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Functional Dyeing of Cellulose-based (Linen) Fabric Using *Bombax Ceiba* (Kapok) Flower Extract

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Abstract: Linen fiber, which is claimed to be more comfortable than cotton, is an important textile fiber obtained from *Linum usitatissimum* (Flax) plant. However, the lack of functional properties of linen limits its application in functional textile products. Nowadays, use of natural colorant for textile dyeing has become an attractive option for sustainable textile coloration. Kapok flower is an unexplored source of natural dyestuff which can also be utilized for functionalization of cellulose-based textiles. In the current work, kapok flower extract (KFC) was utilized as a functional dye for dyeing of linen fabric in the presence of metallic mordants. The dyed fabrics were evaluated for coloration properties (color values, color coordinates, and fastness properties) as well as functional properties viz. antibacterial activity, UV protection, and antioxidant activity. The satisfactory level of dyeing with acceptable fastness ratings was achieved on linen fabrics. The dyed fabrics displayed an excellent antibacterial activity against gram-positive and gram-negative bacteria. The efficient level of UV protection and radical scavenging (antioxidant activity) was also obtained. The mechanism of functional modification of linen using KFC was discussed.

Keywords: Linen, Kapok, Dyeing, Antibacterial, UPF, Antioxidant

Introduction

Linen fiber is an important natural fiber obtained from flax (Linum usitatissimum) plant. It is an upcoming fiber for textile applications with most suitable properties for the products like apparels, bedsheets, and many technical textiles. However, being cellulosic, it lacks very important functional properties like antibacterial activity, UV protection, and flame retardancy. With the development of textile-based wellness products, the antioxidant activity of the substrates is highly demanded. Hence functional modification of such cellulose-based fiber is the new quest for research.

A huge amount of plant sources has been identified for extraction of different vibrant colors and their multifunctional requirements in textile processing and other disciplines. In order to obtain attractive natural dyes, various parts of plants like their flowers, stem, pellets, and also animals have been utilized. Natural dyes, as compared to synthetic dyes, have better biodegradability and compatibility with the environment. These benefits have led to an increasing customer demand for natural coloration of textile products. However, most of the natural dyes suffer from limitations like availability of limited shades, low reproducibility, inferior fastness, requirement of mordant, and higher cost, which restricts the commercial exploration of natural dyes. In most of the natural dyes, the dye-mordant combination governs the properties of dyed materials and the said limitations thus can be overcome using suitable combination of dye and mordant. There are continuous investigations in the various species of plants for identifying new sources of natural dyes and natural mordants [1-13].

The continuous research in the field of natural dyes had led to an exploration of many functional properties of the natural-dyed substrates. The plant extracts based functional finishing of various textile fibers has been reported [14-25].

Kapok plant contains about 26 genera and nearly 150 pantropical species. It is commonly known as *purani, pagun, roktosimul, ceiba*, and *Indian bombax.* Bombax or red silk cotton is an important medicinal plant and widely found in Australia, tropical Asia, and northeast Africa [26,27]. In India, it is found at low altitudes to high altitudes specifically in autumn/winter/spring seasons [28]. Kapok plant has several medicinal properties such as hepatoprotective, anti-inflammatory, antiangiogenic, anti-HIV, antioxidant activities, etc. and is extensively used for curing diseases [29-31]. Kapok flower retains red without fading over a week after falling on the ground [32], which indicates the resistance of kapok colorant towards fading in the presence of sunlight. In short, KFC can be utilized as functional natural dye. However, the literature regarding the use of KFC as a natural dye for textiles is rather scarce. Chung *et al*. studied the dyeing performance of kapok flower colorant on wool fabrics and achieved remarkable results [33].

In the present work, we are reporting the first application of kapok flower colorant (KFC) in simultaneous dyeing and multifunctional modification of linen fabric. The coloration of linen fabric using KFC and metallic mordants was reported. The efficacy of imparted functional properties viz. antioxidant property, UV protection factor, and antibacterial activity was also studied.

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Experimental

Materials

Linen fabrics (EPI-45, PPI-41, and GSM-241.5) were supplied by Jayshree Textiles. The received fabrics were washed, dried, and further used for dyeing. The kapok flowers (red colored) were collected from Institute campus. All the chemicals used in the study were of laboratory grade, which were purchased from Sigma Aldrich and Merck Specialties Private Limited, India.

