Green Dyeing of Silk and Cotton Fabrics using Acacia nilotica seed pod

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Research Article

Keywords: Natural Dye, Acacia nilotica, Mordant, Wash fastness, Light fastness

Posted Date: May 11th, 2022

DOI: https://doi.org/10.21203/rs.3.rs-1470064/v1

Abstract

Natural dyeing is an eco-friendly technique to dye textile fabrics with colors extracted from natural sources like plants, flowers, fruits, minerals, etc. During the last few decades with the increased environmental awareness attention has been paid to natural dye. Worldwide many researchers and research groups are working to develop a better and optimized dyeing process using natural dyes. This research work is concerned with natural dye extraction from *Acacia nilotica* seed pods and its application for textile dyeing. The extracted dyeing agent was characterized by Ultraviolet-visible (UV-Vis) and Fourier transform infrared (FTIR) spectroscopy. UV-Vis absorbance maximum was found to be 250 nm, confirming the presence of phenolic compounds. Alkaline extract of seed pods was applied on silk and cotton fabrics in presence of mordant and without mordant. The effects of washing and sunlight on dyed silk and cotton fabrics have been studied to investigate the fastness properties. The dyed silk fabrics showed good fastness properties than that of dyed cotton. The dyed fabrics without any metal mordant showed promising washing and light fastness properties. This novel approach for *A. nilotica* seed pod based dyeing process of silk and cotton fabrics without any mordant could open new paths to green dyeing and be beneficial to the environment.

Introduction

Textile materials like cotton, silk, and wool used to be colored for price addition, appearance, and customer demand. Prior to the invention of synthetic dyes, textile materials were colored with colors extracted from natural sources. Most dyeing industries shifted towards the use of synthetic dyes after the commercialization of economic synthetic dyes and their ready availability. With the widespread convenience of cheaper artificial dyes and their moderate to wonderful color fastness properties, there was a speedy decline in natural dyes. Synthetic dyes are used in a wide range of industries, but textiles are the largest consumer of these dyes. The rise in the development of synthetic dyes came around the same time as the growth of industrial fabric production. Effective synthetic dyes were eagerly accepted in the expanding industry and nowadays over 7 x 105 tons of synthetic dyes are annually produced worldwide (Chequer et al. 2013). Wastewater from the textile industry contains a variety of polluting substances including dyes and up to 200,000 tons of synthetic dyes are lost to effluents every year during the dyeing and finishing operations (Ogugbue and Sawidis 2011). The use of synthetic dyes and dyeing processes contributes substantially to water contamination across the globe. Waste and unfixed colorants caused by the production and application of synthetic dyes release large quantities of pollutants to the surroundings and disposal of venturous wastes of artificial dyes may be a major environmental and economic challenge.

Moreover, the majority of synthetic organic dyes used in the textile and food processing industries are mainly azo dyes (Rayu and Sitaraman 2014). Aside from producing huge amounts of effluent containing these azo dyes, the textile industry requires a large number of carcinogenic arylamines and naphthols in order to produce dyes. Consequently, after aviation, the textile industry is the second most polluting industry in the world. Several studies found that nearly 20 percent of global water pollution comes from textile dyeing and treatment (Kant 2012). The risk in the use of synthetic dyes also arises from the breakdown of products. Moreover, most of the dyes are water-soluble organic compounds, and the high solubility in water makes it difficult to remove them by conventional methods. Laterally many harmful effects of synthetic dye on the environment and humans have been reported (Lellis et al. 2019; Ratna and Padhi 2012; Islam et al. 2011). With increasing textile industries, the toxic and hazardous dye effluent is increasing in the ecological system due to the unavailability of safe and green approaches for textile dyeing. With the increased environmental awareness of hazards caused by synthetic dyes, the demand and application of non-toxic, eco-friendly dyeing processes have been revived (Bechtold and Mussak 2009).

