

Physicochemical Characteristics and Dyeing Properties of Novel Cellulosic Fibers Derived from Sustainable Agricultural Waste

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Summary: Development of new innovative sustainable fibres from agricultural waste are in great demand these days. For the expansion of these fibres at commercial level, understanding about physicochemical properties of these fibres and specifically their dyeing behavior is essential. In this consequence, in the present study, a natural lignin-cellulosic fibers were extracted by a simplistic route, from *lotus silk*. The fibers were turn in to yarn by hand twisting and then natural as well as chemical dyeing was performed by the standard dyeing method established for cotton dyeing. Natural henna dye, C.I Reactive blue and indigo dye was used in the present study. Physico chemical properties and dyeing mechanism of the prepared fibers were studied and analyzed in detail in comparison with cotton cellulosic fibres. The lotus fibers were described for their organic construction and morphology by FT-IR spectroscopy and Scanning Electronic Microscopy respectively. Tensile strength, moisture regain and elongation percentage at break were also evaluated. The outcomes revealed that lotus fibers are cellulosic in nature same as cotton but with higher moisture absorption and lower crystallinity as compared to cotton fibers. Lotus fibers revealed higher color strength and color co-ordinates when compared to cotton fibers dyeing. Hollow and irregular fiber surface of lotus fibers along with higher moisture absorption and low crystallinity are the major reasons for higher chemical reactivity and absorption of dyes. The tensile strength and elongation observed in lotus fibers are suitable to be used these fibers in textile products. Lotus fibres from the agricultural waste can be a new source of sustainable textile products for future applications.

Keywords: Lotus roots; Lotus fibers; Henna natural dye; Reactive dye; Indigo dyeing, Sustainability.

Introduction

In this era , the micro plastic pollution of synthetic textile fibers [1], unsustainable raw materials and little to no recyclability of textile products are the major challenges. These issues diverted the research trend towards development of the innovative and sustainable fibers to enhance the comfort and luxury by sustainable means. As a result, numerous sustainable fibers from agricultural waste have been developed with enhanced comfort and luxury as compared to the conventional fibers [2]. In this context, cellulosic and/or lingo cellulosic fibers of botanical origin express a novel standard of sustainability and natural comfort [3,4]. Lotus silk from *Neelumbo Nucifera* [5] has emerged as one such kind of luxurious and innovative textile fibre recently for researchers [6,7] as well as for international fashion brands [8].

Lotus; an aquatic plant, belongs to the family of Nelumbonaceae. It is amongst the few natural microfibrils recognized in the world, due to its unique breathability [9], softness [10] and flexibility (more than worms silk), wrinkle free and silk like appearance [6]. Owing to the inherent insulating characteristics, lotus fabrics are adjustable to weather

conditions thus can be worn throughout the year [10]. The lotus, *Neelumbo Nucifera* is a persistent water-loving plant used all over Asia. In history, Lotus plant was termed as spiritual plant due to its healing abilities and wearer of lotus fabric sensed calm, peaceable, and contemplative. Lotus is famous in curing patients from pains, heart sicknesses, asthma, and lung issues [5]. The fiber made from lotus fiber is 100 percent organic as it does not involve any chemicals and oils for its extraction, processing and hence are environmental friendly [7,11]. The rapidly increase in demand of lotus silk products by global visitors and native consumers has progressively indorsed the lotus industry [5].

Lotus roots contain incessant fibers just underneath the skin in the vascular tissues in length wise direction. Lotus roots, after pulling the flowers, are usually left as surplus/waste and did not find any usage [12]. Different parts of Lotus plant and lotus fibers have been extensively studied for its medicinal properties [13], antimicrobial [14, 15], structural [16], fibre extraction [17], single and ply yarn properties [18]. Lotus fibers have also been used in manufacturing of green composites [19, 20].

