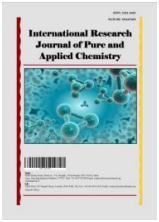

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Assessment of Some Physicochemical Properties of Cellulosic Fabric Esterified Using Varying Volume of *Jatropha curcas* Seed Oil

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Authors' contributions

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

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ABSTRACT

This paper presents the results of the assessment of some physicochemical properties of cellulosic fabric esterified using varying volume of *Jatropha curcas* seed oil. The oil was extracted using Soxhlet apparatus under reflux with hexane as solvent. The percentage yield and moisture content was 47.25% and 0.56% respectively. The fabric (10 cm x 10 cm and 21 cm x 2.5 cm) was identified to be cellulose; it was subjected to purification processes before esterification. The purification processes are scouring, bleaching and mercerization, after which the fabrics were esterified using 10 cm^3 through 60 cm^3 of oil. The results of esterification gave improvement in dry and wet crease recovery angles, and yarn twist. The highest values of dry crease (130º warp and 122º weft), wet crease (74º warp and 68º weft) and yarn twist (25 TPI warp and 23 TPI weft) were obtained with 50 cm^3 of oil. The unesterified (control) fabric recorded lower values of dry crease recovery angles (50º

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warp and 45º weft), wet crease (37º warp and 35º weft) and yarn twist (14 TPI warp and 12 TPI weft). These revealed that esterification is the reason for the observed improvements and was generally better in warp direction than in weft direction. This may be attributed to the difference in the fabric construction. There was increase in bending properties but did not optimize within the volume of oil used. The air permeability and percentage water imbibition was reduced compared to the control fabric; revealing a structural modification and the formation of a hydrophobic ester bond. This may be an indication that the esterified fabric will be more resistant to the formation of mildew during storage. Hence the seed oil of *Jatropha curcas* is recommended for easy care finish in textile industries.

Keywords: Assessment; physicochemical properties; cellulosic fabric; Jatropha curcas; varying volume.

1. INTRODUCTION

The interaction between cellulosic fabric and fatty acid of seed oils in order to enhance the properties of the fabric during use or storage has been a subject of intensive investigation recently. X-ray diffraction techniques have revealed that there is possible interaction between OH of cellulose and COOH in the seed oils of *Jatropha curcas and Balanites aegyptiaca* [1,2]. The elucidated structure was observed to be a cellulose monoester with modified structural properties which include d-spacing, peak width, crystallinity, crystallite size, peak intensity and the angle of diffraction [1,2].

The reaction between the fibre substrate and the fatty acid takes place in acidic condition as shown in equation 1.

(1)

The seed oils used for the esterification act as lubricating agent in the cellulose internal structure and help to provide flexibility by replacing the rigid OH bond with a more flexible –O-CO ester bond.

The industrial application of reactive dyes and resin finishing represents a major innovation in the dyeing and finishing of cellulose fabrics [3–5]. However, the effects of seed oils on structural modification and enhancement of physic-cochemical properties of cellulosic fabric via esterification is completely a new innovation. The intention is to replace toxic and very costly chemicals with more environmentally friendly substances that could yield the same or even better results.

It is a natural phenomenon to find cellulose crease after laundry or in use, thus producing a very rough and ugly surface. Also cellulose material is prone to the formation of mildew during use and under storage in damp condition. This abnormality is caused by the presence of weak amorphous regions, therefore requires to be strengthened by chemical or organic substances. In this time of advancement in technology, it is the sole responsibility of man to improve on the quality of textile fabrics that can meet the world economic standard. Cellulose fabric has gained more economic importance due to its ability to make comfortable clothing and drape. Hence major research and development efforts geared toward cellulose based fabric are ongoing.

The focus of this paper is to present the results of the physicochemical properties obtained by esterification of cellulosic fabrics through varying the volume of *Jatropha curcas* seed oil.

The choice of *Jatropha curcas* is based on its underutilization in this part of the world. The seeds 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 researches to utilize the oils for biodiesel production is ongoing [6,7]. In 2017, Omizegba *et al*. obtained oil yield and moisture content of 47.25% and 0.65% respectively for the seed oil of *Jatropha curcas*.