To overcome the negative impact of synthetic dyes bio-based dyeing process has been developed where dye is extracted from natural sources. The word 'natural dye' alludes to any or all assortments determined from normal sources. Natural dyes can be obtained from various parts of plants including roots, bark, leaves, flowers, and fruits which give various kinds of shades to fabrics. Although historically, plants have been used for the extraction of a majority of natural dyes researchers are continuously searching for new natural sources including twigs, seeds, stems, shells, heartwood, wood shavings, hulls, husks, etc. As their production and application do not require strong acids, alkalies, and hazardous chemicals the demand for natural dyes is increasing continuously. Still, the use of natural dyes for the coloration of textiles has been limited to small-scale dyers, craftsmen, and small-scale exporters but day by day the use of non-toxic and eco-friendly natural dyes on textiles fabrics has become a matter of significant importance. In the recent past, the dyeing effect of the henna leaf (Alam et al. 2007), tamarind seed (Tepparin et al. 2012), acacia bark (Saleh et al. 2013), turmeric spice (Gargoubi et al. 2015), pomegranate peel (Kulkarni et al. 2011), areca nut (Jain and Vasantha 2016), coconut shell (Akhter et al. 2014) on textile fabrics have been reported. Plant-derived and biodegradable natural dyes can be extracted easily from nature. Despite the availability of natural dyes, appropriate and standardized dyeing techniques should be developed with different color shades, acceptable color fastness, and reproducible color yield for the successful industrial use of natural dyes. As natural dyes are non-substantive towards fabrics, a metal mordant is required, which limits the eco-friendliness of the dyeing process (Geelani et al. 2017). An eco-friendlier dyeing process will require molecular level interactions to attract the dye molecules to the fabric without using any mordant.

Regarding the invention of new resources of natural dye and its application, technique the present research work aims to extract a novel natural dye from *Acacia nilotica* and its application through a simple dyeing process. *A. nilotica* is locally known as Babla, Babul, Gum Arabic, etc. Babla is a medium to large-sized, thorny, nearly evergreen, and the widely spread tree that can reach a height of 20-25 meters but may remain a shrub in poor growing conditions. It is indigenous to India, Saudi Arabia, Bangladesh, Burma, Sri Lanka, Egypt, Sudan, and some other tropical areas of the world. In Bangladesh, it is found mostly in the Northwest region. *A. nilotica* is a multipurpose tree that is economically used as a source of timber, fuel, tannins, gums, and animal feed. The fruits of *A. nilotica* are abundantly available, linear and narrow, flattened pods which contain various polyphenolic compounds like flavonoids, alkaloids, tannins, saponins, etc. (Abbasian et al. 2015; Karim and Azlan 2012). The seed pods of *A. nilotica* is also a good source of fiber, protein, fat, and carbohydrate. According to Tanner 1990, the *A. nilotica* fruits (pods with seeds) contain maximum soluble phenolics compared to other Acacia species. A recent study showed that the pods of *A. nilotica* have potent antioxidants and were found effective in protecting plasmid DNA and human serum albumin protein oxidation induced by hydroxyl radicals (Abdallah 2016). The anti-nutritional components (e.g. flavonoids, tannin) of *A. nilotica* seed pods could be used as coloring agents (Dulo et al. 2021). Alhaji et al. reported the production of tannins from *A. nilotica* pods and their application for tanning of leather 2020. Thus the present study was conducted to develop a simple dying process using natural dyes and to evaluate the effectiveness of the *A. nilotica* seed pod based natural dye on silk and cotton fabrics. Generally dyeing with natural dyes requires a metallic salt known as mordant, for ensuring a reasonable fastness of the color to sunlight and washing (Hegde and Goutham 2015; Chavan and Ghosh 2015). In this investigation silk and cotton fabrics are dyed with *A. nilotica* seed pod extract using potash alum, copper sulfate, and ferrous sulfate mordant. Fabrics are also dyed without any mordant for comparison to those dyed by using mordant. The Wash and light fastness of all dyed fabrics have been studied.

Materials And Methods

Sodium hydroxide, acetic acid, potash alum, copper sulfate, and ferrous sulfate used were laboratory-grade reagents. The standard detergent of SDCE ECE Non-Phosphate Type 2 (A), UK was used for washings.