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To the best of our knowledge, there is no study dealing with the color-lotus fibre interaction and color characteristics of this new promising fibre/fabric. To be used lotus fibres as a future luxurious textile raw material, understanding the dyeing behavior and mechanisms is of prime importance. Therefore, in this study, an attempt has been made to dye the fibres extracted from the lotus plant and study the detailed dyeing mechanism of this luxurious sustainable fabric/fibre with natural as well as chemical dyes. The data obtained was compared with that of cotton fibers to see its efficacy with other natural and/or regenerated fibers. A possible dyeing fiber mechanism is also proposed in this study. The physico chemical properties of the fibers were also examined to see its properties to be tailored as a future sustainable textile raw material.

Experimental

Materials

Lotus roots were obtained from the lotus grounds from Dadu region in Pakistan. All the petioles were cleaned and dip in water for 3-4 hrs. Before extracting fibers. The Sulphuric Acid (98%), NaOH (48^oBe), Meta-cresol, HCL acid, sodium hydrosulfite, Soda ash, Hydrogen peroxide, of analytical grade were purchased from Dae-Jung Korea. Sirrix Antox, Sifa stabilizer, leveling agent: drimagen E2R were purchased

from Archroma Pakistan LTD. Natural henna dye with purity of (98 + %) from Alfa Aesar was used to dye lotus fibers naturally. By chemical means, Henna lawsone chromophore is naphthoquinone and have a chemical structure of 2-hydroxy-1,4-naphthoquinone commercially named Natural Orange 6 [21] and in color index as C.I. 75480. Natural alum was used as a bio mordant in natural dyeing of cellulosic fibers. For synthetic coloration of cotton and lotus fibers, synthetic drimeren Navy C.L reactive and Denisol Indigo 30 liquid from Archroma Pakistan limited was applied.

Methods

Fibres withdrawal and twisting

Lotus fibers from the long petioles were extracted by cutting them from center and pulling in reverse direction to draw the spiny soft inner fibers and then turning lightly by hand at the same time to give the fibers enough strength. Peduncles containing natural cellulosic strands organized in spherical loop form that springs into 0.5–1 m lengths. The extraction was stopped till the fibers extracted from the lotus roots did not expand more. About 5-6 pieces of the peduncles were gripped in the hands at a time.

