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Effect of Alkali Treatment on the Chemical Composition and Dyeability of Sisal Fibers

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Abstract

Recently, natural fibers have gained significant attention from many researchers due to the global demand for natural products with low environmental impacts. Sisal (*Agave Sisalana*), is among the important natural fibers with substantial potential to flourish due to their outstanding properties, including high strength, good absorbency, and abundant availability. However, utilization of these fibers is yet to be fully explored, probably due to their stiff and coarse nature, which limits the overall applications of this sustainable fiber. This study, therefore, investigated the effect of alkali treatment on the chemical composition and dyeability of sisal fibers as a way of adding value to the fiber and potentially broadening their applications. The findings of this study have shown that alkali treatment decreased significantly the amount of hemicellulose and lignin from 10.6% and 10.7% to 4.9% and 6.4%, respectively. However, the cellulose content slightly increased from 64.6% to 64.7%. The decrease in amounts of hemicellulose and lignin was thought to be due to their high sensitivity to the action of alkali. Furthermore, the chemical treatment improved the dyeability of sisal fibers when both reactive and vat dyes were used, resulting in fibers with higher levels of dye exhaustion and improved wash fastness properties. Overall, the results suggest that alkali treatment not only modified the surface structure of sisal fibers but also improved their dyeability with both reactive and vat dyes. This will contribute to expanding the practical applications of sisal fibers, making them more viable for use in sustainable textile production in future

Keywords: Sisal fibers, Alkali treatment, Reactive dyes, Vat dyes, Colorfastness

Introduction

Nowadays, the use of natural fibers is getting much attention as an alternative to petroleum-based synthetic fibers due to their low cost, biodegradability, renewability properties and good mechanical strength (Reddy et al. 2016, Aaditaa and Jahan 2018, Charca et al. 2022). In addition, the necessity to preserve the environment and the increasing depletion of petroleum resources has led to increased use of natural fibers in various applications, including textiles, polymer composites, geotextiles, and building materials (Reddy et al. 2013, Rasigha et al. 2015, Reddy et al. 2016). Among the natural fibers that have received great attention is the sisal fiber, a plant species of *Agave sisalana* that is indigenous

to the southern region of Mexico but widely cultivated in many tropical parts of the world (Anandjiwala and John 2010, Sharma and Varshney 2012). Tanzania is among the large sisal-growing countries, with other main producers being Brazil, China and Kenya (FAO, 2017).

Sisal is an interesting leafy fiber characterized by high tensile strength, moisture absorbency and affinity to various dyestuffs. In addition, the fiber is eco-friendly and biodegradable in nature, properties that make the sisal fiber attractive in the field of textiles and other end usages worldwide (Agrawal and Rastogi 2018). In Tanzania, more than 70% of its raw sisal is sold outside the country, while 30% is mainly used to produce non-textile products such as ropes, agricultural twines, mats, and handbags (Fednand et al. 2022). Globally, sisal fibers are also increasingly used as reinforcement in composites upon chemical modifications (Hajiha et al. 2014, Rasigha et al. 2015). Despite these promising attributes the full potential of sisal fibers, little has been done to enhance the utilization of sisal fibers in textile applications, and particularly in clothing and apparel manufacturing. This is probably due to its stiffness and coarseness nature, resulting from the relatively low cellulose content (43-78%) and high content of the non-cellulosic substances such as hemicellulose (10-14%) and lignin (4-12%) contents (Mwaikambo 2006, Mbugua 2009, Benítez-Guerrero et al. 2017). For that matter sisal fibers are categorized as 'hard fibers' (Kabir et al. 2012). The presence of non-cellulosic substances is particularly reported to negatively influence the processing of sisal fiber, including spinning, weaving, as well as dyeing, printing and finishing. For instance, the non-flexible nature of sisal fibers prevents the possibility of producing fine sisal yarns on traditional cotton spinning systems (Zwane et al. 2019). This in turn, limits the applicability of sisal fibers in producing value-added textiles. Therefore, for many applications, it may be necessary to chemically modify the fibers to reduce the amount of non-cellulosic substances, thus improving the fiber properties, including enhancing the affinity of fibers for chemicals and dyes (Ding et al. 2014, Reza et al. 2014, Arju et al. 2015).

