

# SURFACE FUNCTIONALIZATION OF LINEN FIBER AND ITS DYE-FASTNESS ASSESSMENT USING (4-[(E)-(4-AMINO-2-HYDROXYPHENYL) DIAZENYL] BENZENE-1-SULPHONIC ACID AND 4-[(E)-(4-{(Z)-[4-(DIMETHYLAMINO) PHENYL] DIAZENYL}-2-HYDROXYPHENYL) DIAZENYL] BENZENE-1-SULPHONIC ACID

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## Abstract

Textile fibres find application in many fields, such as; Apparel manufacturing, Medical, Agriculture, Packaging, Civil construction, Shipping. Some of these fibres lack special characteristics required for some specific application without any additional treatment. Large scale production of linen fibre has been limited by the tendency of the fibre to absorb water, degradation by micro-organisms and sunlight. These factors ultimately lower the strength and the service life of the fibre. Hence, the need to improve the hydrophobicity of natural linen fibre before application. Poor dye-fibre interaction has been an area of constant interest in the textile industry. This has been expressed by bleeding, crocking, bleaching of fabric resulting to fading of the fabric. These shortcomings are sometimes due to poor treatment, manufacturing processes and customer handling. Natural linen fiber is an eco-friendly ligno-cellulosic fiber known for its strength, resistance to degradation, hydrophobicity when pointedly modified. In this research, chemical modification of scoured and bleached linen fibre by Acetylation, Methylation and Benzoylation was carried out. The chemically modified fibres were evaluated by techniques such as SEM and FTIR spectroscopy. The mechanical property (strength) was determined after each modification. The result obtained revealed an absorption band at  $3432\text{ cm}^{-1}$  -  $3450\text{ cm}^{-1}$  confirming the OH group in the linen fibre. The band at  $704\text{ cm}^{-1}$  indicated the presence of a benzene ring from benzoyl chloride. The chemical modification changed the morphology of the linen fibre for improved dye-fibre interaction. The chemically modified species were dyed and further subjected to a dye fastness test. The results revealed at  $4.33^a \pm 0.69$ ,  $5.00^a \pm 0.00$  and  $4.67^a \pm 0.58$  as against  $3.00^a \pm 1.00$ ,  $3.33^a \pm 0.58$  and  $2.67^a \pm 0.58$  for light fastness, wash fastness and rub fastness respectively when subjected to ANOVA analysis. In conclusion, benzoylated linen fibre was observed to most improved dye-fibre interaction.

**Keywords:** Linen Fiber, Acetylation, Methylation, Benzoylation, SEM, FTIR and Dye - fastness

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## Introduction

The textile industry is in constant search for new and innovative techniques to produce at a lower cost and with improved quality with a keen check on the impact on the environment (Anamaria and Cornelia, 2015). Surface modification/functionalization is usually done to improve some properties of textile fibre by impacting it with certain functional group. Modification processes are applied to improve adhesion, surface characteristics, to impart special properties and resistance to degradation (Anamaria and Cornelia, 2015). Linen fibre (a cellulosic fibre) is known for its strength, when weaved into a fabric, produces a lint free products. This eco-friendly lingo-cellulosic natural fibre is biodegradable and cool-natured. It also stands out among other fibres because of its good ventilation properties, lightweight and moisture adsorption properties of this cellulosic fibre leading to an increasing demand for linen fibres globally for various areas of application (Akbari *et al.*, 2020; Bright *et al.*, 2022). Global market for Bed and Bath linen in this COVID-19 crisis is estimated at US\$ 74.4 Billion in the year 2020 at compound annual growth rate (CAGR), this is projected to reach revised size of US\$ 99.7 Billion in 2027. Growing at a rate of 4.3% over 2020-2017 period (Global Industry analysts, 2021). These details have revealed a huge demand for linen fibre in the coming years.