According to Centre for Jatropha Promotion (CJP) and Biodiesel [8], *Jatropha curcas* is a drought resistant perennial plant that grows well in very poor soil and can produce seeds for a span of 50 years. The oil of the plant is biodegradable and has much medicinal importance [9–11]. The oil is non-edible, hence will not compete for food but could be very useful industrially if fully harnessed. These, to mention but a few are the advantageous reasons why *Jatropha curcas* is chosen for this study.

2. MATERIALS AND METHODS

2.1 Materials

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

The equipment include oven (Menmert 854 Schwabach and Gallenkamp size one Bs), Digital weighing Balance (Model UJO7932 Florham Park U.S.A), Heating Mantle (Clifton), Shirley Crease Recovery Tester (Model No. 308), Shirley Crease Loading Device (Model 308), Stop Clock (Raffin), Hook's travelling Microscope (Serial No. 901879).

2.2 Methods

2.2.1 Extraction of oils

Extraction of the oils was according to Pearson [12]. 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 cm^3) was poured into the flask and the apparatus set for extraction and allowed to run for 6 hours.

2.2.2 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;

$$Percentage\ yield = \frac{w_2 - w_3}{w_2 - w_1} \times 100 \quad (2)$$

2.2.3 Moisture content

A 3 g of oil sample was weighed into an empty crucible (w_1), so that the weights of the crucible and oil sample were recorded as (w_2). The crucible and its content were then placed in an oven at 105ºC for 4 hours after which it was removed, cooled in a desiccator and reweighed (w_3). The process of heating and cooling was repeated until a constant weight was obtained [12]. Percentage of moisture content is calculated as:

$$Percentage\ moisture\ content = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \quad (3)$$

2.2.4 Purification of fabric

Standard method based on the [13,14,15] was employed.

2.2.4.1 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.

2.2.4.2 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 cm^3 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.

2.2.5 Mercerization of bleached fabric

The bleached fabric was immersed in 20% solution of NaOH at 5ºC 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.

2.2.6 Esterification of mercerized fabric

Methanol (100 cm^3) and the oil (10, 20, 30, 40, 50 and 60 cm^3) respectively were mixed; 0.5 cm^3 of concentrated H_2SO_4 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 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°C for 20 minutes then weighed again.

2.2.7 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 cm^3) 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.

2.2.8 Wet crease recovery

The esterified samples were immersed in distilled 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.

2.2.9 Bending length

This test was carried out using a Cantilever 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.

2.2.10 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.

2.2.11 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.

2.2.12 Water imbibitions

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 80ºC in an oven for 5, 10, 15, 20, 25 and 30 minutes. At each of these intervals, the weights of the samples were recorded using analytical balance. The temperature of the laboratory was maintained at 25ºC 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 cm^3). The experiment was repeated for the control sample.

$$Water\ imbibition\ (regain)\% = \frac{mass\ of\ water\ retained}{mass\ of\ dry\ sample} \times 100 \quad (4)$$

3. RESULTS AND DISCUSSION

3.1 Percentage Yield of Oil

The percentage yield of the oil after extraction with hexane under reflux was 47.25% [1,16]. The oil remained liquid at room temperature and a light yellow colour. This is indication that the oil is predominantly composed of low molecular weight unsaturated fatty acid [17]. The oil has no odour and is tasteless, therefore is suitable for easy-care finish of cellulose fabric via esterification.

3.2 Moisture Content

The moisture content obtained was 0.65%; a value slightly above 0.55% standard of moisture content by ASTM [15] for edible oil. With this, it is evident that *Jatropha curcas* seed oil is not good for food but could be very useful industrially. Again, esterification process requires little or no moisture, hence the suitability of the oil for esterification is guaranteed.

3.3 Fabric Identification

The fabric being subjected to identification test through burning and treatment in concentrated

sulphuric acid and sodium hydroxide proved to be cellulose as shown in Table 1. The fabric burned rapidly with a yellow flame, powdery ash residue smelling like burned paper, which are characteristic physical properties of cellulose.