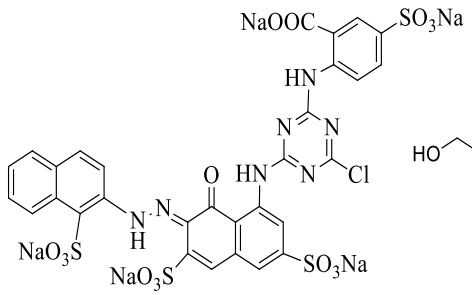
Different sisal fiber modification processes have been conducted to improve the mechanical and structural properties of sisal fibers thereby adding value to this sustainable fiber. Alkali treatments have particularly been used to enhance the technical applications of natural fibers including sisal fibers, particularly for composite reinforcement (Oladele et al. 2010, Wang et al. 2011, Deng et al. 2013, Hajiha et al. 2014, Rasigha et al. 2015). The resulting natural fiber reinforced composites find application in wide areas such as construction, automotive, biomedical, defence and sport (Mahmud et al. 2021). However, no attempt, according to our knowledge, has been made to establish the effect of alkali treatment on the dyeability of sisal fibers for the purpose of improving its color quality and appearance in general. In addition, relatively few literatures have reported on the effect of alkali treatment on the chemical composition of sisal fibers. It is in this regard that the current study investigated the effect of alkali treatment on the chemical composition and the dyeing properties (exhaustion and washing fastness properties) of sisal fibers using varieties of reactive and vat dyes.

Materials and Methods

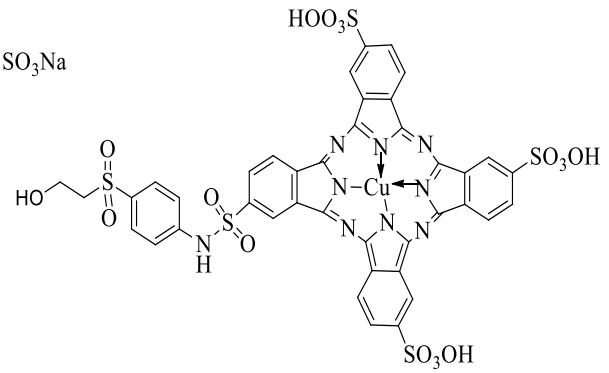
Materials

Decorticated sisal fibers used in this study were kindly supplied by 21st Century Holdings (MeTL) in Dar es Salaam, Tanzania. Three commercial reactive dyes, namely; C. I. Reactive Red 31 (RR31), C. I. Reactive Blue 21 (RB21) and C. I. Reactive Black 5 (RB5), as well as one vat dye, namely C. I. Vat Black 25 (VB25) were kindly provided by 21st Century Textile Mills in Morogoro, Tanzania. The chemical Structures of the dyes are presented in Figure 1. Other chemicals used, including sodium hydroxide, sulphuric acid, glacial acetic acid, sodium carbonate, sodium silicate, sodium lauryl sulphate, magnesium chloride, ethanol, hydrogen peroxide and potassium hydroxide were of analytical grade obtained from

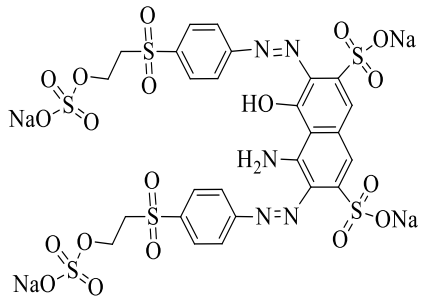
different suppliers within and outside Tanzania.



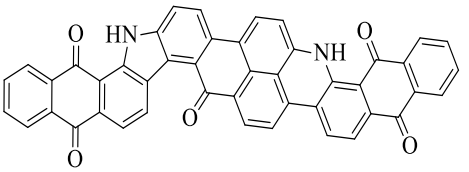
C.I. Reactive Red 31



C.I. Reactive Blue 21



C.I Reactive Black 5



C.I Vat Black 25

Figure 1: Chemical structures of the dyes used in this work.