Extraction of dyestuff from *Acacia nilotica* seed pods

100 g of fresh *Acacia nilotica* green seed pods (Fig. 1) were collected from the tree near BCSIR Laboratories, Rajshahi. 100 g of seed pods were smashed in a mortar pestle and then immersed in 500 mL 0.05% NaOH solution. The intense black color was formed. The mixture was heated at 70˚C for 30 minutes with continuous stirring to ensure the maximum extraction of the coloring matter. Then the residue of pods and seeds was separated by fine cloth filtering. The filtrate was collected and filtered again to ensure the separation of insoluble parts. Finally, the filtrate was collected and the dye extract was dried in an oven at 70˚C.

Spectroscopic Analysis

The Fourier transform-infrared (FTIR) absorption spectroscopy of dried *A. nilotica* seed pods based dye and dyed fabrics were taken over a scan range of 400-4000 cm-1 using the Spectrum Two FTIR-ATR Spectrometer, Perkin Elmer, UK. Maximum absorbance of a dilute solution of the dye was taken in the range of 200-800 nm using SP-UV 500DB, Spectrum, Germany.

Preparation of Fabrics

The cotton fabrics used for dyeing in the present work were collected from the local market and the silk fabrics were collected from the sericulture industry, Rajshahi. Cotton and silk fabrics of size 10 x 15 cm were heated with 1% detergent solution at 80˚C for 30 minutes to degum. Finally, the fabrics were washed thoroughly 2-3 times with distilled water.

Pre-Mordanting

Mordanting is the process that allows making a bond between the fiber and the dyestuff. It was observed that the pre-mordanting technique with metal salts imparted good fastness properties to the cotton, wool, and silk fabric due to the formation of complex, and the flavone-based compounds are known to form stable complexes with metal cations (Bukhari et al. 2017). All samples were heated at 80˚C with 0.1% acetic acid solution for 30 minutes. Then the 3 sets of the sample were treated with mordant in three separate beakers containing three different mordants. The concentration of aqueous potash alum, copper sulfate, and ferrous sulfate was 2%. The liquor-to-material ratio was 1:30. After 30 minutes the fabrics are rinsed with distilled water and transferred to a dyeing bath.

Dyeing

Four separate dyeing baths were prepared with 4% of extract of *A. nilotica* seed pods and the temperature of the dye bath was 80˚C. All the mordanted fabrics and one set of un-mordanted fabrics were dipped in the four separate dye baths allowed to stand for 1.5 hours. After dyeing, the dyed material was washed with cold water 3 times and dried at room temperature.

Fastness Property

The fastness of the color is the predominant property of dyed garment materials, which refers to the resistance of color to fade or bleed of dyed garment material to various types of external influences e.g. washing, sunlight, rubbing, perspiration, etc. Washing fastness was investigated by, dipping the dyed fabric in 1% standard detergent solution, and it was kept at 45˚C temperature for 30 minutes. After 30 minutes the fabrics were rinsed with distilled water, squeezed, and allowed to dry in defused sunlight. The washing procedure was repeated 7 times and after every wash, the dried samples were compared with the controlled sample. The color change was assessed using the SDC-3305 ISO Grey Scale. The results are shown in Table 1. To evaluate light fastness, the dyed samples were exposed to sunlight for 6 hours a day for seven consecutive days. The changes in shades after every day were measured with the help of Greyscale. The results are shown in Table 2.

Results And Discussion

The UV-Vis absorption spectrum is presented in Fig. 2. The alkaline extract of *A. nilotica* seed pod extract shows characteristic absorption maxima (λ_{max}) at 250 nm. The λ_{max} indicates that the alkaline extract of *A. nilotica* seed pod mainly contains flavonoids with aromatic chromophores (Sisa et al. 2010). The light absorption properties of flavonoids in the visible ultraviolet light region are responsible for the colors associated with them. Thus the UV-Vis spectral studies confirm the presence of flavonoids, which may impart color to textile materials. Furthermore, natural dyes can absorb a higher amount of UV radiation

(Baliarsingh et al. 2015). Sun burning or Skin damage is caused by UV radiation, so using cloth dyed with natural dyes is a safer way to protect individuals from sun burning (Pisitsak et al. 2018).