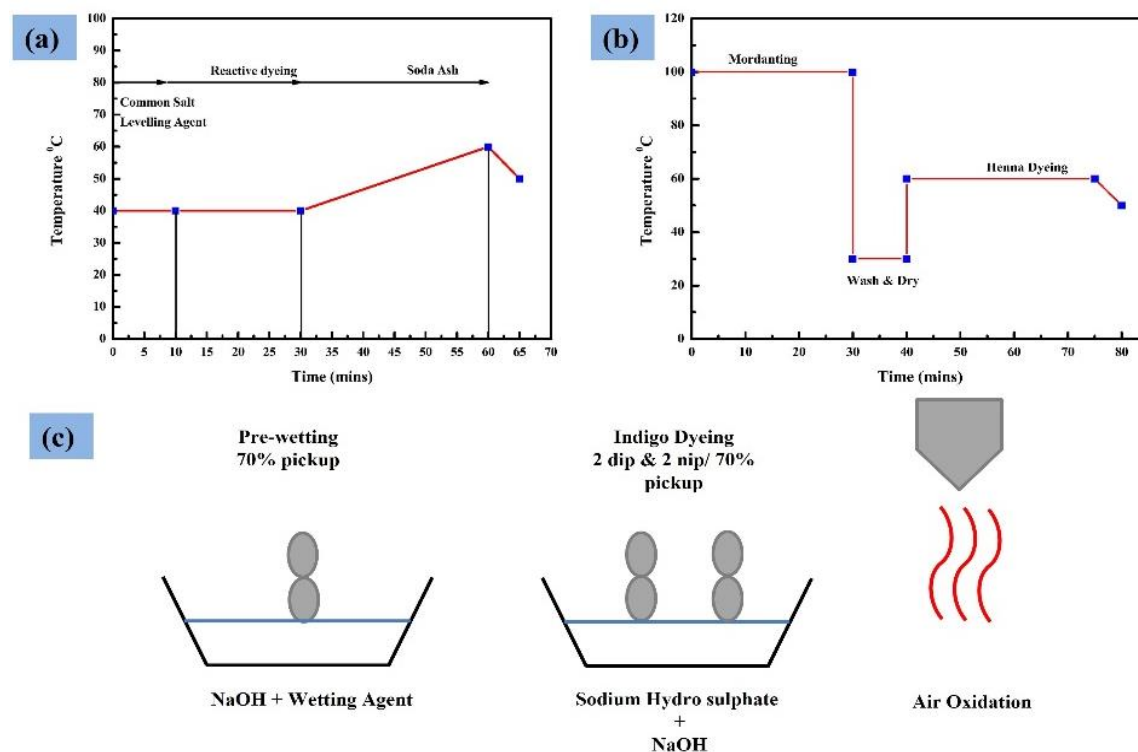


Fig. 1: Schematic diagram for the recipe for dyeing parameters of reactive (a), Henna (b) and indigo (c) dye.

Lotus and Cotton dyeing

The cellulosic cotton and lotus fibers were premordanted before the application of natural henna dye. After mordanting, henna dye was applied on both fibers by exhaust method on Rapid Labortex CO. LTD H-12c 5423. The schematic diagram for the recipe follow for premordanting and dyeing by exhaust method is given in Fig. 1 (a). The reactive dye on cotton and cellulosic fibers were applied by exhaust method with the standard recipe well established for cotton as given in Fig. 1 (b). The indigo dye was applied by pad 2 dip 2 nip method with 70 % pickup on Rapid Labortex CO. LTD. model P-BT 734 as the recipe is given in Fig 1 (c).

Characterization

To determine the fiber length, a pair of forceps and measuring tape was used. Fiber tensile strength and elongation% (ASTM D-3822: 07) were determined by universal strength tester, Titan 3-910. A gauge length of 75 mm, and a test speed of 300 mm/min were used for the test. Reaction of different substances like Sulfuric acid(H₂SO₄) (98%), hydrochloric acid(HCL), alkali: Sodium hydroxide (48 Be°); and organic solvent meta cresol on the lotus fibers was analyzed by examining 1 gram of fiber in a spotless dry beaker and reacting it with the above chemicals. Moisture absorption of the extracted fibres was measured by using the standard used for moisture measurement i-e ASTM D2495. According to this method, the lotus fibres samples were dry in an oven at 105 ± 2 °C for 1 hour and then weight is measured. After that, the conditioning of these fibre samples were performed for 3 hours at standard relative humidity i-e and temperature i-e at 65% relative humidity and at 20 °C temperature and weighed again (Fresh sample). The moisture regain was calculated according to the following equation:

$$\text{Moisture regain \%} = \frac{\text{Fresh sample weight} - \text{oven dry sample weight}}{\text{Fresh sample weight}} \times 100$$

Effect of solomatic bleaching was observed after treating lotus fibers with Caustic soda (48 °Be), and Hydrogen per Oxide 4% with stabilizer and sequesting agent at temperature of 60 °C for 40 mints. Surface images of lotus fibres were achieved by using Scanning electron microscope (SEM) (Zeiss, Switzerland) at sophisticated intensifications and functioning voltage of 5 and 10 KV. Dried lotus fibres were attached on aluminum disc by the assistance of carbon adhesive tape and covered with 5 nm gold palladium at a void range of 8–10 mb and unlocked the covered sample in argon gas to avoid any reaction before scanning. Gold covered samples were again

positioned inside the vacuum chamber of microscope for image investigation. Perkin Elmer FTIR apparatus was used to analyze the infrared spectroscopic (FTIR) spectra of lotus fibers. The spectral analysis were performed in range of 4000–500 cm⁻¹ at a firmness of 4 cm⁻¹.

For the detailed study of the color of the lotus twisted fibres, Reflection percentage values of individually colored sample were analyzed on a Data color SF600 spectrophotometer with illuminant D 65 and UV and specular factor involved. Every sample was analyzed at three dissimilar positions and the mean value was considered. With the help of these reflectance value at determined absorption, the color yield (K/S) was calculated with the Kubelka–Munk equation Eq. 1 [21].