Method

Extraction of KFC

Kapok flowers were dried in an oven at 45 °C for 24 h and powdered in a mixer-grinder. Extraction of KFC was carried out using an aqueous extraction method. The kapok flower powder (5 g) was suspended in water (100 m*l*) and the extraction was carried out at 90 °C for 60 min using a water bath (Khera, India). After the completion of extraction time, the liquor was filtered to collect the extract. The volume of the final extract was made to 100 m*l* using water and the prepared stock solution of KFC (5 %) was used for dyeing. In order to study the absorption pattern of extract in UV-Visible region, the KFC extract was analyzed using a UV-Vis spectroscope (Shmadzu, Japan).

Mordanting and Dyeing of Linen Fabric

The pre-mordanting process was used for this experiment. The mordants used for this study were different metallic salts such as copper sulfate, ferrous sulfate, and potash alum which were used in the concentration of 1 %, 5 %, and 10 % (on weight of fiber, owf), respectively. The metal salts were dissolved in distilled water to make an M:L ratio of 1:10. The fabric samples were immersed in the mordant solution and treated at 90 °C for 60 min. After mordanting, the fabric samples were removed, squeezed, and subjected to further dyeing process.

The mordanted samples were dyed with the KFC (20 % shade owf) by using the M:L ratio of 1:10. The mordanted fabric samples were dipped in dye extract and dyeing was carried out in a water bath at 90 °C for 60 min. After the completion of dyeing, the samples were removed from the dye solution and washed with cold water for 10 min. The sample names were abbreviated as AKFC (alum mordanted and dyed), CKFC (copper sulfate mordanted and dyed), and FKFC (ferrous sulfate mordanted and dyed).

FTIR Analysis

FTIR spectra of the dyed fabrics were recorded using a Thermo Scientific Nicolet iS50 FTIR Spectrometer. The samples were recorded and analyzed with 15 scans in the wavenumber range 400-4000 cm^{-1} with a 4 cm^{-1} resolution.

Color Strength Measurement

The reflectance (R %), CIE $L^*a^*b^*$ values and color strength (K/S value) of the dyed fabric samples were analyzed using a spectrophotometer (Color-EYE 7000A). The method involves Kulbelka-Munk K/S equation. K/S value denotes the fabric color strength. Based on the reflectance (R %), color strength value can be calculated by the following formula:

$$K/S = \frac{(1-R)^2}{2R}$$

where K is absorption coefficient) and S is scattering coefficient.

Fastness Test

The dyed fabrics were analyzed for washing fastness according to standard test method ISO II [34]. The rubbing fastness was evaluated as per the standard test method ISO105-X12.

Antimicrobial Activity

The antibacterial activity was assessed by using the agar plate method (AATCC 100-2004) [35]. The ability of the fabric samples to prevent microbial growth and retention was evaluated using *S. aureus* (a gram-positive bacterium) and *E. coli* (a gram-negative bacterium) cultures. The following equation was used to calculate the bacterial reduction:

$$\text{Bacterial reduction (\%)} = \frac{(B-A)}{A} \times 100$$

where A=the number of bacterial colonies recovered from the inoculated treated test specimen swatches in the jar incubated for 24 h contact period and B=the number of bacterial colonies recovered from the inoculated untreated control test specimen swatches in the jar immediately after inoculation (at 0 contact time).

Antioxidant Activity

The antioxidant activity of the dyed fabric samples was tested using the 2.2-diphenyl-1-picryl-hydrazil (DPPH) reagent, as per method available in the literature [36]. One-inch square samples of linen were added to a test tube which is containing 4 m*l* of freshly prepared DPPH radical (1.0×10^{-4} mol/*l*) in a methanol solution and the reaction was allowed for 30 min at 25 °C in the dark using a shaking incubator. The decolorizing result of the samples was analyzed by measuring the absorbance at 517 nm by a UV-VIS spectrophotometer (Shimadzu, Japan). The following formula was used to determine the antioxidant activity (%):

$$\text{Antioxidant activity (\%)} = \frac{(A_c - A_s)}{A_c} \times 100$$

where A_c is absorbance of the control and A_s is absorbance of the sample.