Fig. 3 shows the FTIR-ATR spectrum of *A. nilotica* seed pod extract. According to the literature the aqueous extract of *A. nilotica* mainly contains flavonoids, tannin, glycoside, saponins, and carbohydrates (Auwal et al. 2014). An intense peak at 1571 cm^{-1} was observed which can be assigned to the C=C stretch of the benzene ring in aromatic compounds. The wavenumber range between 3500-3200 cm^{-1} corresponds to the -OH functional group. The band at 2924 cm^{-1} could be related to The C-H, CH_2, and CH_3 stretching vibrations. The bands at 1370, 1058 and 869 cm^{-1} would be related to C-H bending vibration, C-O stretching vibration, and C-C stretching vibration respectively.

The FTIR-ATR spectra of raw cotton and dyed cotton using *A. nilotica* seed extract in presence of different mordants are presented in Fig. 4. A broad peak was observed at 3100-3600 cm^{-1} due to O–H stretching. The absorption band at 2885 cm^{-1} corresponds to the C-H stretching vibrations (Chung et al. 2004). In addition, the characteristic absorption band due to C-O stretching vibration was observed at 1027 cm^{-1} (Soleimani-Gorgani and Karami 2016). It can be seen that the spectra of the raw cotton and dyed cotton using different mordants look similar, except for the changes in their intensities. It can be also seen that the bands specific to the raw cotton fabric groups decreased in intensity after dyeing (Rosu et al. 2021).

Fig. 5 shows the FTIR-ATR spectra of raw silk and dyed silk using *A. nilotica* seed extract in presence of different mordants. Peaks corresponding to various groups in silk fibroin were also presented in the region of 1,200–2,000 cm^{-1}, indicating very little or no change in pure silk and dyed silk (Wei et al. 2011). Presence of amide I, amide II, and amide III on the surface of silk were confirmed by the peaks at 1625, 1515, and 1224 cm^{-1} respectively (Hong et al. 2021; Dodel et. al. 2016). However, it was not possible to distinguish individual dye components in silk fabric dyed with *A. nilotica* seed extract using the FTIR technique.

The experimental results showed that silk and cotton fabrics were successfully dyed with vegetable dye using *A. nilotica* seed pod extract. By using potash alum, copper sulfate, and ferrous sulfate mordant the vegetable dye gives golden, lightly golden, and dark brown color shades respectively. The images of dyed silk and cotton fabrics by *A. nilotica* seed pod extract in presence of mordant and without mordant are presented in Fig. 6. The deep dark brown color of silk fabrics in presence of ferrous sulfate is due to the strong coordination tendency of the metal ion. Fe^{2+} enhances the interaction between the fiber and the dye, resulting in high dye uptake (Jothi 2008). In presence of potash alum, the golden color of the silk is very shiny. Without any mordant, the vegetable dye gives a golden-brown shade to silk.