Methods

Preparation of Sisal Fibers

Sisal fibers were pre-treated by scouring and bleaching techniques in order to remove any natural and added impurities using a procedure described in our previous paper (Fednand et al. 2022). Both scouring and bleaching were carried out in a Mathis Labomat beaker dyeing machine using stainless steel pots. Scouring was done by treating the fibers at 90 °C for 60 minutes in a solution containing 10 g/L of wetting agent, 5 g/L of sodium hydroxide and 1 g/L of sodium silicate using a material-to-liquor ratio of 1:10. Scoured fibers were washed thoroughly in running water and neutralized with 1% acetic acid for 10 minutes followed by rinsing using distilled water and air-drying.

Scoured fibers were then bleached for 60 minutes at 90 °C in a solution containing 4.5 g/L hydrogen peroxide, 7 g/L sodium silicate, 1.2 g/L Sodium hydroxide and 1.8 g/L Sodium carbonate with a liquor ratio of 1:10. Bleached fibers were washed with tap water, neutralized with 1% acetic acid for 10 minutes followed by rinsing with distilled water and air-drying.

Alkali Treatment of Sisal Fibers

The alkali treatment was chosen due to its well-documented effectiveness in removing non-cellulosic components such as hemicellulose and lignin from natural fibers. The alkali treatment of the scoured and bleached sisal fibers was done following a procedure described in our previous paper (Fednand et al. 2022). The treatment was done in a Mathis Labomat beaker dyeing machine using optimum parameters, where sisal fibers were treated for 2 hours in 5 g/L NaOH and at 110 °C with a liquor ratio of

1:10 Treated fibers were then washed thoroughly in running water, neutralized in 1% acetic acid for 10 minutes followed by water rinsing and air-drying.

Dyeing of Sisal Fibers

To determine the effect of treatment on dyeing, the alkali-treated fiber samples were dyed with four commercial synthetic dyes namely; C. I. Reactive Red 31 (RR31), C. I. Reactive Blue 21 (RB21), C. I. Reactive Black 5 (RB5) and Vat black (VB25) dyes. Dyeing was carried out in Mathis Labomat beaker dyeing machine using a liquor ratio of 1:30. For comparison purposes, dyes were also applied on untreated sisal fibers.

Reactive dyes were applied on sisal fibers at 2% (o.w.m.) depths of shade using an exhaustion method described by Arya (2013) with some modifications as presented in the schematic Figure 2. Sisal fibers were first immersed in the dyeing liquor, initially set at 50 °C, followed by the addition of sodium chloride (70 g/L) and sodium carbonate (20 g/L). The dyeing process was then carried out for 75 min. Finally, the dyed samples were then washed thoroughly in warm water followed by cold water washing and air-drying.