Ultraviolet Protection Factor (UPF)

The ultraviolet protection factor (UPF) of the dyed samples was evaluated as per AS/NZS 4399:1996 standard [37] using a UV-visible spectrometer (Cary, USA). The *UPF* was determined by using following formula;

$$UPF = \frac{\sum_{290}^{400} E_\lambda S_\lambda \Delta\lambda}{\sum_{290}^{400} E_\lambda S_\lambda T_\lambda \Delta\lambda}$$

where S_λ (Erythema action spectrum), E_λ (solar irradiance), $\Delta\lambda$ (wavelength increment), and T_λ (specimen spectral transmittance).

The UPF range and the corresponding category of protection is available in the literature [37].

Results and Discussion

Characterization of KFC Extract and Dyed Sample

The FTIR spectra of extracted and purified KFC were recorded using a Thermo Scientific Nicolet iS50 FT-IR Spectrometer with a wavenumber range of 4000-400 cm^{-1} and the most characteristic functional chemical groups are present in the colorant are shown in Figure 1. The characteristics peaks were observed at 3270 cm^{-1} (O-H stretching band), 2920 cm^{-1} (CH-vibration), 2110 cm^{-1} (CH_2 symmetric stretching), 1730 cm^{-1} (C=O stretching vibration), 1610 cm^{-1} (C=C stretching and -N-H stretching), 1440 cm^{-1} (CH_2 deformation, C-H deformation), 1290 cm^{-1} (C-O stretching vibration), 1050 cm^{-1} (C-O and C-O-C vibration), 778 cm^{-1} (-C-H vibration).

The FTIR spectrum of KFC showed various peaks indicating the presence of natural pigments like anthocyanin and carotenoid. The presence of these pigments was reported in earlier studies from various researchers [38,39]. Apart from this, kapok flowers contains various active compounds like flavones (apigenin, cosmetin, isovitexin, linarin, saponarin, vicenin 2, xanthomicrol [40], flavonols (3,5-dihydroxy-4'-methoxy-flavon7-yl-O-α-l-rhamnopyranosyl-(1→6)-β-d-glucopyranoside [41], kaempferol, quercetin [42]), anthocyanidins and anthocyanins (cyanidin-3,5-diglucoside [43], pelargonidin-5-β-d-glucoside, cyanidin-7-methyl-ether-3-β-d-glucoside [44], triterpenes (α-amyrin [40]), etc.

The FTIR spectra of undyed and dyed fabric samples are shown in Figure 2.

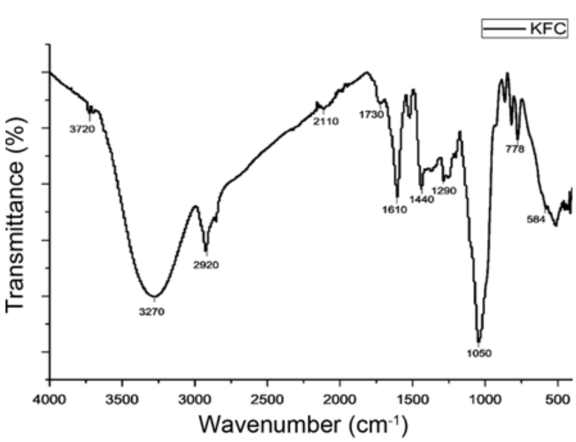

Figure 1. FTIR of KFC extract.

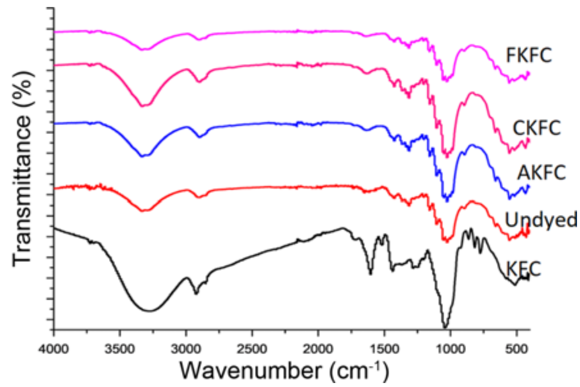

Figure 2. FTIR of undyed sample, KFC extract, AKFC, CKFC, and KFFC.