Fabric from linen fibre are mainly used during summer or hot seasons, resulting from good air penetration, hence allowing maximum comfort during this season (Atalie *et al.*, 2021). Traditionally, it is known to possess poor spin property, poor anti-wrinkle properties, poor elasticity properties and feels hard when in contact with the skin (Atalie *et al.*, 2021). In contrast, high level of crystallinity and orientation is observed in linen fibre. In the microstructure of the flax fibre, larger area of crystalline region comparing with amorphous one was observed (Borsa *et al.*, 2016). This disadvantage is result in the poor dyability and the fastness property of linen. Therefore, this paper attempts to improve some of these shortcomings with a major look on the advantages of linen fibre to improve the performance on application in textile by chemical modification (acetylation, benzoilation and methylation) of the linen fibre. Several method of chemical modification to improve dyeability and dye-fastness of line has been attempted (Wang *et al.*, 2011; Oun and Rhim, 2015). Mesoporous range modification of linen fibre showed a decreased porosity under mild alkali/ultrasound condition with no substantial

alteration on the diameter of the fibre (Borsa *et al.*, 2016). Akbari *et al.* (2020) reported treatment using Ultrasonic environment to improve the adherence of reactive dyes to the linen fibre. This also accelerate the dyeing rate of the fibre by providing supplementary energy to the dye molecules interacting with the fibre surface. The report by Wang *et al* (2021) shows that increase in sodium hydroxide concentration resulted in improving dyeing rate of linen fibre. The result obtained when line fabric is screen-printed with pigments, reactive dyes and vat dyes reveals a good fastness to rubbing, washing and light. It was also observed that the tensile strength and resistance to abrasion remained unaltered (Tavcer *et al.*, 2011).

Flax (linen) fibre consists majorly of cellulose estimated to be 70-75% of the dry weight. Flax fibre also contains lignin, pectin, hemicellulose, and aromatic compounds in small amounts (Prabhu and Li, 2015). The most abundant polymer on earth is cellulose. It is renewable, biodegradable and non-toxic. It is with plant origin with a structural polymer that gives it its mechanical properties. Purification of cellulose from plant fibers is usually done by chemical treatments consisting of acid extraction, alkali extraction and bleaching (Dufrensne, 2013). Cellulose has a well-ordered structure and semicrystalline nature, consequently, nanoparticles can be extracted from this naturally occurring polymer using a top-down mechanically or chemically induced deconstructing strategy (Dufrensne, 2013). The well-ordered structure of natural fibers is reason for the unique strength and high performance properties of different species of plants (Dufrensne, 2013). The uniqueness in properties of linen fibre being a vegetable fibre, with good tensile strength, mechanical strength, hydrophobicity and dyeability resulting from the abundance of cellulose as a constituent is the major drive toward the choice of linen fibre as the fibre for modification (Prabhu and Li, 2015; Dufrensne, 2013). Atalie *et al*, (2021), reported chemically modified linen fibre by acetylation only and find out that acetylation improved all th fibre properties and spinnability.

There is a constant need to improve the quality of clothing available to users, the information available on the effect of methylation and benzoilation on linen fibre and also, the fact that natural linen fibre is an eco-friendly ligno-cellulosic fibre known for its strength, resistance to degradation, hydrophobicity when pointedly modified. Hence, the study aimed to improve the hydrophobicity of scoured and bleached linen fibre by acetylation, methylation and benzoilation.

## Materials and Methods

### Materials

FTIR (Cary 630 Agilent technologies USA series) was used to monitor the functional group chemically modified linen fibre, SEM (Phenom Pro X) was used to study the surface morphology of the linen fibre, Tensile Strength Machine (Instron Universal tensile machine) was used to determine the tensile strength of the linen fiber. The glasswares used in this research were soaked and washed in a mixture of detergent and dilute  $\text{HNO}_3$ , Then, rinsed and dried at  $105^\circ\text{C}$ .

### Pre-treatment of linen fibre

#### Scouring

The scouring was done with little modification of method according to : A was  $\text{NaOH}$  (0.2 g) dissolved in distilled  $\text{H}_2\text{O}$  (100 ml), B was  $\text{Na}_2\text{CO}_3$  (0.1 g) dissolved in distilled  $\text{H}_2\text{O}$  (100 ml) and C was surfactant (0.2 g) dissolved in distilled  $\text{H}_2\text{O}$  (100 ml). A, B and C (equal volume) was mixed and linen fibre (1 g) was boiled in 30 ml of the mixture in a 250 ml beaker at  $95^\circ\text{C}$  using heating mantle. The 30 ml mixture (A, B and C) was allowed to boil for about 30 min. and replace with another 30 ml of the mixture when the volume reduced to about 5 ml. This was repeated for another 1 h with replacement of the mixture when reduced for proper scouring. The fibre was removed and neutralized in acetic acid (0.5 %) and severally washed in distilled  $\text{H}_2\text{O}$  and finally dried in open air for 12 h.