$$K/S = \frac{(1-R)_{\lambda_{\max}}^2}{2R \lambda} \quad (\text{Eq 1})$$

In the above equation, R represents amount of the incident light reflected in reflectance %age; K shows the amount of light absorbed in terms of absorption co-efficient; and S is the amount of incident light scattered in terms of scattering co-efficient of dyes. CIE Lab coordination was used to determine the color characteristics of the fiber samples. According to this system, L* represents the lightness assessment of color (from 100 = white to 0 = black), higher lightness value means the lower the color produced by colored fabric. Moreover, a* and b* value represents the tendency of the color towards primary color. It means that, positive values of a* and b* shows that sample shade is towards redder and yellower tones whereas negative values of a* and b* are the represents of greener and bluer tones correspondingly. Furthermore, C* attribute shows the chroma or strength of color and h° relates to the hue angle. Washing fastness of the dyed cotton and lotus fibers were measured by ASTM-105:C03.

Results and Discussion

Lotus fibers physicochemical properties

The roots used for the extraction of the fibers are shown in Fig. 2 (a) are 1-2 feet long. In Fig. 2 (b), extraction and twisting of fibers are shown. The physicochemical properties of the fibers are shown in Table 1. Hand extracted and twisted fibers gave the optimum length of 0.5-1 meter. Lotus fibers shows same response to most common acids and alkalis as other cellulosic fibers, such as resistance to alkali and degraded/dissolved by acids. The whiteness index of the lotus fibers are 67 %. This gave the lotus fibers yellowish white color as shown in Fig. 2 (c), similar to

cotton fibers. However, this color of the lotus fibers vastly changed to white after solomatic bleaching as shown in Fig. 2 (d) that confirms the removal of non-cellulosic impurities from the fibers [16].

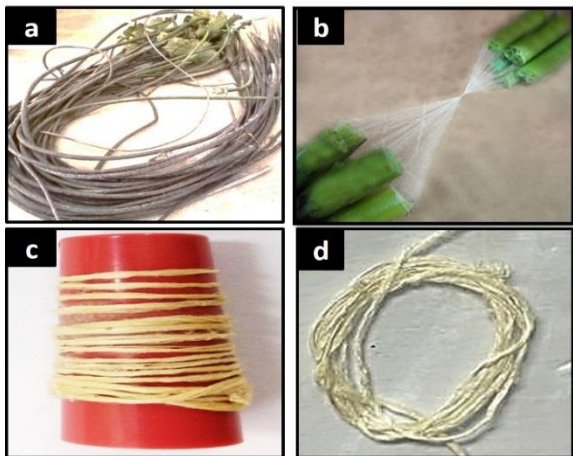


Fig. 2: (a) Lotus roots (b) fibers extraction and twisting (c) twisted fibers/yarn (d) fibers after solomatic bleaching

The moisture regain of the tested lotus fibers were 16 %, more than cotton i-e 8.5 %. This might be due to the smallest crystal size of the lotus fibers, i-e 2.5 nm as compared to the crystal size of cellulose obtained from cotton i-e 6.1 nm [22]. According to [23], small crystal size shows that fibers have large surface area which is the main reason that lotus fibers have much increased moisture as well as chemical absorptions of the fibers. The tensile strength and elongation of lotus fibers were 1.1 N for the count of approximately 20 Ne and 5.6 % respectively. Lotus fibers showed diverse interconnections and dissimilar diameter uniformity in different growing stages which might be the reason for low tensile strength of the tested roots. Low crystallinity of lotus fiber may also account for this value [24]. Although lotus fibers have enough strength to be used as a textile material and superior breaking elongation as compared to other cellulosic fibers which make it better than cotton to be used for comfortness.

Table-1: Physico chemical characteristics of the extracted lotus fibers.

	Lotus
Length	0.5-1 m
Moisture regain %	16.5
Elongation	5.6 %
Tensile strength	1.1
Whiteness index	67 %
resistance to Sulphuric acid	Dissolve completely
Hydrochloric acid	Fibers disintegrate
NaOH	No visible effect
Meta cresol	Turns into brown

Morphological and cross sectional structure of the fibers

The SEM images of the lotus fiber samples are shown in Fig. 3 (a, b and c) at different magnifications. The surface of the lotus fibers are rough having very fine fibers. This roughness and very fine diameter of the lotus fibers are responsible for their silk like softness and luxurious feel and high moisture absorption of the fibres. The cross sectional view of the lotus silk yarn is slightly round and irregular as shown in Fig 3 (d).