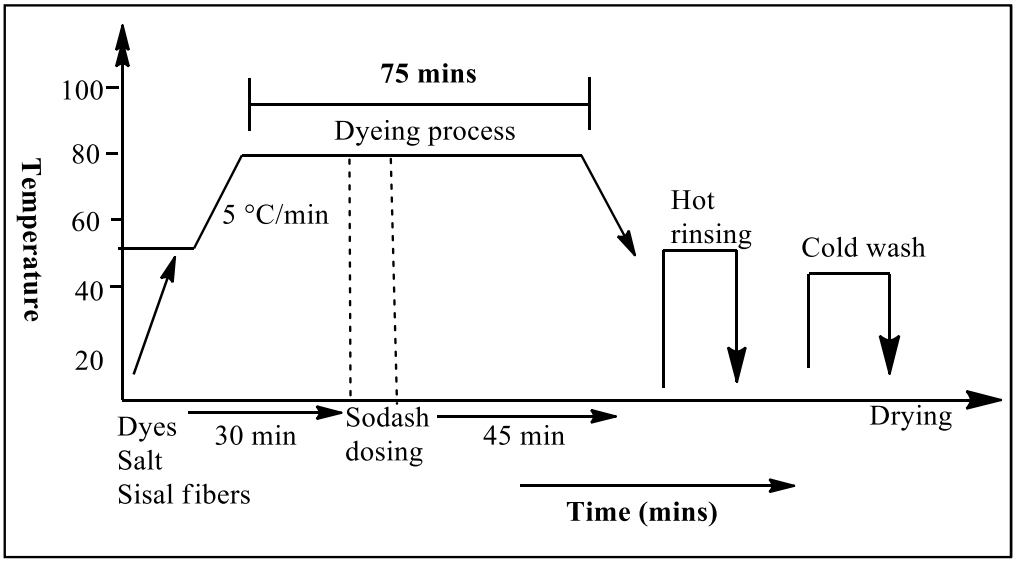


Figure 2: Dyeing procedure for reactive dyeing of sisal fibers.

C. I. Vat Black 25 was applied on sisal fibers at 5% (o.w.m.) using a method reported by Arya (2013) with some modifications. Initially, the dye was dissolved in 100 mL distilled water at 40 °C followed by adding 15 g/L sodium hydroxide and 15 g/L sodium dithionite for vatting to occur. Sisal fibers were then immersed in a blank bath containing 3 g/L sodium hydroxide and 5 g/L sodium dithionite before adding half of the reduced vat dye into this blank dye bath and allowed to stand for 10 minutes. The remainder of the vatted dye and 15 g/L of sodium chloride were then added into the dyeing pots, and the dyeing process continued for a further 60 minutes at 50 °C, as shown in Figure 3. The dyed samples were then rinsed thoroughly in warm water, followed by cold water washing and air drying.

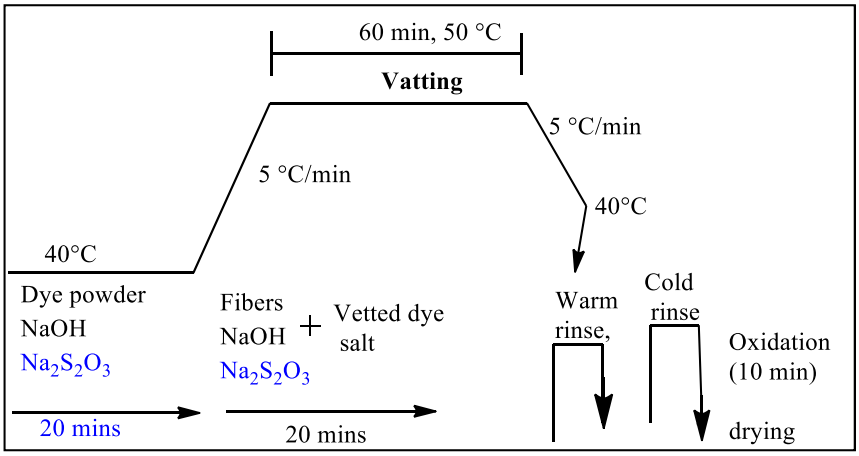


Figure 3: Dyeing procedure for vat dyeing of sisal fibers.

Determination of Chemical Composition of Sisal Fibers

The percentage weight of α-cellulose, hemicelluloses and lignin existing in treated and untreated sisal fibers was estimated through the standard method recommended TAPPI standard method (1971). The percentage of holocellulose in the treated and untreated sisal fibers was estimated through the method reported by Kataoka et al. (2022). The hemicellulose content was calculated as the difference between the holocellulose and α-cellulose content.

Determination of Extent of Dye Exhaustion (%E)

The extent of dye uptake by the sisal fibers was obtained by monitoring the changes in the concentration of the dye bath in the exhaustion stages according to the method described by Yohana et al. (2018). About 0.5 mL of the liquor was sampled from the dye bath before and after the dyeing process and was diluted using distilled water to allow examination of the liquors using a UV-Vis Spectrophotometer. The absorbance of the original sample and that of the exhausted dye bath were determined on a UV-VIS Spectrophotometer (SPECORD 210-Germany) at the maximum wavelengths of the respective dyes. The extent of dye exhaustion (% E) was then determined using Equation 1 (Kabir et al. 2017, Yohana et al. 2018).

$$\% E = \frac{A_i - A_f}{A_i} \quad (1)$$

Where: A_i is the initial absorbance of the dye bath before dyeing and A_f is the final absorbance of the dye in the bath after dyeing.