Improved fastness properties of dyed garment materials are essential for consumer expectations. In the case of properly dyed fabric, all the fibers absorb the dye and the color is affixed to the fiber, so that it may not be affected by different mechanical, physical, and chemical treatments. On the other hand, the poor colorfastness property of fabric indicates that the fabric was not dyed satisfactorily or the dye selected for dyeing was not appropriated as compared to fabric criteria. The wash fastness and light fastness values are shown in Table 1 and Table 2 respectively. After 7 times washing the fair fastness properties were recorded for the silk fabrics dyed without any mordant. The light fastness value after 42 hours of exposure was found to be 3-4. Slight alteration of color was observed in both cases and dyed fabrics showed fair-good fastness properties. From the results, it can be assumed that the vegetable dye is strongly fixed on silk fabrics without any mordant. By using different mordants the color shade can be changed and the dyed fabrics show moderate wash and light fastness. After 7 times washing the fastness value for dyed silk fabrics with copper sulfate, ferrous sulfate, and potash alum were found 3, 2-3, 2-3 respectively. After 42 hours of exposure, the light fastness values were found 3-4, 3, and 3 for the same fabrics. Appreciable loss of color was observed after 7-time washings and very slight loss after 42 hours of exposure to sunlight. Moderate fastness was found for all cotton fabrics after 42 hours of exposure to sunlight and distinct alteration in color were observed after 7-time washings. The wash fastness values were found 2-3 for dyed cotton fabrics in presence of copper sulfate mordant and without mordant. These values were found 1-2 for dyed cotton fabrics in presence of ferrous sulfate and potash alum mordant. After 42 hours of exposure to light, the same fastness values were found for dyed cotton fabrics in presence of copper sulfate mordant and without mordant. These values were found 2-3 for dyed cotton fabrics in presence of ferrous sulfate and potash alum mordants. The neutral surface of cotton fabrics is responsible for the distinct color loss, poor fastness value, and gloomy shades. Moreover, natural dyes have a fewer presence of reactive groups in their terminal part to react with the fibers (Alam 2020). So the distinct color loss can be attributed to the poor attraction between the cotton fiber and dye molecules. On the other hand, the static positive charge on the silk surface shows moderate fastness values even without any mordant. In 2020, Karabulut and Atav reported the fastness properties of cotton fabrics dyed with 40 different dye plants without any mordant. They found 6 of the dye plants (Catechu, Indigo, Myrobalan, Pomegranate, Turmeric, and White Onion) were able to produce high color yield and good fastness properties. Fig. 7 shows the light and wash fastness properties of the dyed fabrics without any mordant. The fastness properties of silk and cotton were found to be fair to good. Remarkable alteration of the color of the dyed fabrics in the presence of mordant was observed after 6 to 7-time wash and those sowed poor fastness values. With the results and analysis derived from the present research, metal mordant is only responsible for the different shades of dyed fabrics using *A. nilotica* seed pod. In spite of the goal of mordanting in dyeing to increase dye absorption and stability, the recorded fastness values indicate that metal mordant has no significant effect on dye stability. Without any mordant, the dyed fabrics showed good to fair fastness properties which can be explained by the presence of sufficient tannin materials in the extracted dye. Historically polyphenolic tannin molecules are used in textile and adhesive industries which can act as natural mordants (Hong 2018). Previous research reported that the *A. nilotica* seed pod is a good source of tannin and the tannin content varies from 30 to 50% (Kamal et al. 2005; El-Sissi et al. 1965). So it can be concluded that *A. nilotica* seed pod based natural dye contains sufficient tannin materials which may replace the metals salts. Having successfully implemented the novel *A. nilotica* seed pod based dyeing technique, it would be possible to skip the mordanting part of the natural dyeing process.

Table 1: Wash fastness of silk and cotton fabrics dyed with *A. nilotica* seed pod extract

Washing times	Wash fastness							
	Silk				Cotton			
	Without mordant	Copper sulfate	Ferrous sulfate	Potash alum	Without mordant	Copper sulfate	Ferrous sulfate	Potash alum
Day-1	4	4	4	4-5	3-4	3-4	4	3-4
Day-2	4	3-4	3-4	4	3	3	3	3
Day-3	3-4	3-4	3	4	3	3	2-3	3
Day-4	3-4	3	3	3	3	3	2	2
Day-5	3	3	3	3	3	3	2	2
Day-6	3	3	2-3	3	2-3	2-3	1-2	1-2
Day-7	3	3	2-3	2-3	2-3	2-3	1-2	1-2

Table 2: Light fastness of silk and cotton fabrics dyed with *A. nilotica* seed pod extract

Exposure time (Hours)	Light fastness							
	Silk				Cotton			
	Without mordant	Copper sulfate	Ferrous sulfate	Potash alum	Without mordant	Copper sulfate	Ferrous sulfate	Potash alum
6	4	4	4-5	4-5	4	3-4	4	4
12	4	3-4	4	4	3-4	3-4	3	3
18	3-4	3-4	4	4	3	3	3	3
24	3-4	3-4	4	3-4	3	3	3	2-3
30	3-4	3-4	3-4	3-4	3	3	2-3	2-3
36	3-4	3-4	3	3	3	3	2-3	2-3
42	3-4	3-4	3	3	3	3	2-3	2-3

