without forming a solution is called imbibitions. It is a property of many biological substances including cellulose, starch and some proteins (Robinson, 2016). For any substance to imbibe any liquid, affinity between the absorbent and the liquid is a pre-requisite. Different types of organic substances have different imbibing capacities of which cellulose is the least. The result of this investigation is given in Fig. 8 there was a decrease in water imbibitions as the volume of oil increased with exception of 50cm^3 which recorded the lowest imbibitions as the drying time increased.

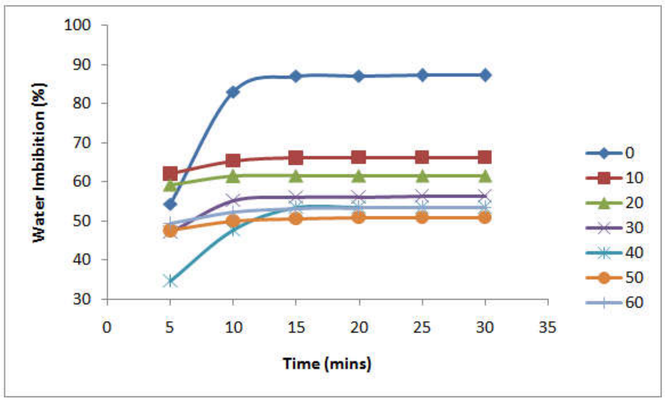


Fig. 8. Water Imbibition Property of Cellulosic Fabric Esterified with Varying Volumes of *Balanites aegyptiaca* Seed Oil

This may be attributed to ester linkage on 1 out of 3 OH groups on the cellulose chain as revealed by x-ray diffraction results in Omizegba *et al.* (2017), thereby made the esterified fabrics less hydrophilic. The reduction in water imbibition may also be linked to the increase in yarn twist as a consequence of reduced porosity of the fabric due to esterification. It is expected that this fabric will retain less moisture and become less susceptible to microbial attack during storage.

Conclusions

Esterification of cellulose fabric by varying the volume of *Balanites aegyptiaca* seed oil was attempted. The effects of this treatment on the physicochemical properties of the fabric were investigated. Generally, there were remarkable improvements in the dry and wet crease recovery angles and yarn twist but more along the warp direction than weft way. The optimum volume for the crease recovery and yarn twist was 50cm^3 of oil. Bending properties increased but did not optimize within the limit of the volume of oil used in this research. The reduction in air permeability and water imbibitions is an improvement because the resulting fabric will be very comfortable for clothing during the spring season. Hence this fabric will have better resistance to mildew and other micro organisms during storage. This research has contributed immensely to knowledge because this is the first time that biodegradable organic seed oil like *Balanites aegyptiaca* is used to modify the physicochemical properties of cellulosic fabric through the process of esterification. This oil could be used in replacement of the present day toxic chemicals used in textile finishing of cellulosic fabric.

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