The solubility test showed that the fabric was soluble in concentrated sulphuric acid. It did not dissolve in concentrated sodium hydroxide but the threads coiled and shrank. This implies that the fabric is made of 100% cellulose (cotton) as shown in Table 1.

3.4 Fabric Purification

The change in the physical properties of fabrics because of purification process is outlined in Table 2. After the removal of waxes, fats, proteins, nitrogenous compounds and other mechanically adhered dirt, the fabric appearance was cleaner and brighter because of scouring with 2% concentration of sodium hydroxide. The result also showed that the bleaching process with 5% hydrogen peroxide solution gave a permanent white fabric. The hydrated cellulose obtained after mercerization with 20% solution of sodium hydroxide obviously had a physical property different from the original fabric. There was noticeable shrinkage on both the vertical and longitudinal direction, accompanied with lateral swelling. The fabric appeared more lustrous and badly creased or wrinkled [7,16,18,19].

3.5 Esterification of Mercerized Fabrics

The cellulosic fabric was esterified under reflux by varying the volume (10 cm^3 – 60 cm^3) of oil. The results of the x-ray diffraction revealed structural modification leading to the formation of cellulose monoester with internal properties different from that of the unesterified fabric [1]. The increase in weight and slight resistant to water absorption further proved the presence of a more bulky, hydrophobic ester group in the new cellulose [1,20]. It is believed that these structural modifications might be responsible for the observed improvements in the physicochemical properties of the fabrics.

3.5.1 Effect of varying volume of *Jatropha curcas* oil on dry crease recovery angle of esterified cellulosic fabric

Crease in fabric is a deformation that is capable of distorting the aesthetic appeal of the fabric [21], mainly cellulose base as a result of the presence of hydrogen bonds that are easily broken by moisture. Crease is ugly and undesirable, therefore can be reduced or if possible eliminated. The ability of a fabric to go back to its original position is termed crease recovery and it gives information about the elasticity and the flexibility of the material and how easy it can recover from deformation.

With respect to Fig. 1 as the volume of oil increased, the dry crease recovery angle increased for the esterified fabrics compared with the control fabric (0 cm^3). The recovery in warp direction ranged from 115º to 130º optimum at 50 cm^3 of oil, while that of the weft direction was 108º to 122º. The dry crease recovery angle of unesterified (control) is 50º warp and 45º weft. The reason why crease recovery is more in the warp direction than in the weft direction may be because warp yarns are better in strength and quality and are kept under tension during weaving [22,23]. The observed improvement in dry crease recovery is commendable and the reason may be attributed to the replacement of the rigid hydrogen bond in cellulose with a more flexible ester bond in the cellulose monoester [1,20,24].

3.5.2 Effect of varying volume of *Jatropha curcas* oil on wet crease recovery angle of esterified cellulosic fabric

Wet crease recovery is the measure of the strength of a fabric during wetness [25]. It is the ability of a material to bounce back from deformation after wet treatment. Crease recovery depends on yarn construction, twist of yarn, pressure and time [23]. A material that easily recovers from distortion while wet or dry would be certainly desirable especially for clothing [26]. In Fig 2, the wet crease recovery angle recorded a remarkable improvement. The values ranged from 65º to 74º along warp direction at 50 cm^3 optimum volume of oil. The control fabric gave the lowest wet crease recovery angle of 37º and 35º for warp and weft direction respectively. Generally, the wet crease recovery angle is less than the dry crease recovery angle. This according to Klemn et al. [27] is attributed to the shift in the position of the hydrogen bonds in water by the cellulose structure. By implication, it means that the hydrogen bond that is usually weakened by water has been strengthened and improved by esterification. Therefore, it is expected that the esterified fabric will have improved creasing quality after laundry compared to unesterified cellulosic material.