#### Bleaching

Sodium hypochlorite (50 ml, 5 % v/v) in a 250 ml beaker was mixed with  $\text{Na}_2\text{CO}_3$  (50 ml, 5 % g/v). The pH was adjusted to pH 9.5 using acetic acid and  $\text{Na}_2\text{CO}_3$ . The scoured fibre was then charged into the mixture for bleaching for about 1 h with occasional agitation. Antichlore treatment was done using 0.5 ml acetic acid to neutralize the base. The fibre was thereafter washed repeatedly with distilled water and then dried in open air for 2 h.

### Acetylation of linen fibre (Acetylation of cellulose to form cellulose acetate)

The linen fibre consisting majorly of cellulose was pre-treated by scouring and bleaching and then modified chemically by treating it with acetic anhydride with little modification of method according to Vardhini *et al.* (2016): The de-waxed fibre (0.5 g) was immersed in 10%  $\text{NaOH}$  solution for 1 h at  $30^\circ\text{C}$ , and then rinsed with distilled  $\text{H}_2\text{O}$ .

The fibre was then transferred into a separate beaker containing glacial acetic (0.2%) acid for 1 h at  $30^\circ\text{C}$ , it was decanted and soaked in acetic anhydride (7.5 ml) containing two drops of concentrated  $\text{H}_2\text{SO}_4$  for 10 min. at  $30^\circ\text{C}$ . the fibre was the removed washed severally with water and dried in open air.

### Methylation of linen fibre (methylation of cellulose methylcellulose)

The linen fibre consisting majorly of cellulose was pre-treated by scouring and bleaching and then modified chemically by treating it with acetic anhydride with little modification of method reported by Viera *et al.* (2007); Kumar *et al.* (2012), with some modification as shown: Chemically pre-treated fibre (0.5 g) was mercerized by using a 50%  $\text{NaOH}$  for 1 h at room temperature. The excess of  $\text{NaOH}$  was squeezed out and acetone (9 ml) was added as a solvent. Dimethyl sulphate (3 ml) was added drop-wise and the reaction was allowed to proceed at  $50^\circ\text{C}$  with gentle and constant stirring. After 1 h and 30 minutes of the reaction, the system was filtered and fresh reactants (DMS and acetone) were added, maintaining the same previous proportions for the next 5 h of reaction. At the end of the reaction, the material was neutralized by 10% acetic acid solution, filtered on a sintered crucible, and then washed with acetone. The methylcellulose produced was dried in an oven at  $50^\circ\text{C}$  for 6 h.

### Benzoylation of the fibre

The benzoylation was done by submerging (1 g) of the bleached fibre in 18%  $\text{NaOH}$  for 30 min and 10%  $\text{NaOH}$  in 50 ml of benzoyl chloride for another 30 min followed by rinsing in conc. Ethanol for 1 hr to remove the benzoyl chloride and it was washed severally with distilled  $\text{H}_2\text{O}$  and dried at  $80^\circ\text{C}$  for 24 h.

### Dyeing of Linen Fibre

The linen fibre was dyed according to the method reported by Polya *et al.* (2015), 2% of the synthesized dyes were applied on weight of fibre. The solution of the dyes was formed by the dye in  $\text{H}_2\text{O}$ . 10% sodium sulphate and 4% acetic acid of 30% concentration was added to the solution  $35 - 40^\circ\text{C}$ . Then the previously wetted linen fibre (1 g) was dipped into the dye bath and dyed for 30 min at this temperature. The dye bath was heated to boiling for 30 min. the fibre was removed after dyeing was completed, thoroughly rinsed with cold running water and dried at room temperature.

## Result and Discussion

### Functional Group Characteristics Grey linen fibre

Curve 1 in Figure 1 reveals a characteristic broad absorption bands at  $3272.6\text{ cm}^{-1}$  for stretching vibration of the hydroxyl group in cellulosic linen fibre. This was not in variance with the report of (Poletto *et al.*, 2011; Abdellatif *et al.*, 2020). This peak includes intra-molecular hydrogen bond vibrations in cellulose (Turki *et al.*, 2018). The band at  $2914\text{ cm}^{-1}$  is possibly attributed to CH stretching vibration of all hydrocarbon constituent in cellulose. This was not in variance with the work done by Poletto *et al.* (2011). This possibly indicate the presence of waxes and other impurities which washed off after scouring resulting to the disappearance as shown in the absorption band in curve. 2 as indicated in Figure 1. The FTIR absorption band at  $1051\text{ cm}^{-1}$ , was for C-O bond that confirmed the presence of OH group in the Cellulose structure. This also was in conformity with the work done by Diana *et al.* (2011).