Conclusion

As natural dyes come from natural sources, they neither contain harmful chemicals nor carcinogenic components, which is common to synthetic dyes. Almost every synthetic dye is synthesized from petrochemical sources, so the application of natural dyes has a strong potential to reduce the consumption of fossil fuels. In contrast, natural dyes have been proven to be safe for human skin contact and are generally non-hazardous. Natural dyes are fascinatedly termed as green chemicals but still, now natural dye processes are not yet ready for industrial production. In this study, the dyeing effect of *A. nilotica* seed pods was determined and results revealed that using *A. nilotica* seed pods based dye silk fabrics shows an appreciable depth of color and good fastness (light and wash) properties in presence of mordants. This natural dye can also be applied without any mordant, which is the novel feature of this vegetable dye. The color depth and fatness properties of silk and cotton fabrics indicate that the dye will be more suitable for silk fabrics. Without any types of metal mordant the textile dye effluents will be less contaminated with metal salts, which would be advantageous for the human being. The novelty of this research is the simple extraction and easy dying process without any metal salt, which is appropriate for industrial scale-up and pollution control. We have to make more and more research to find out the new color source and its sustainable large production for industrial applications. Successful introduction of vegetable dye into technical dyeing processes, some additional demands can be fulfilled. Extensive research will be required on green dyes to minimize the use of toxic synthetic petroleum-based dyes.

Declarations

ACKNOWLEDGMENT

The authors are grateful to the Bangladesh Council of Scientific and Industrial Research (BCSIR) for providing all research facilities.

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Figures

Figure 1

Green *Acacia nilotica* seed pods

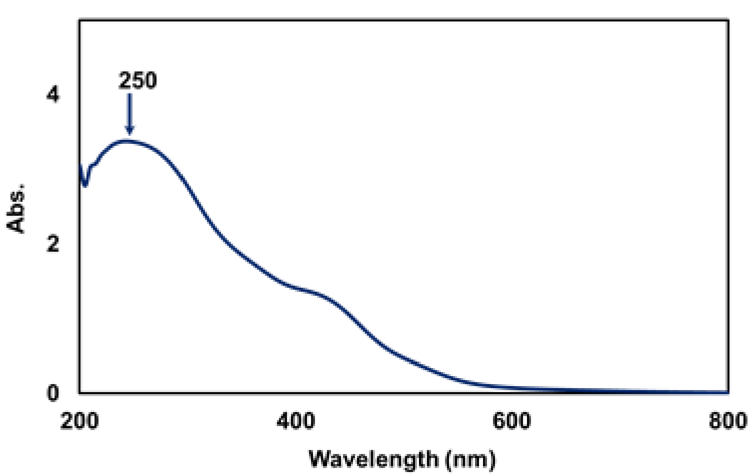

Figure 2

UV-Vis spectrum of *A. nilotica* seed pod extract

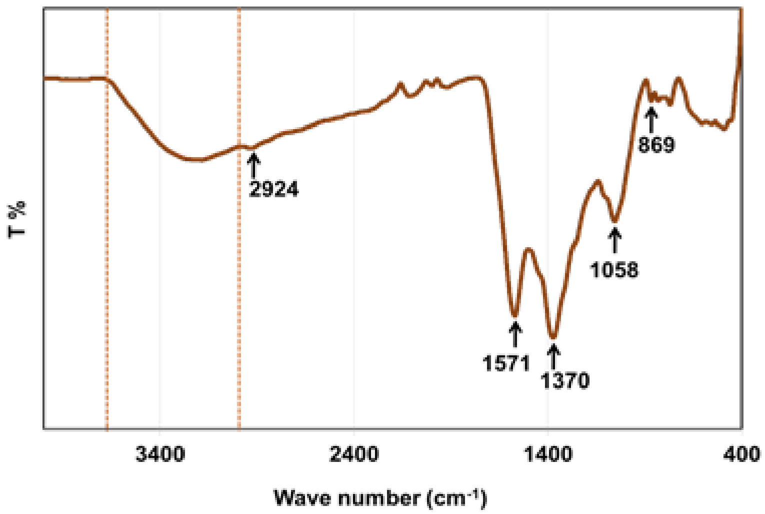

Figure 3

FTIR-ATR spectrum of *A. nilotica* seed pod extract.