3.5.3 Effect of varying volume of *Jatropha curcas* oil on bending properties of esterified cellulosic fabric

Polymer chains have the ability to adopt any number of possible configurations. This is important and possible due to freedom of rotation about single bonds in the chain resulting to high degree of flexibility [28]. Bending in polymer for instance, fabric is related to fibre fineness and flexibility. It was stated by Amutha [29] that for a given type of yarn of given count or for a fabric of given weight per unit area, made from a given type of raw material, the resistance to bending diminishes as the fineness of the fibre increased. This is in essence responsible to the depth of dye shade; hence the finer the fibre, the lighter the apparent shade [30].

Table 1. Results of fibre identification

Test	Observation	Inference
Burning	Yellow flame and white ash residue which smells like burnt paper	Fibre is Cellulosic
Alkali Test	Fibre insoluble	Fibre is Cellulose
Acid Test	Fibre degraded	Fibre is Cellulose

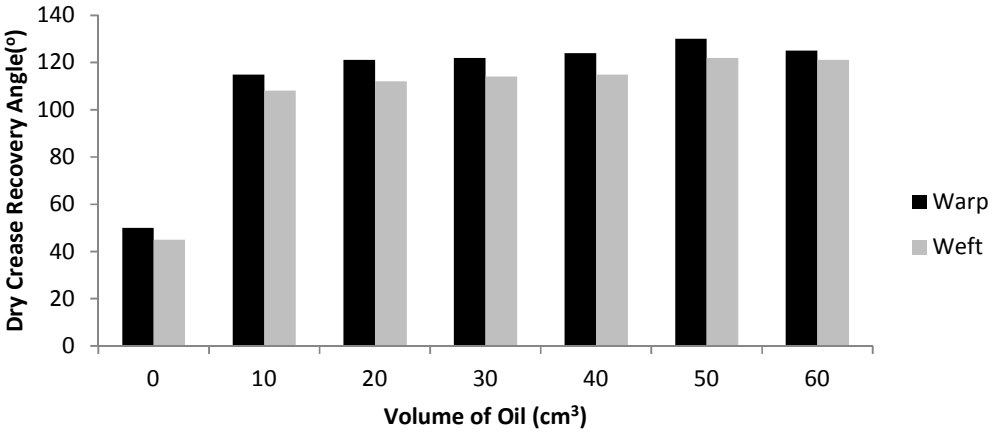

Fig. 1. Dry crease recovery angle of cellulosic fabric esterified with varying volume of *Jatropha curcas* Seed oil (warp and weft directions)

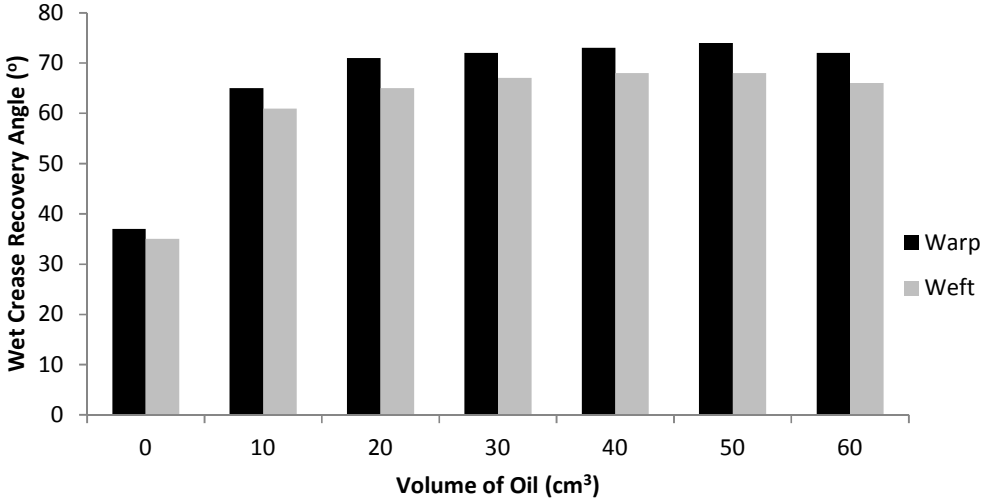

Fig. 2. Wet crease recovery angle of cellulosic fabric esterified with varying volume of *Jatropha curcas* seed oil (warp and weft directions)

Table 2. Results of fabric purification

Test	Observation	Inference
Scouring	Fabric became cleaner and softer.	Fat waxes, protein and nitrogenous compounds degraded, mechanically adhering dirt are loosened.
Bleaching	Fabric became white.	Removal of colouring matter.
Mercerization	Fabric shrank longitudinally and swelled laterally, increase in luster, wrinkled very badly.	Formation of cellulose alkali.