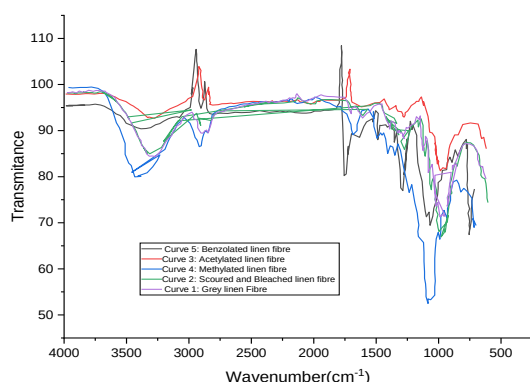


Figure 1. FTIR bands for modified linen fibre

### Bleached linen fibre

The scouring and bleaching of the linen fibre were to remove wax and colourants in the fibre. Hence, the main difference in the spectra in the adsorption band for curve. 1 and 2 as shown in Figure 1, when compared is the disappearance of the doublet band (aldehyde C-H bond) and appearance of singlet band at  $2914\text{ cm}^{-1}$  which is characteristic of  $\text{sp}^3$  C-H bond. This agreed with the work done by Titok *et al.* (2010). The curve. 2 also reveals a characteristic broad absorption bands at  $3272.6\text{ cm}^{-1}$  for stretching vibration of the hydroxyl group in cellulosic linen fibre (Poletto *et al.*, 2011). The FTIR absorption band at  $1051\text{ cm}^{-1}$ , was for C-O bond that confirmed the presence of OH group in

the Cellulose structure. This also was in conformity with the work done by Diana *et al.* (2011).

### Acetylated linen fibre

The band at  $3,275\text{ cm}^{-1}$  shown in the curve. 3 revealed the presence of hydroxyl group. The intensities of the band for OH group decreased due to the acetylation, replacing some of the OH group with the acetyl group (Mohebbi, 2008). This is an indication that the number of OH groups was decreased due to the substitution of the acetyl groups during the acetylation of the cellulose in the linen fibre. Many reports have shown that acetylation improves hydrophobicity of a material (Rowell, 2010). The characteristic sharp absorption band at  $1638\text{ cm}^{-1}$  show the C=O stretching vibration confirming the presence of C=O group in the acetylated fibre. The FTIR absorption band at  $1051\text{ cm}^{-1}$ , was for C-O bond that confirmed the presence of OH group in the Cellulose structure. This also was in conformity with the work done by Diana *et al.* (2011).

### Methylated linen fibre

The band at  $3432\text{ cm}^{-1}$  shown in the curve. 4 revealed the presence of hydroxyl group. It was observed that the characteristic absorption band intensity decreased attributed to the stretching vibration of hydroxyl group (OH) of the cellulose, which was partially substituted by methyl ( $\text{CH}_3$ ) group during the methylation reaction. It was also observed that there was a significant increase in the intensity of band at  $2,899\text{ cm}^{-1}$  which is characteristic of  $\text{sp}^3$  bond (Da Crus *et al.*, 2011). The FTIR absorption band at  $1,051\text{ cm}^{-1}$ , was for C-O bond that confirmed the presence of OH group in the Cellulose structure. This also was in conformity with the work done by Diana *et al.* (2011).

### Benzoylated linen fibre

The band at  $3339\text{ cm}^{-1}$  shown in the curve. 5 revealed the presence of hydroxyl group. The characteristic sharp absorption band at  $1718\text{ cm}^{-1}$  shown in curve. 4 revealed the presence Benzoyl groups in the cellulose structure due to the C=O stretching vibration. Another absorption band at wave number  $1021\text{ cm}^{-1}$  confirmed the presence of Ether linkage (C-O-C). The characteristic absorption band at  $704\text{ cm}^{-1}$  confirmed the presence of benzene ring in the Benzoyl Chloride used for treatment.