Assessment of Wash fastness properties of Dyed Fibers

Color fastness to washing of the dyed fibers was done to determine how permanent the color was on the fibers. Testing was performed by using Tanzania Standard, TZS 24:1979 – Test for color fastness to washing (TBS, 1979), where dyed fibers were immersed in a solution containing 2 g/L sodium carbonate and 5 g/L detergent using a liquor ratio of 50:1. Treatment was done for 2 hours which was followed by thoroughly washing the samples in clean water and finally air-dried. Change in shade of the treated samples was evaluated using a grey scale and graded 1 to 5, with 5 indicating excellent and 1 being poor fastness to washing.

Results and Discussion

In this study sisal fibers were treated in optimal alkali treatment parameters. The effect of the treatment on chemical composition and dyeability of the sisal fibers

were established, and the results are hereby presented and discussed.

Effect of Alkali Treatment on Chemical Composition of Sisal Fibers

The quality of the fiber is greatly affected by its chemical components such as cellulose, hemicelluloses and lignin (Aaditaa and Jahan 2018). The chemical composition of fibers determines the physical-chemical properties of the resultant fibers. For instance, high amount of cellulose in the fibers contributes to the good mechanical properties and hydrogen bonding with other molecules, such as reaction with dyes (Sari et al. 2018). However, high lignin content triggers fiber rigidity and negatively affects the fiber structure, properties, and morphology (Indran et al. 2014). In this study, the effect of alkali treatment on the chemical composition of sisal fibers was studied, and the results are summarized in Table 1. It is clear from the table that the optimal alkali treatment parameters used in this study had minimal effect on the cellulose content of the sisal fibers. This is because, compared to untreated fibers, the amount of cellulose was observed to increase slightly upon alkali treatment. This may be due to damage to some part cellulose molecules during the treatment. On the other hand, the increase in weight percent of cellulose is associated with the removal of the amorphous component of lignin, wax, and hemicellulose as a result of fiber treatment by sodium hydroxide. These findings are in line with related studies on other cellulosic fibers; Vardhini et al. (2016) noted an increase in cellulose content from 67.1 to 76.9% on banana fibers after alkali treatment. The findings are also in close conformity with the findings by Liu et al. (2011), who noted an improvement in cellulose composition from 43.4 to 50.2% after alkali treatment on bamboo fibers. Different linkages in fibers have been reported to be broken as a result of sodium hydroxide treatment, this causes the release of lignin from the fibers and exposes cellulose in the fibers (Oladele et al. 2010, Reddy et al. 2013).

Table 1: Chemical composition of untreated and optimally treated sisal fibers

Fiber Property	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Untreated	64.6	10.6	10.7
Optimally Treated	64.7	4.9	6.4

The alkali treatment decreased the amount of hemicellulose in sisal fibers from 10.6 to 4.9%. This decrease is thought to be due to the high sensitivity and susceptibility of hemicellulose to alkali solution due to the presence of ester acid groups in its structure. The findings are in agreement with Reddy et al. (2013), who noted a decrease in the amount of hemicelluloses after alkali treatment of century fibers from 22.2 ± 0.9 to 7.0 ±1.0. Similar behavior was also reported by Reddy et al. (2016) on alkali-treated Ficus (*Peepal Tree*) leaf fibers. They observed a decrease in hemicellulose content from 30.5 ±1.2 to 12.6 ±0.9 upon treatment in NaOH. Hemicellulose is affected by alkali treatment due to the high sensitivity of hemicelluloses to the action of sodium hydroxide than lignin or cellulose (Reddy et al. 2013, Reddy et al. 2016 Teli and Terega 2019).