The result of this investigation along the warp and weft direction is presented in Fig. 3 & 4 respectively. The general trend was observed to be 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 material esterified with 60 cm^3 of oil recorded the highest bending angles (61 warp and 83 weft). It implies that there is increase in fibre fineness, free rotation and flexibility along the oxygen linking the carboxyl group of the ester and the cellulose chain [28]. These findings showed that the control fabric had the lowest bending angle (42º warp and 74º weft). This again may be attributed to the rigidity of the hydrogen bonds between the chains thereby keeping the chain in a fixed position [4]. It means that the control fabric is stiffer and may be the reason for the observed lower crease recovery angle compared to the esterified fabric.

3.5.4 Effect of varying volume of *Jatropha curcas* oil on yarn twist properties of esterified cellulosic fabric

Twist is inevitable in fabric construction because of the unique role it plays such as in water absorption, strength, flexibility, wearing abrasion and piling. The twist of the yarns which the fabrics are made of is one of the main parameters affecting the fabric behavior such as bending, stiffness, and shearing property [31]. The result of this investigation is depicted in Fig. 5. There was a progressive increase in yarn twist as the volume of oil increased. The highest twist (25 TPI warp and 23 TPI weft) was achieved at 50 cm^3 optimum volume of oil, while the control fabric had the lowest twist (14 TPI warp and 12 TPI weft). It is observed that the value of the warp twist is more than that of the weft twist; no wonder there was observed decrease in bending angle (less flexibility) of the fabric along warp direction as revealed in Fig. 3. In other words, the flexibility of the esterified fabric is contributed more by the weft yarns. This is an indication that the warp yarn is more in strength and stiffness than the weft yarn [32]. In terms of moisture absorption, increase in yarn twist may mean reduction in fabric pore size leading to reduced capacity of moisture retention. The observed improvement in crease recovery of the esterified fabrics in Fig. 1 and 2 may also be attributed to this increase in yarn twist.

3.5.5 Effect of varying volume of *Jatropha curcas* oil on air permeability of esterified cellulosic fabric

Air permeability is the volume of air in millimeters which passed in 1 second through 100 mm^2 of the fabric at a pressure difference of 10 mm head of water [33]. It is mainly dependent upon the fabrics thickness, porosity and twist [34]. The importance of air and ventilation cannot be overemphasized. Just as the rooms need free movement of air to give freshness to the house and to eliminate bad odour, so does the body need air to keep it dry, and comfortable. Clothing material must be able to exhibit this quality in order to be considered suitable and comfortable [5]. With respect to Fig. 6, it was observed that the air permeability increased as the volume of oil increased. Clearly, the control fabric recorded the highest air permeability which means that this material is less dense and more porous compared to the esterified fabrics. This is obviously not surprising because one of the effects of esterification is increase in weight of the fabric due to the formation of a bulky ester group [1].

Again, there is reduction in porosity due to the increase in yarn twist as observed in Fig. 5. However, 60 cm^3 of oil esterified fabric recorded the lowest air permeability, which implies that esterified fabrics may be more suitable in spring season when the temperature is slightly low.