### SEM Result for Linen fibre

#### SEM analysis

Morphological studies of the fibre showed that the acetylation, benzoylation, methylation treatment of linen fibre improved the surface

properties, resulting to a higher tensile strength than that of the untreated linen. This was not in variance with the work done by Kamathe and Nennehalli, (2021). The chemical modification changed the smooth to surface of the fibre a rough irregular surface, compressed to permeable, wide to narrow, spherical and cylindrical. This was in agreement with the studies made by Gasiorowski *et al.* (2012). It was observed that the dyes adhere well to the fibres after their modification, and this is similar depending on the form of pre-treatments on comparing the SEM micrographs. Although after the dye fastness test, sample M<sub>m</sub> and D<sub>m</sub> gave a best fastness when dyed with 4-[(E)-(4-amino-2-hydroxyphenyl) diazenyl] benzene-1-sulphonic acid (Dye 1) and 4-[(E)-(4-{(Z)-[4-(dimethylamino) phenyl] diazenyl}-2-hydroxyphenyl) diazenyl] benzene-1-sulphonic acid (Dye 2)

#### Surface characterisation of linen fibre

SEM images of untreated and treated linen fibres are shown. The Grey linen fibre as shown in Figure 2 clearly reveals a smooth surface, oils and waxes and other impurities are suspected to be responsible for small particles attached to the surface as shown in the SEM image. The smooth surfaces noticed in the micrograph as shown in Figure 3 and Figure 4 explained the removal of waxes and other similar impurities. This agrees with research done by Abdellatif *et al.* (2020). Treatment with acetic anhydride probably showed the surface was covered with crystals of acetic anhydrides as shown in Figure 5. In case of benzoyl chloride, the fibre bundles are clean and quite well separated as shown in Figure 6. It can be also observed that in Figure 7, the surface of the fibres is broken. This may be as a result of excess reagent used the fibres after methylation treatment the fibre look jagged and show very rough and narrow surface Figure 8, present the measured tensile strength and elongation of untreated, scoured, acetylated, benzoylated and methylated linen fibre. Mechanical properties (e.g., tensile strength) and the corresponding Young's modulus were measured using Universal Instron testing Machine, Model No: 3369. Table 1, expresses the tensile strength, elongation at break and Young's Modulus of some natural fibres. Variations in tensile strength, elongation at break and Young's Modulus depend on the type of treatment, load, and length of fibres. The Grey (Black) fibre exhibited a high uniaxial tensile stress of 69.95, elongation at break 14.51 and Young's modulus of 712.85 this might be due to the presence of non-cellulosic materials such as lignin and hemicellulose in the cell wall. On treating the fibre with alkaline the uniaxial tensile stress 89.77, elongation at break 7.54 and its Young's modulus

1040.62 exhibited by the scoured fibre, showed an increasing trend from the grey fibre, although, corresponding increases for other treatment conditions under ambient condition were less remarkable. The scouring treatment has improved the tensile properties of the linen fibre significantly and therefore exhibited highest tensile strength as compared to the other treated linen fibres. The removal of its impurities led to the increment of resistance and energy required to break the specimen. By contrast, methylated linen demonstrated less tensile strength under all loads. This reveals that alkali treatment significantly improves the mechanical properties of natural fibres by modifying their crystalline structure, as well as by removing certain weak components like hemicelluloses, pectin, wax, oils and lignin from the fibre structure as also reported by Cruz and Fanguier (2016), giving way to the enhancement in strength. Also, as the leaches out due to the alkali treatment close packing of cellulose chain in fibre takes place owing to the release of internal strain leading to higher tensile strength and elongation at break of the alkalinised fibre (Roy *et al.*, 2012)

The acetylated fibre, gave a uniaxial tensile stress of 42.70, elongation at break 13.38 and a Young's modulus of 271.75 this showed a decrement as compared to the scoured fibre, although the acetylation of some of the hydroxyl groups on the surface of fibers, consequently brought about reduced moisture absorption in the fiber cell wall with good wetting capability of the fibres, although this weaken the fibre resistance and toughness these observations are qualitatively similar to those reported by Akhatara *et al.* (2016).