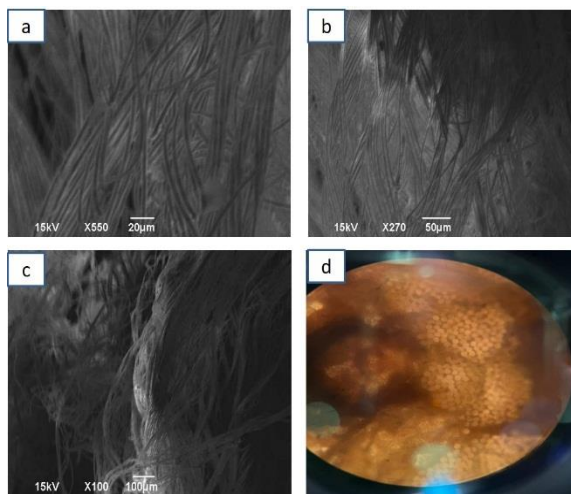


Fig. 3: (a, b, c) SEM and (d) cross sectional view of lotus fibers.

Fourier Transform Infrared Spectroscopy Analysis

Chemical structure and chemical groupings of lotus fibres was observed with FTIR and was compared with the chemical structure of most common cellulosic fibres i-e cotton fibers. FITR bands of lotus and cotton fibers is shown in Fig. 4. 1034 cm^{-1} peak observed is due to C–O (carbonyl) extending and vibration in lotus fibers. Moreover, 1241 cm^{-1} peak is because of CH stretching of cellulose, similar to cotton [12]. The band at 1733 cm^{-1} is responsible for carbonyl (C=O) vibration, arrives from non-cellulosic constituents such as lignin and hemicellulose of these cellulosic fibres [16]. This confirms that lotus fiber contains more lignin and hemicellulose than cotton. The wide-ranging peak positioned on 3148 cm^{-1} is OH extending and it is extremely subtle in case of lotus. Louts fiber follows more or less same trend as cotton fibers, which means that lotus fiber is chemically similar to cotton i-e cellulosic in nature but chemical constituents and composition is very less as compared to cotton fiber [12]. In case of lotus fibers, OH stretching is more pronounced at 3785 cm^{-1} .