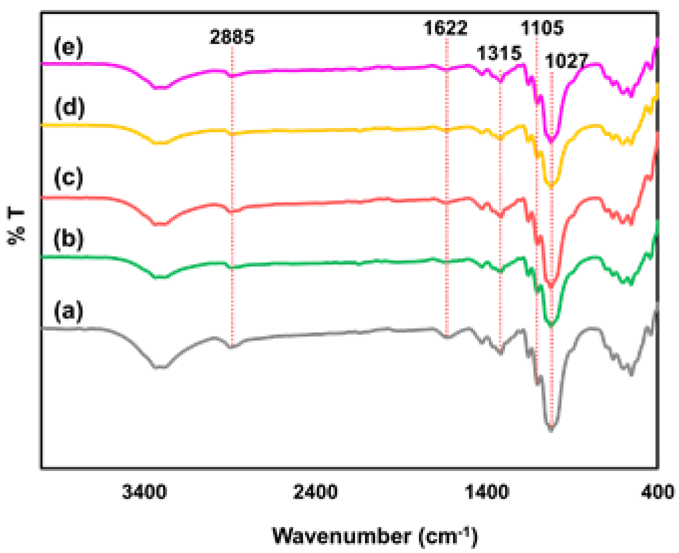

Figure 4

FTIR-ATR spectra of (a) raw cotton and dyed cotton with *A. nilotica* seed pod extract (b) without mordant, and with (c) potash alum, (d) $CuSO_4$, (e) $FeSO_4$ mordant

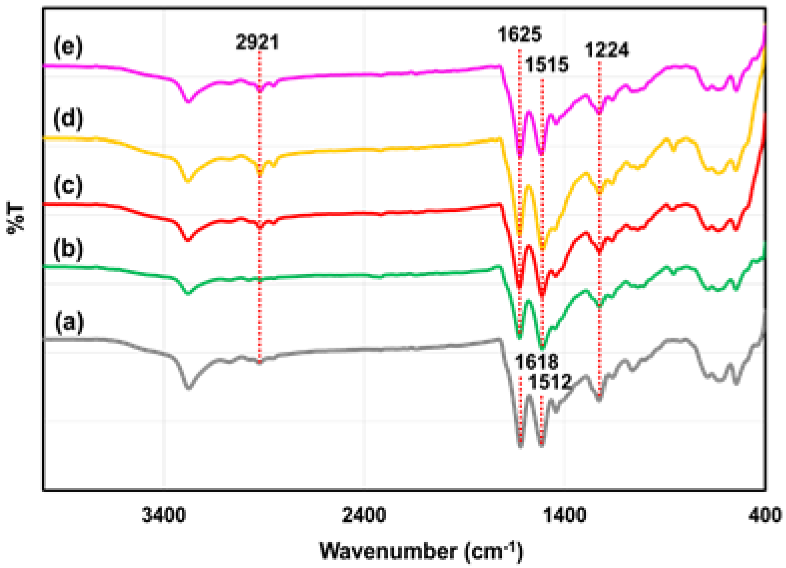

Figure 5

FTIR-ATR spectra of (a) raw silk and dyed silk with *A. nilotica* seed pod extract (b) without mordant, and with (c) potash alum, (d) $CuSO_4$, (e) $FeSO_4$ mordant