The result for the benzoylated linen fibre, shows the value of uniaxial tensile stress 53.00, elongation at break 29.06 and its Young's modulus 140.47. Meanwhile, the maximum value of uniaxial tensile stress for methylated linen fibre was achieved at 5.13, elongation at break 20.35, and Young's modulus 23.78, this is in agreement with values for elongation and Young's Modulus for flax, agave sisal and others as shown in Table 1. The is very low when compared with that of scoured fibre, where the Young's modulus of tensile strength was about 110% higher than that of the methylated fibre. Notably, this poor tensile strength exhibited by the methylated linen fibre may be due to the presence of the harsh sulphate acid used during the treatment as cellulose is sensitive to acidic medium, thereby attacking the main structural components of the fibres, resulting in the wearing out of tinny fibril on the surface of the fibre. This in turn leads to further weakening of fibre strength and the tensile strength decreases.

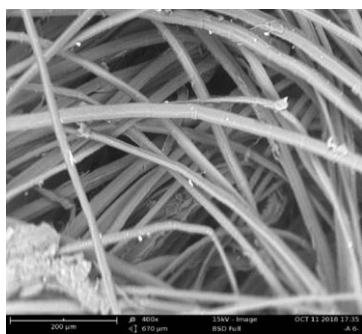


Figure 2. SEM Result for Grey Fibre

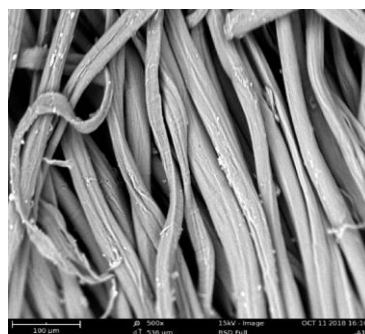


Figure 5. SEM Result for Acetylated Fibre



Figure 3. SEM Result for Scoured Fibre

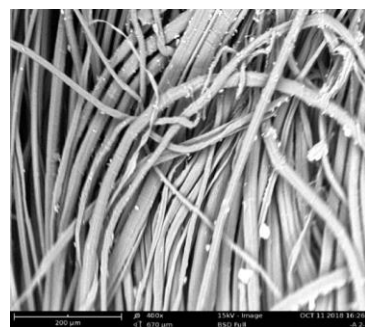


Figure 6. SEM Result for Benzoylated Fibre

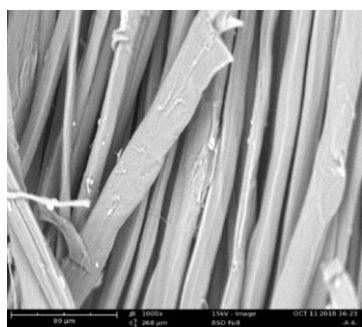


Figure 4. SEM Result for Bleached Fibre



Figure 7. SEM Result for Methylated Fibre

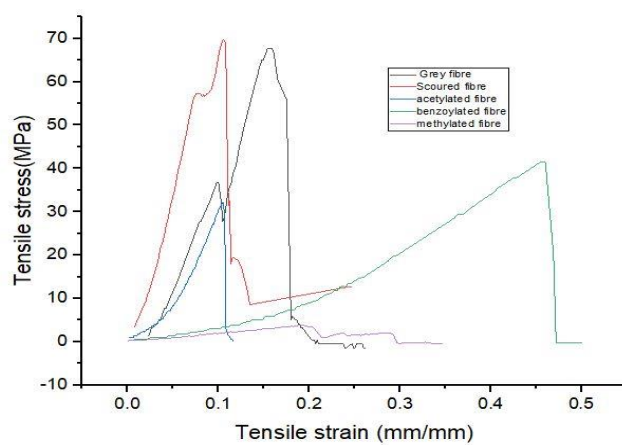


Figure 8. Stress Strain curve of Grey and Modified Fibre

**Table 1. Mechanical properties of natural fibers (adapted from Tavares *et al.*, 2020).**

|                                                       | Tensile Strength (MPa) | Elongation at Break (%) | Young's Modulus (GPa) |
|-------------------------------------------------------|------------------------|-------------------------|-----------------------|
| Jute<br>( <i>Corchorus capsularis</i> )               | 393.0–773.0            | 1.5–1.8                 | 13.0–26.5             |
| Flax<br>( <i>Linum usitatissimum</i> L.)              | 345.0–1100.0           | 1.3–10.0                | 27.6                  |
| Hemp<br>( <i>Cannabis sativa</i> )                    | 550.0–900.0            | 1.6                     | 30.0–70.0             |
| Kenaf<br>( <i>Hibiscus cannabinus</i> )               | 295.0–1191.0           | 3.5                     | 53.0                  |
| Ramie<br>( <i>Boehmeria nivea</i> )                   | 348.0–938.0            | 1.2–8.0                 | 44.0–128.0            |
| Cotton<br>( <i>Gossypium</i> sp.)                     | 264.0–800.0            | 7.0–8.0                 | 5.5–12.6              |
| Milkweed<br>( <i>Calotropis gigantea</i> )            | 381.0                  | 2.1                     | 8.2                   |
| Coir<br>( <i>Cocos nucifera</i> )                     | 131.0–175.0            | 15.0–25.0               | 4.0–6.0               |
| Kapok<br>( <i>Ceiba pentandra</i> )                   | 90.0–95.0              | 1.8–4.2                 | 4.0                   |
| Sisal<br>( <i>Agave sisalana</i> )                    | 500.0–800.0            | 2.0–25.0                | 9.4–22.0              |