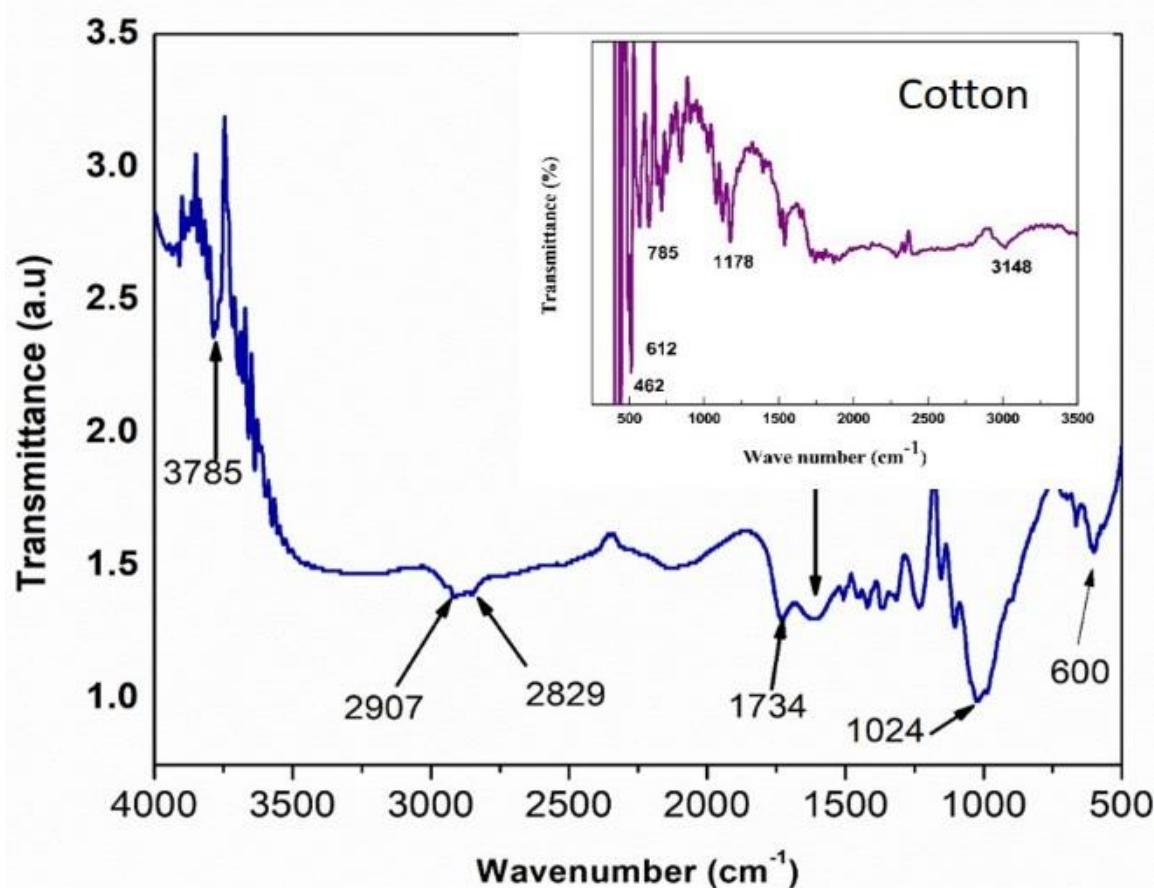


Fig. 4: FTIR spectra of Lotus yarn.

Dyeing Characterization

Lotus-henna dye interaction

The results for the dyeing characteristics of henna dye on lotus and cotton fibers are given in Table 2. The K/S of lotus fibers dyed with natural henna dye is higher (10) as compared to henna dyed cotton fibers (8.7). This might be due to high amount of hemicellulose and low crystallinity [24] of lotus fibers as compared to that of cotton fibers. This also confirms by the lower L^* values of the lotus fibers. The lower L^* values of the lotus henna dyed samples showed that lotus fibres have more strength of color as compared to cotton. It means that lotus fibers absorbed much more dyes as compared to cotton fibres. Subsequently, the reduction in c^* value of the lotus fibers designates an increased penetration of color inside lotus fibres. The low crystallinity with roughness on the exterior may contribute to the high immersion of the dye on the fibers. The possible reaction mechanism between henna dye and lotus cellulosic fibers are given in Fig. 5. Lawsone

chemically is a fragile acid and forms solvable salts in alkaline solution (Gujjar et al, 2014). Henna dye gives maximum absorption peak at 450 nm due to tautomeric form of lawsone chromophore in alkaline solution that gives rise to a distinguishing orange hue of the sample dyed with henna [25] as shown in Fig. 5. It is well established that, the ionic presentation of the lawsone dye and its typical color on particular fibre be determined by pH of the media and hence fibre – dye chemical bonding and reactivity. For cellulosic fibres, Henna due to its minor negative charge at pH 8-9 interact with hydroxyl groups of the cellulose resulted in hydrogen bonding and provided optimal color strength [26]. At more pH values, both develops anionic charge and resisted each other caused less dye aptitude. Additionally, the positive a^* and b^* values for both cotton and lotus fibers shows that the combination of red and yellow natures resulted to the orange color of the dyed fabric sample which is the illustrative of the natural color of lawsone pigment of henna dye. Mordanting before dyeing increased the bond strength between henna dye and lotus cellulose fibres.

Table-2: Dyeing characteristics of Lotus and Cotton fibers.