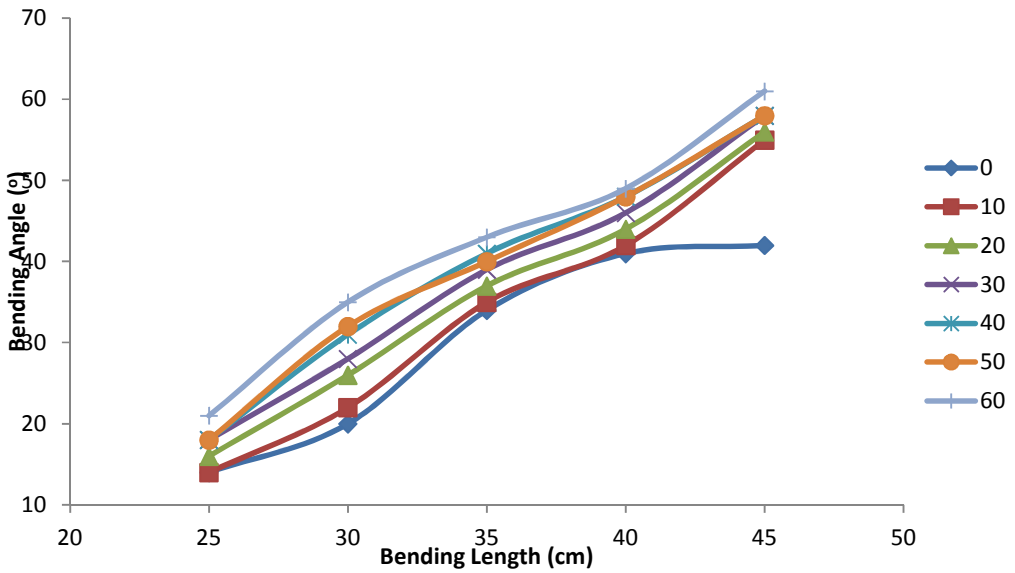

Fig. 3. Bending properties of fabric esterified with varying volume of *Jatropha curcas* seed oil (warp direction)

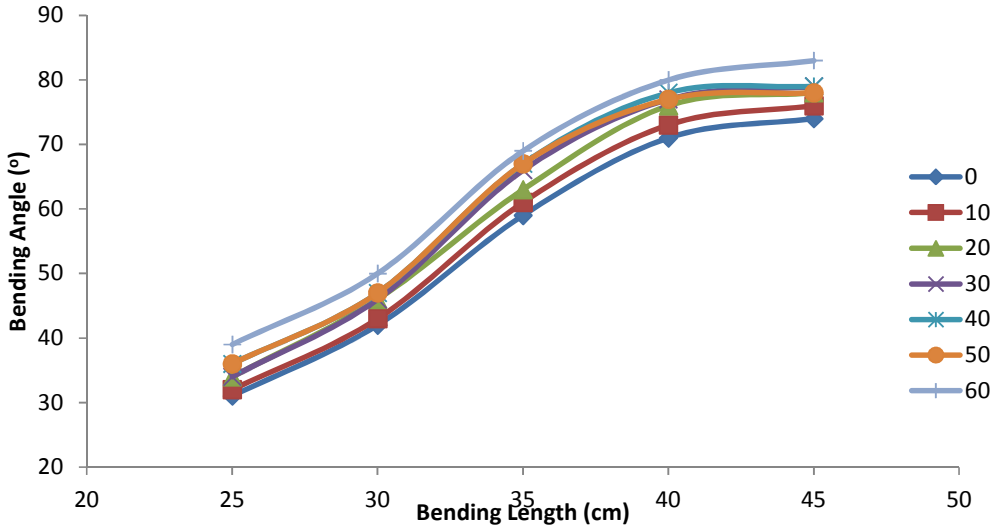

Fig. 4. Bending properties of fabric esterified with varying volume of *Jatropha curcas* seed oil (weft direction)

3.5.6 Effect of varying volume of *Jatropha curcas* oil on water imbibition of esterified cellulosic fabric

The property of absorbing moisture is a valuable feature of clothing materials [30]. It has a direct utility in keeping the skin dry, cause the fabric to act as a heat reservoir, protect the body from sudden changes of external conditions. The uptake or absorption of water by the substance without forming a solution is called imbibition.