| Pineapple<br>( <i>Ananas comosus</i> )                | 170.0–1627.0           | 2.4                     | 60.0–82.0             |
| Agave<br>( <i>Agave americana</i> L.)                 | 430.0–580.0            | 3.0–4.7                 | 13.2                  |
| Banana<br>( <i>Musa sapientum</i> )                   | 529.0–914.0            | 3.0                     | 27.0–32.0             |
| Sugarcane bagasse<br>( <i>Saccharum officinarum</i> ) | 20.0–290.0             | 1.1                     | 17.0                  |
| Bamboo<br>(Bambusoideae)                              | 140.0–230.0            | --                      | 11.0–17.0             |
| Wheat<br>( <i>Triticum</i> sp.)                       | 275.0                  | --                      | 4.5–6.5               |
| Silk<br>( <i>Bombyx mori</i> )                        | 650.0–750.0            | 18.0–20.0               | 16.0                  |
| Wool<br>( <i>Ovis aries</i> )                         | 120.0–174.0            | 25.0–35.0               | 2.3–3.4               |

**Table 2. Fastness property of Linen Fibre**

| TEST<br>SAMPLE | Light Fastness |    |   | Wash Fastness |   |    | Rub Fastness |    |    |
|----------------|----------------|----|---|---------------|---|----|--------------|----|----|
| U <sub>F</sub> | 3              | 3+ | 3 | 3+            | 4 | 3  | 2+           | 3  | 3+ |
| M <sub>A</sub> | 3              | 5  | 4 | 5             | 4 | 3  | 3+           | 4  | 4  |
| M <sub>B</sub> | 5              | 4  | 5 | 5             | 5 | 4+ | 5            | 4+ | 5  |
| M <sub>M</sub> | 3+             | 4  | 5 | 4+            | 5 | 4  | 4            | 5  | 4  |
| D <sub>A</sub> | 5              | 4+ | 4 | 4             | 5 | 3  | 4            | 3  | 5  |
| D <sub>B</sub> | 5              | 5  | 4 | 5             | 5 | 5  | 5            | 4  | 5  |
| D <sub>M</sub> | 4+             | 4  | 5 | 3+            | 5 | 4  | 5            | 4+ | 3  |

**Fastness Test Result**

**Keys:** U<sub>F</sub> = Untreated fibre, M<sub>A</sub> = Monoazo Acetylation, M<sub>B</sub> = Monoazo Benzoylated, M<sub>M</sub> = Monoazo Methylated, D<sub>A</sub> = Disazo Acetylated, D<sub>B</sub> = Disazo Benzoylated, D<sub>M</sub> = Disazo Methylated. Grey Scale Rating: 1- Poor; 2-Below Average; 3- Average; 4- Good; 5 Excellent; + -

**Fastness Properties of the fibres**

The mean fastness ratings of the fibre under different treatments are shown in Table 3.

Generally, the three samples exhibited similar behaviour to all the agencies except for U<sub>F</sub>. This was probably due to the similarities of dyestuffs and textile materials (linen) used, hence both samples possibly exhibited similar types of dye-fibre interactions (Jabar, 2014) and this is a function of fastness (Shan, 2020). Their overall mean fastness ratings can be considered being good based on a scale of 5 for light and other fastness test. The factors and mechanisms of fading of dyes were reported in literature that fibre fade easily when they

are dyed with dyes that are not meant for them (Forster *et al.*, 2017; Momodu *et al.*, 2020). The fastness properties of the fibre depreciated in detergent. This probably may be as a result of the chemicals used in the detergent production.