No significant difference was observed between the FTIR spectra of undyed and dyed linen samples. The dyed fabrics showed some changes in the peaks between 1500-1750 cm^{-1}. A change in the peak intensity of absorption band of -OH groups was observed between dyed and undyed fabrics. The change can be observed in peak intensity at 2920 cm^{-1} when linen dyed with KFC. The peak intensity change between 2800-2950 cm^{-1} may be attributed to the C-H groups of from KFC. A significant change of peak intensity was observed in various peaks viz. 1440-1750 cm^{-1} assigned to the C=O vibration at 1730 cm^{-1}, C=C stretching and -N-H stretching at 1610 cm^{-1} and CH_2 deformation, C-H deformation at 1440 cm^{-1}. This might be attributed to complex formed between metal ions and KFC with the cellulose backbone. As the metal ions are held on cellulose because of -OH groups which later form a complex with the active compound of KFC extract, a big shift in FTIR spectrum was not expected. Apart from this, linen itself contains various functional groups which are identical to those present in KFC extracts which makes it difficult for FTIR spectrum to give very clear distinction between undyed and dyed linen.

Color Values of the Dyed Sample

Most of the natural dyes have no or moderate affinity for textile substrates and mordants must be used to improve the color strength and fastness. To improve dye affinity, there is a need of mordanting agent which can produce a molecular bond between the fiber and dye molecules. The metal salts like potash alum, copper sulfate, ferrous sulfate, etc. form complex with dye molecules and show efficient mordanting action for the natural dyes. After mordanting, the metal salts attached to the fibers attract the dye molecules towards the fibers and finally develops the link between the dye molecules and the fiber by forming stable complexes. Most of the metallic mordants attached to any textile fiber, chemically combine with particular chemical functional groups present in the natural dyes and bond with the help of hydrogen bonds, ionic bonds, and other important bonding

Table 1. Color values and color coordinates of dyed samples

Sr. no.	Sample	Mordant concentration	L^*	a^*	b^*	*K/S*
1	Undyed		91.39	-0.89	5.10	0.23
2	AKFC	10	78.11	-0.95	14.15	1.04
3	CKFC	1	73.14	2.77	16.75	1.14
4	FKFC	5	56.12	-0.39	10.28	4.13

Table 2. Fastness properties of dyed samples

Sr. no.	Sample	Wash fastness		Rubbing fastness	
		Change in shade	Staining	Dry	Wet
1	AKFC	3-4	4	4-5	4
2	CKFC	4	4-5	4-5	4
3	FKFC	4	4-5	4-5	4

forces [45]. The validation of the requirement of mordant for dyeing of linen fabric using KFC has been confirmed as the fabric dyed without mordant showed negligible color strength which was further reduced after washing.

The color strength of dyed samples was evaluated with the help of Kubelka-Munk equation and the results are presented in Table 1.

The satisfactory color strength was observed on dyed linen fabrics, which indicates the suitability of KFC as a natural dye. The beautiful shades were built on linen fabric (Figure 3), the depths and tones of which varied with mordant type. The AKFC, CKFC, and FKFC showed yellow-brown, brown, and grey shades, respectively. The use of different mordants not only caused the variations in hue and significant changes in color strength (*K/S* values), but also affected the brightness index values and L^* values. The importance of mordants in providing the higher color strength values to dyed fabric may be attributed to their ability to form complexes between the fiber and the dye molecules thus resulting in fixation of higher amount of dye on fiber. Combination of kapok colorant and linen was affected by the presence of metal ions, resulting in a change in shade. The highest *K/S* value was found in case of FKFC sample which represents ability of Fe ion to interact with higher number of dye molecules.

Table 1 shows CIE $L^*a^*b^*$ values in which positive value a^* and b^* represent red and yellow shades while a negative value of a^* and b^* represent green and blue shades, respectively. The AKFC, CKFC, and FKFC displayed green-yellow, red-yellow, and green-yellow tones, respectively. This gives an immense potential where a shade and tone of the dyed fabrics can be varied using different mordants when a single natural dye is available for dyeing.