Many biological substances like cellulose, starch and some proteins have the potential to imbibe moisture [35]. The affinity between the absorbent and the liquid plays a significant role in moisture absorption of any substance. The result of this investigation is given in Fig. 7. Clearly, there was a decrease in water imbibition as the volume of

oil increased with exception of 50 cm^3 which recorded the lowest imbibition as the drying time increased. It is suspected that the formation of a single ester bond on the cellulose chain [1] may be responsible for this observation. The reduction in porosity due to increase in yarn twist may also be responsible for the observed reduction in water imbibition; hence the esterified fabric is slightly hydrophobic. By implication, this fabric will retain less moisture and would be less susceptible to microbial attack during storage or after being used in damp condition.

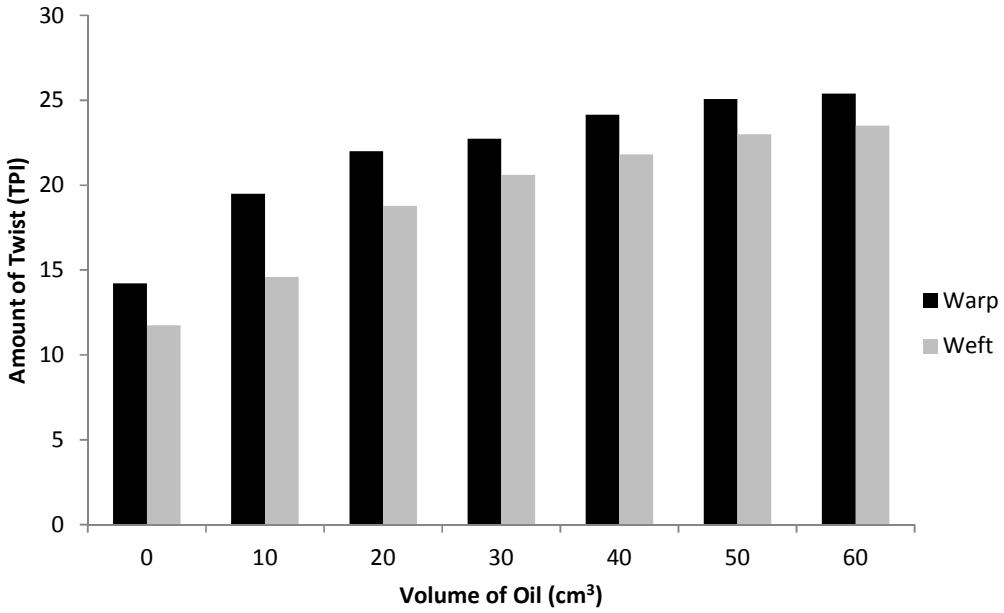

Fig. 5. Amount of yarn twist (z-direction) property of cellulosic fabric esterified with varying volume of *Jatropha curcas* seed oil (warp and weft directions)