**Table 3. Statistical Analysis Result (One way Anova)**

| TEST SAMPLE    | LIGHT FASTNESS          | WASH FASTNESS           | RUB FASTNESS            |
|----------------|-------------------------|-------------------------|-------------------------|
| U <sub>F</sub> | 3.00 <sup>a</sup> ±1.00 | 3.33 <sup>a</sup> ±0.58 | 2.67 <sup>a</sup> ±0.58 |
| M <sub>A</sub> | 4.00 <sup>a</sup> ±1.00 | 4.00 <sup>a</sup> ±1.00 | 3.67 <sup>a</sup> ±0.58 |
| M <sub>B</sub> | 4.33 <sup>a</sup> ±1.00 | 4.33 <sup>a</sup> ±0.58 | 4.33 <sup>a</sup> ±0.58 |
| M <sub>M</sub> | 4.67 <sup>a</sup> ±0.58 | 4.67 <sup>a</sup> ±0.58 | 4.66 <sup>a</sup> ±0.58 |
| D <sub>A</sub> | 4.33 <sup>a</sup> ±0.58 | 4.00 <sup>a</sup> ±1.00 | 4.00 <sup>a</sup> ±1.00 |
| D <sub>B</sub> | 4.33 <sup>a</sup> ±0.69 | 5.00 <sup>a</sup> ±0.00 | 4.67 <sup>a</sup> ±0.58 |
| D <sub>M</sub> | 4.33 <sup>a</sup> ±0.58 | 4.00 <sup>a</sup> ±1.00 | 4.00 <sup>a</sup> ±0.00 |

### Light fastness

The Dye 2 gave a higher light fastness than the Dye 1 especially at the M<sub>B</sub> and D<sub>B</sub> row in the Table 2 (Otutu, 2012). This may also be attributed to the molecular structure of the dye, polarity, dye substituent and number of conjugated bonds that shielded the dye from radiant energy on azo chromophores, therefore, this effect conferred good light properties on the dyes.

### Wash fastness

The Table 2 shows that the Dye 2 dyes gave a better wash fastness than the Dye 1 (especially at the M<sub>B</sub> and D<sub>B</sub> row in the Table 2) which also gave a very good wash fastness property, this may be attributed to the additional conjugated bonds of the dyes. The dyeing period is also a factor, hence, once the dyes are in, migration out will be difficult, thereby increasing the fibre resistance to washing.

### Rub fastness

Both dyes gave almost entirely similar rubbing fastness as shown in Table 2. This may be traced to the molecular moiety of the dyes and fibre-dye covalent bonding according to Kamel *et al.* (2014).

### Statistical Analysis Result (One way Anova)

Sample M<sub>M</sub> as shown in Table 3 reveals the highest mean value at 4.67<sup>a</sup>. This appearing twice for light and wash fastness with dye 1, but there is no significant level of difference between the mean of the values obtained for light, Wash and Rub fastness. Sample D<sub>B</sub> also show the highest mean value at 5.00<sup>a</sup> for wash fastness. Rub fastness and light fastness at 4.67<sup>a</sup> and 4.33<sup>a</sup> respectively show values that is similar samples under the same conditions. The equal mean value for Sample D<sub>M</sub>,

D<sub>B</sub> and D<sub>A</sub> (4.33<sup>a</sup>) show that all these samples react to light almost the same way.

## Conclusions

This research article has been able to show how linen fibre is modified chemically to improve surface morphology, hydrophobicity and strength. Different treatments such as acetylation, methylation and benzylation were applied for the chemical modification of linen fiber to obtain a maximum dye-fiber interaction for improved dye-fastness of the chemically modified linen fiber. The SEM micrograph for benzyolated linen fiber indicated a high level of impacted hydrophobicity and strength, hence preparing the fiber for excellent dye absorption. Dyed Methylated linen fiber though exhibiting good dye fastness was noticed to cause cleavages of the fiber. This consequently reduced the strength of the fiber.

The results obtained after dyeing process revealed that the dyed benzyolated linen fibre using Dye 1 and Dye 2 gave excellent light, wash and rub fastness shown in M<sub>B</sub> and D<sub>B</sub> results

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