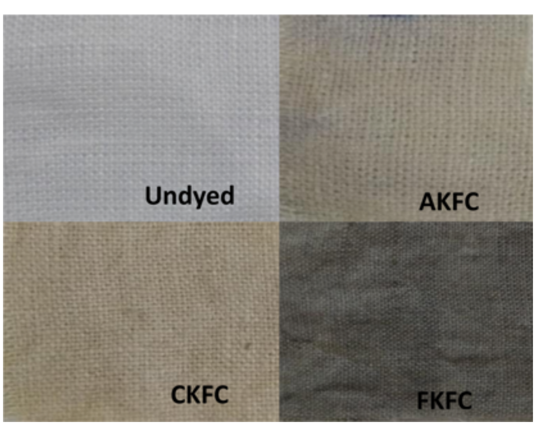

Figure 3. Dyed linen fabrics using kapok flower extract.

Fastness Properties of Dyed Sample

Fastness properties, which indicate the resistance of dye on dyed fabric to come out in presence of various agencies, were evaluated and the results are summarized in Table 2.

The washing fastness varied with the use of different mordants; however, all samples viz. AKFC, CKFC, and FKFC displayed an acceptable level of fastness. This further confirms the wash-fast interaction between dye and fiber using metallic mordants. The efficient mordanting action was achieved because of a complex formation between the dye and mordant which holds the dye firmly on cellulose and prevents bleeding in the wash liquor. The wash fastness achieved may also be attributed to the formation of an additional bond between the fiber, metal ions, and KFC molecules, which could overcome the force applied during washing. Staining was negligible on the adjacent fabric used for testing of wash fastness which also indicates limited affinity of KFC towards cellulose in the absence of mordant. All dyed samples displayed excellent rubbing fastness indicating stability of dye-mordant complex during an abrasive action. Among all mordants studied, FKFC was found to give the best fastness; however, it is also the question of optimization of mordant and dye concentration.

Antimicrobial Activity of Dyed Fabrics

The microbial contamination on textile materials is considered as a severe limitation for its use in health and hygiene applications. The efficacy of dyed fabrics as

Table 3. Antibacterial and antioxidant properties of kapok colorant dyed linen fabrics

Sr. no.	Sample	Bacterial colony reduction (%)		Radical scavenging activity (%)
		E. coli	*S. aureus*	
1	Undyed	N	N	7.52
2	AKFC	84	78	89.39
3	CKFC	95	88	90.27
4	FKFC	97	92	91.87

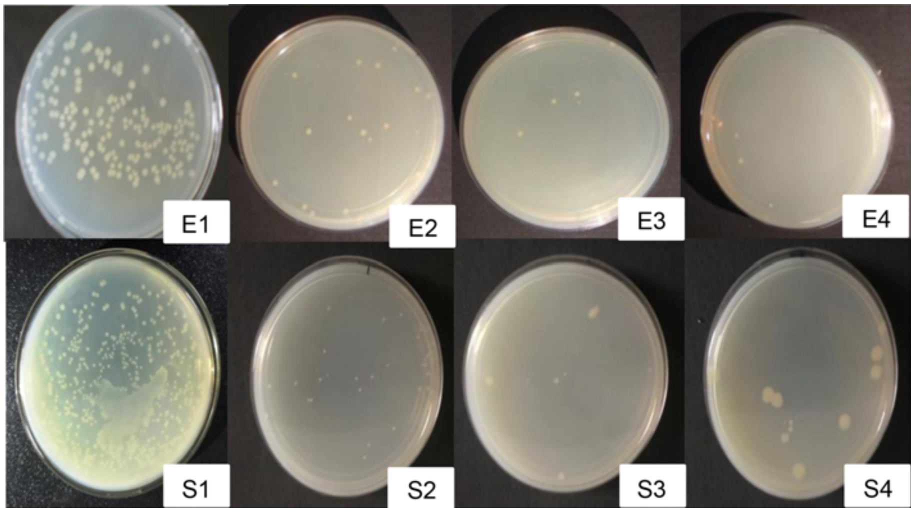

Figure 4. Pictorial representation of Antibacterial activity of dyed fabrics; (E1) Blank *E. coli*, (E2) AKFC against *E. coli*, (E3) CKFC against *E. coli*, (E4) FKFC against *E. coli*, (S1) Blank *S. aureus*, (S2) AKFC against *S. aureus*, (S3) CKFC against *S. aureus*, and (S4) FKFC against *S. aureus*.

antibacterial material was evaluated against gram positive and gram negative bacteria and the results are presented in Table 3. The pictorial presentation of bacterial colony reduction is presented in Figure 4.