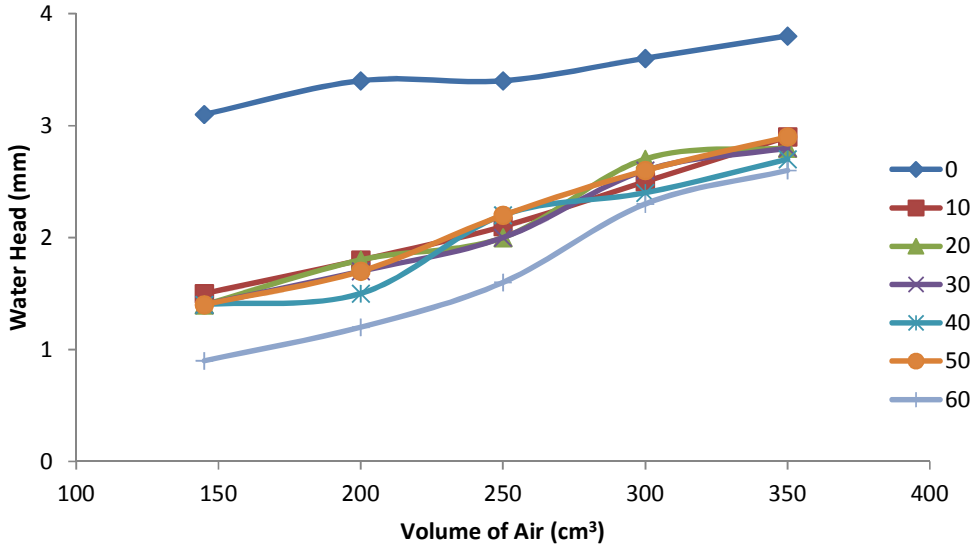

Fig. 6. Air permeability property of cellulosic fabric esterified with varying volume of *Jatropha curcas* seed oil

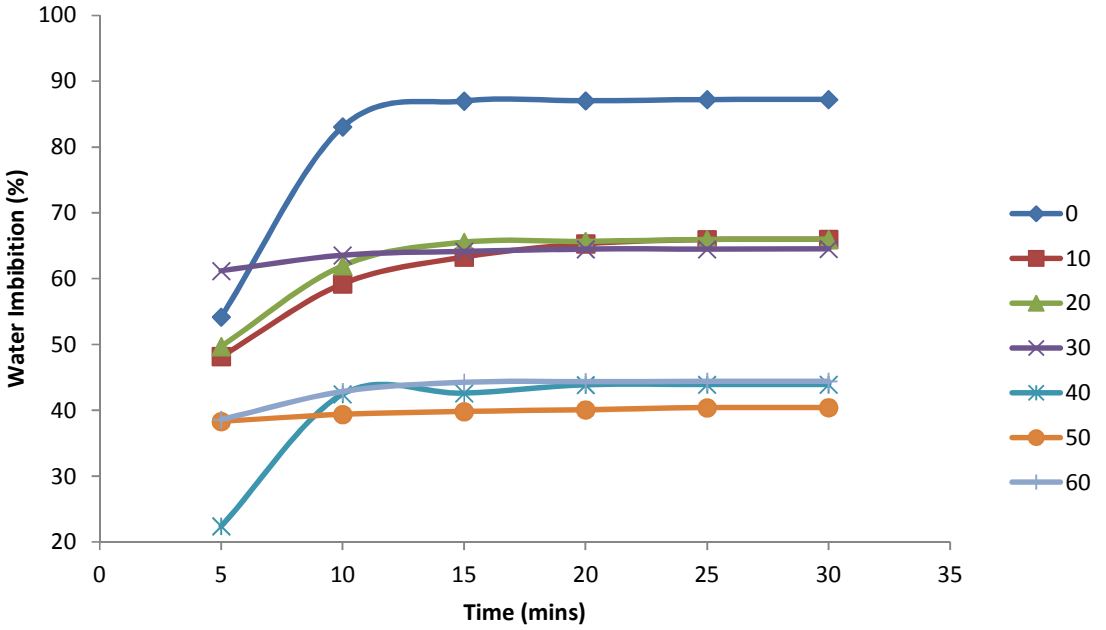

Fig. 7. Water imbibition property of cellulosic fabric esterified with varying volume of *Jatropha curcas* seed oil

4. CONCLUSION

Application of *Jatropha curcas* seed oil by varying the volume on cellulose based fabrics via esterification, remarkably improved the physicochemical properties of the fabrics. There were improvements in the creasing properties and yarn twist, more along the warp direction than weft direction as the volume of oil increased. These properties gave their optimum values at 50 cm^3 of oil. The 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 imbibition is an improvement; showing that the esterified fabric will make comfortable clothing during slightly low temperature seasons and will have better resistance to micro organisms and mildew during storage and after use in damp condition. In view of these, it can be correctly said that this research has made great contributions to knowledge because for the first time biodegradable organic seed oil like *Jatropha curcas* is used for the modification of physicochemical properties of cellulosic fabric through the process of esterification. Hence, this oil is recommended for textile finishing so that it can replace toxic and very expensive chemicals that has in decade been used in industries and also to safe our environments from unnecessary toxic effluents that enhance degradation.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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