The amount of lignin on sisal fibers also decreased from 10.7 to 6.4% as a result of the alkali treatment. This may be due to the ability of alkali treatment to separate the fibers and dissolve the lignin materials, which could have then been washed away upon subsequent water washes. The findings of this study align with the study by Vardhini et al. (2016), who reported a significant decrease in lignin content of banana fibers from 17 to 10.3% after sodium hydroxide treatment of the fibers. Previous studies have also noted a decrease in lignin compositions on *Pongamia Pinnata L.* bark fiber (Umashankaran and Gopalakrishnan, 2021) and *Sesbania rostrata* treated fibers (Raja et al. 2021).

Effect of Alkali Treatment on Dyeability of Sisal Fibers

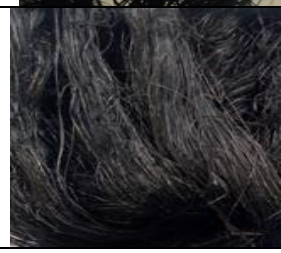
In this study, the untreated and alkali-treated sisal fibers were dyed with four

synthetic dyes namely; C. I. Reactive Red 31, C. I. Reactive Blue 21, C. I. Reactive Black 5 and C. I. Vat Black 25. The quality of dyeing was evaluated visually by measuring the dye bath exhaustion (*E %*) and by assessing the washing fastness properties of the dyed sisal fibers.

Visual Assessment of Dyed Sisal Fibers

Table 2 visually compares the appearance of the alkali-treated and untreated sisal fibers that were dyed with reactive and vat dyes. It can be seen from the table that the shades developed on alkali-treated sisal fibers appeared to be brighter and felt softer compared to the shade developed on untreated sisal fibers. The brighter shades could be due to removing the non-cellulosic component (i.e. lignin and hemicelluloses), which impedes the dye adsorption (Ding et al. 2014). On the other hand, alkali treatment results in an increase in the hydroxyl groups (OH) on the surface of the cellulose, which might have resulted in the formation of strong covalent bonds with dyes, thus developing the desired level of shade.

Table 2: Visual appearance of untreated and alkali treated sisal fibers dyed with reactive and vat dyes

Dye type	Untreated sisal fibers	Treated sisal fibers
C. I. Reactive Red 31 (RR31)		
C. I. Reactive Blue 21 (RB21)		
C. I. Reactive Black 5 (RB5)		
C. I. Vat Black (VB25)		

Assessment of Dye Exhaustion (% E) on Sisal Fibers

The extent of dye exhaustion (*% E*), which is defined as the transfer of a dye from the dye bath through absorption or adsorption to the fibers (Bhuiyan et al. 2013) was established by monitoring the changes in the absorbance of the dye bath during the dyeing process. The significance of measuring the extent of dye exhaustion was to determine the changes in absorbance of the dye bath due to the adsorption of the dye molecules onto the fibers and their diffusion extent into the fibers. The results of the percent exhaustion obtained in this study are given in **Table 3**.

Table 3: Exhaustion (%) of optimally treated and untreated sisal fibers dyed with reactive and vat dyes.

Parameters	Untreated Sisal fibers				Treated Sisal fibers			
	RR31	**RB21**	**RB5**	**VB25**	**RR31**	**RB21**	**RB5**	**VB25**
A_i	1.08	0.92	1.40	1.49	1.08	0.92	1.40	1.49
A_f	0.18	0.22	0.48	0.32	0.17	0.19	0.78	0.31
%E	**83%**	**76%**	**65%**	**78%**	**84%**	**79%**	**44%**	**79%**

Key: *A_i = initial absorbance of the dye bath, A_f = final absorbance of dye bath, % E = Exhaustion percent.*