The dyed fabrics displayed varying level of antibacterial activity which is ultimately dependent on the mordant-dye combination; however, all dyed samples can be regarded as antibacterial materials. In general, the dyed samples displayed better antibacterial properties against *E.coli* as compared to that against *S. aureus.* The antibacterial activity of KFC dyed fabrics was ranked in the order FKFC > CKFC > AKFC and the trend was valid against both *S. aureus* and *E. coli*. This is due to the fact that metal ions of mordants exhibit toxic effects against the pathogens and prevent bacteria attack [46] along with the inherent antibacterial properties of KFC. The presence of various active compounds like flavones, flavonols, kaempferol, quercetin, anthocyanidins and anthocyanins, pelargonidin-5-β-d-glucoside, cyanidin-7-methyl-ether-3-β-d-glucoside triterpenes (α-amyrin), etc. are known for antimicrobial properties and thus also impart antibacterial properties to the dyed samples. In case of natural dyeing, the antibacterial activity of dyed fabric is a combined effect of the mordant-dye complex. The non-leaching type of mechanism of antibacterial activity is expected in such cases where the active compound does not leach out and kill the bacteria. It remains bound to fiber and kills the bacterium coming in contact with it.

Antioxidant Properties of Dyed Samples

Results in Table 3 clearly showed the absence of antioxidant activity in case of undyed linen; however, all dyed samples show efficient radical scavenging activity. The tannins and flavonoids contain a huge number of free, glycosylated, and esterified hydroxyl groups attached to the aromatic and heterocyclic rings which are responsible for radical scavenging activity. The number and position of these free hydroxyl groups on the ring affect the radical scavenging activity significantly. DPPH is a stable radical and can easily undergo scavenging in the presence of antioxidant compounds containing hydroxyl groups which can scavenge radicals and help protect human cells against oxidative damage [47,48]. The antioxidant activity of dyed fabrics was mainly due to the presence of polyphenolic compounds and flavonoids which have the tendency to transfer hydrogen or electron thus stabilizing free radical. In case of dyed linen samples, the antioxidant activity depends on the amount of KFC extract, containing polyphenolic compounds, exhausted on the fiber as well as the free functionalities of the biomolecules present on the dyed sample. All dyed samples displayed an excellent antioxidant activity indicating the availability of sufficient phenolic groups for the scavenging of DPPH radical.

UV Protection of Dyed Samples

The UV radiation emitted by the sun has a broad spectrum from the high to low-energy with varying wavelengths, which is classified as UVC (below 280 nm), UVB (280-315 nm), and UVA (315-400 nm). It is highly desirable to develop UV protective clothing that can protect user from harmful effects of such radiations. In general, lower value of UV transmittance indicates better UV protection property of the sample [49-53]. The UV protection offered by dyed samples are presented in Figure 5 and Table 4. As evident from Figure 5, the UV transmittance spectra showed a significant difference between the dyed fabric samples and undyed sample.