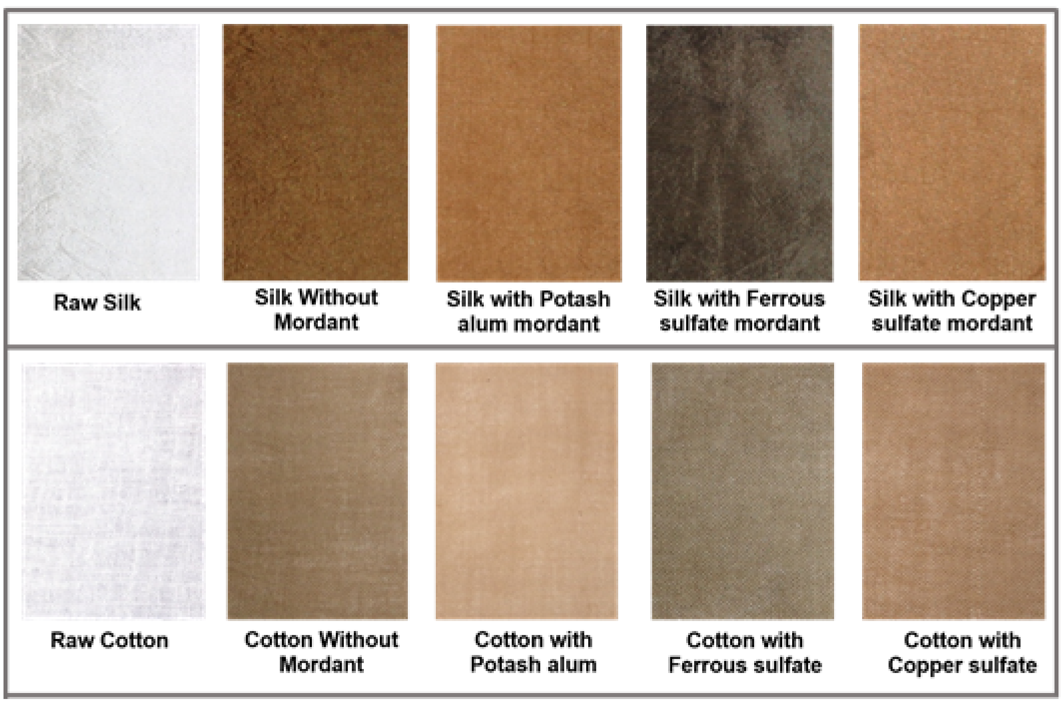

Figure 6

Images of silk and cotton fabrics dyed with *A. nilotica* seed pod extract

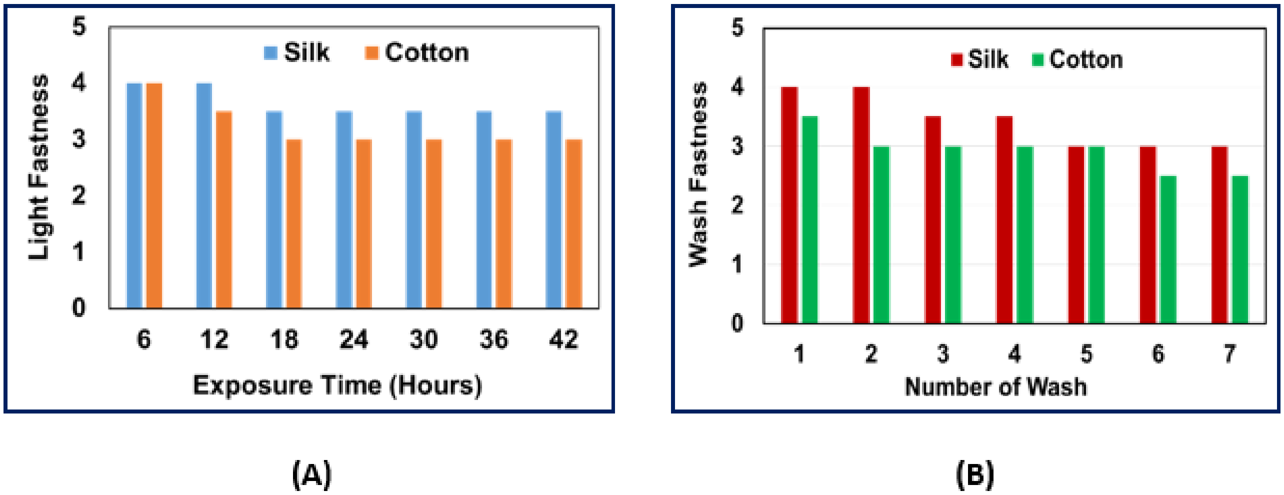

Figure 7

(A) Light fastness (B) Wash fastness of dyed fabrics without mordant

Supplementary Files

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- GraphicalAbstarctCleanTechEnvironPol.png