It is clear from the table that extent of exhaustion (*%E*) on untreated sisal fibers was slightly lower than the treated fibers except for the C. I. Reactive Black 5, which showed contrary behavior. The slight increase in *% E* of for the treated sisal fiber could be explained by the fact that treatment of sisal fibers might have removed most of non-cellulosic components that impede dye absorption, consequently increasing the hydroxyl groups (OH) for the formation of bonds with dyes and develop the desired level of exhaustion. In addition, the treatment of fibers might have enhanced the dye-sites and swelling in the cellulose macromolecule of sisal fibers (Ding et al. 2014, Reza et al. 2014, Arju et al. 2015, Zhao et al. 2022). As a result, this could have increased the penetration, absorption and accessibility of dyestuffs into the fibers, resulting in a substantial increase in dye uptake and, hence, the overall increased exhaustion of the dye bath. On the other hand, unexpected decreases in % E for the treated fibers dyed with C.I. Reactive Black 5 despite similar alkali treatment is probably related to differences in molecular structure and size of the dye, diffusion properties and interaction of the dye with modified sisal fiber. It is possible that the alkali treatment altered the surface structure of the sisal fibers in a way that affected the affinity for Reactive Black 5, leading to reduced exhaustion levels compared to other reactive dyes. Generally, the findings of this study are consistent with related studies on other cellulosic fibers. For instance, Reza et al. (2014) and Arju et al. (2015) observed higher dye exhaustion in modified jute fibers as compared to the unmodified jute fibers while Ding et al. (2014) reported an increase in dye uptake of kapok fibers from 53 to 73% after the alkali treatment.

Assessment of Color Fastness to Washing

In this study, assessment of the color fastness to washing was done according to TZS 24 (TBS, 1979) and the results are indicated in **Table 4** and **Figure 4**. It is evident from the data that the level of fastness to washing achieved for both dyes can be classified as very good except in the case of C. I. Reactive Blue 21 where the fastness to washing was observed to be good. The results also indicate that dyed sisal fibers were noted to have good resistance to color fastness to washing since most levels met the minimum requirement on color fastness to washing.

Table 4: Ratings for Color fastness to washing on treated and untreated sisal fibers

Sample	RR31	RB21	RB5	VB
Untreated Sisal	4/5	3/4	4/5	4/5
Treated Sisal	4/5	3/4	4/5	4/5

(Rating scale key: 5= Excellent, 4= Very good, 3= Good, 2=fair 1= poor)

The good level of fastness to washing may be attributed by strong chemical interaction between dye functional group and functional groups of sisal fibers. The findings concur with Mbugua (2014) who found that sisal fibers (*Agave Americana*) dyed with vat dye displayed good to very good washing fastness properties. Finding by Chattopadhyay et al. (2006) also support the present work, since they reported to observe very good (4 - 5) wash fastness rating on reactive dyeing of cotton. Overall, the washing fastness of sisal fibers dyed with reactive and vat dyes was good to excellent which augmented the possibility to be used in textile apparel.

Figure 4: Visual appearance of dyed alkali treated sisal fibers: A, B, C and D= represent unwashed and washed sisal fibers dyed with RR31, RB21, RB5 and VB dyes respectively.

Conclusions

This study explored the effect of alkali treatment on the chemical composition and the dyeability properties of sisal fibers. The findings of the study revealed that, alkali treatment resulted in decreased amounts of hemicelluloses and lignin while slightly increasing the amount of cellulose in treated sisal fibers. This phenomenon was thought to be due to the ability of alkali treatment to break the bonds present between hemicellulose, lignin and cellulose thereby releasing the cellulose from these impurities, which were washed away upon subsequent water washes. The study has also shown that the dyeability of alkali-treated sisal fibers increased significantly compared to the untreated sisal fibers, with darker shades that had good to very good wash fastness properties being observed. The findings of this study have broader implications for the textile industry, as it has indicated that alkali treatment could unlock the potential of the treated sisal fibers for higher-textile applications, particularly in dyeing and clothing manufacturing. Further studies are needed to fully understand the additional modifications and scalability that could expand the utility of sisal fibers in the textile industry. It can therefore be concluded that, alkali treatment of sisal fibers could be used in enhancing both the chemical composition and reactive and vat dyeing properties of this sustainable fiber.

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Conflicts of interest

The authors declare no conflict of interest regarding preparation and publication of this manuscript.

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