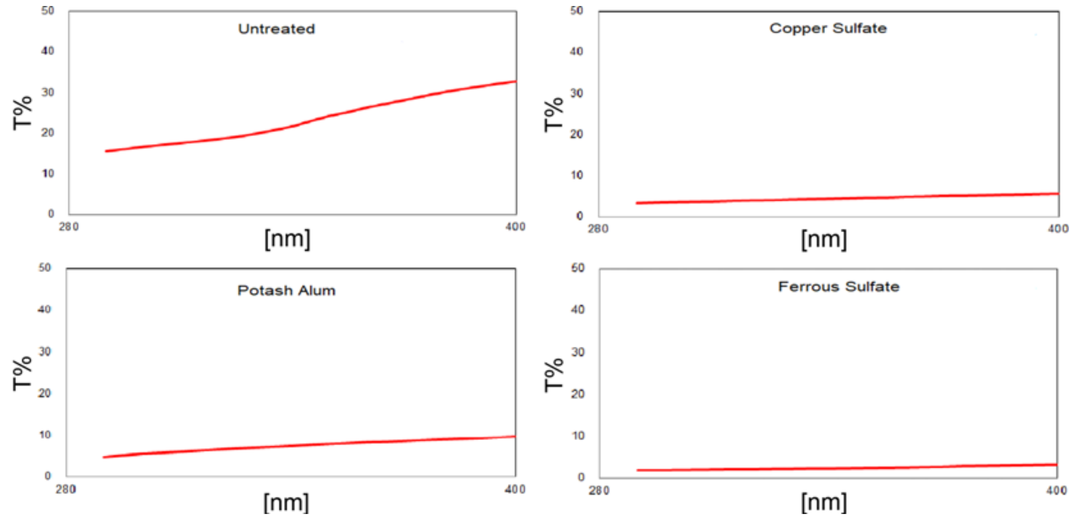

Figure 5. UV transmittance spectra of the undyed and dyed fabric samples.

Table 4. Comparison of UV transmittance spectra of the untreated and dyed fabric samples

Mordant	UVA	UVB	UPF
	315-400 nm	290-315 nm	290-400 nm
Undyed	25.46	16.80	5.62
AKFC	8.08	5.66	16.29
CKFC	4.72	3.56	26.73
FKFC	2.55	1.90	50.38

The UV transmittance of the undyed linen was higher than the dyed samples. This is well reflected in UPF value of untreated linen which was found to be 5.62. Hence the poor UV protection offered by the undyed sample.

Evidently, less UV transmittance was detected when fabric samples were dyed with KFC that ultimately displayed good UV protection properties. As shown in Table 4, it is clearly noticed that different mordants had different effects on the spectral transmittance of fabrics dyed with KFC. However, the UPF values displayed by all dyed samples were satisfactory to provide efficient UV protection. This may be due to the metal salts bridging the fibers and the dyes molecules resulting in the formation of differently coordinated complexes of π-bonds [52]. Therefore, fabrics dyed with KFC can block ultraviolet radiation. The UPF of FKFC was 50.38 which shows better ultraviolet protection as compared to that of AKFC and CKFC. The KFC dyed wool fabric was reported to offer efficient UV protection [33]. The UV-Visible spectra of KFC showed (Figure 6) strong absorption in UV region which clearly indicates the reason behind the enhanced UPF of dyed fabrics.

In short, the functional properties of the dyed samples were found dependent on the color values of the dyed samples. This is obvious because the higher color values indicate the presence of higher amount of biomolecules on fabric which are responsible for imparting functional properties to the linen fabric. Hence, among the dyed samples, FKFC displayed the best antimicrobial properties, UV protection, and radical scavenging activity.

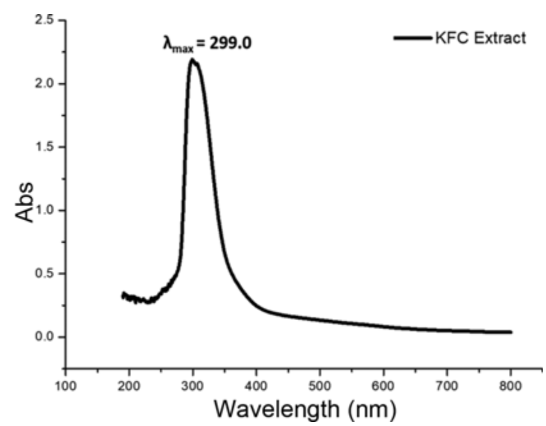

Figure 6. UV-Visible spectra of KFC extract.

Conclusion

The successful development of multifunctional linen was carried out using kapok flower colorant as a natural dye. Beautiful shades were obtained on linen using KFC. All the dyed samples showed excellent color strength along with satisfactory fastness ratings. The change in shade from reddish to brownish yellow and grey was observed depending on the presence of metal ions in the mordants. The KFC dyed linen demonstrated interesting radical scavenging activity and antimicrobial properties against both gram-positive and gram-negative bacteria. All dyed samples also displayed higher UPF values indicating efficient UV protection offered by such substrates. It can be concluded that the kapok flower extract is a functional dye when utilized in the presence of metallic mordants which can serve as a promising candidate of natural dyestuff and can be used in simultaneous dyeing and textile finishing to impart antibacterial, antioxidant, and ultraviolet protection properties to cellulose-based biological macromolecules.

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