DYE	Fibers	K/S	L	a	b	c	h°
Henna Lawsone	Lotus	10	52.72	24.22	48.55	54.26	63.48
	Cotton	8.7703	55.55	27.67	46.90	54.45	59.46
Reactive	Lotus	8.8951	22.23	-4.89	-8.82	10.08	241.00
	Cotton	6.3826	25.27	-4.93	-10.28	11.40	244.39
Indigo	Lotus	10.6020	14.84	1.79	-10.10	10.25	280.08
	Cotton	8.0921	20.45	-0.07	-14.64	14.64	269.72

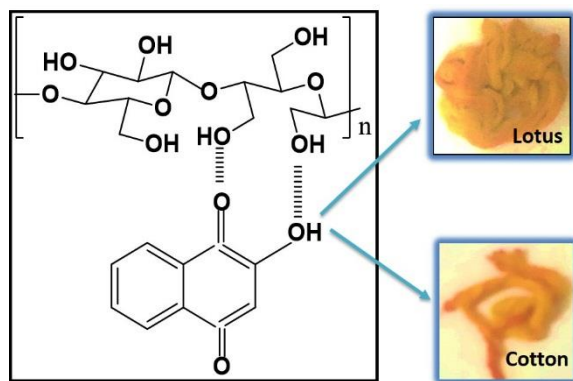


Fig. 5: Henna dye interaction mechanism with lotus fibres

Lotus –reactive dye interaction

The results for the colorimetric characteristics of reactive dye on lotus and cotton fibers are given in Table 2. The color strength of reactive dyed lotus fibers is higher (8.8) than cotton fibers (6.3). This might be due to high moisture content and Low crystallinity [24] of lotus fibers as compared to that of cotton fibers. The low crystallinity of lotus fibers i-e 48 % with rough microstructures on the surface as compared to cotton fibers (about 65%) [27] resulted in greater hygroscopicity and chemical reactivity of the reactive dye on lotus fibres. The possible reaction mechanism between reactive dye and lotus cellulosic fibers are given in Fig. 6. Reactive dyes contain a chromogen, that comprises of one or more exchanged aromatic rings which bring sulfonate/sulfonic acid groups. This group bind with a reactive group that assist the dyes to make a covalent bond with nucleophilic hydroxyl groups of the cellulosic material [28]. The dyes are applied under aqueous alkaline (e.g. Na₂CO₃) medium which deprotonate the hydroxyl groups of cellulose (Cell-OH) and produce the more strong nucleophilic, ionized hydroxyl groups. This in turn reacts with dyes to give dye-fiber bonds [29]. In the applied dye, a chlorine particle is replaced by a high molecular weight alkoxy group when reacting with cellulose due to nucleophilic substitution [30], as shown in Fig. 6. Electrolytes assisted in the dyeing process to overwhelmed the long-term revulsion forces operating between slightly negative charge of fibers and

negatively charged dye particles [31]. Consequently, promotes the extent of dye-fibre complex (i-e covalent bond formation of the dye to the cellulose of the material).

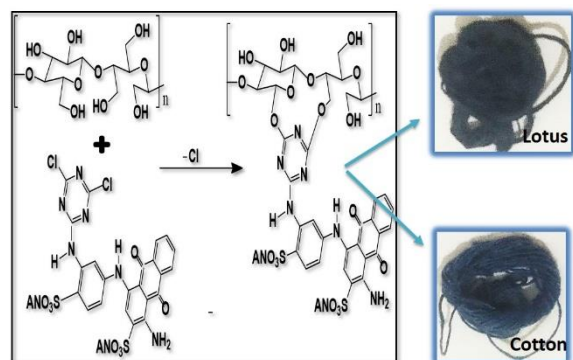


Fig. 6: Reactive dye interaction mechanism with Lotus fibres.

Lotus-indigo dye interaction

The results for the colorimetric characteristics of Indigo dye on lotus and cotton fibers are given in Table 2. The color strength of henna dyed lotus fibers is higher (10.6) as compared to henna dyed cotton fibers (8).the results of indigo dyeing on lotus are in accordance with the results obtained by other two dyes. The lotus cellulosic fibres interaction with indigo dye is shown in Fig. 7. Indigo is a type of vat dye which is practically insoluble in water. It must be reduced by a reducing agent such as sodium hydrosulfite to form the water soluble. Upon reduction, indigo becomes acid leuco indigo, which has partial attraction for cellulosic fibres. Addition of alkali forms alkaline leuco indigo which is enthusiastically soluble in water. This leuco indigo have more attraction towards cellulosic fibres and easily make an attachment to cellulose through van der Waals and dipolar forces [32], predominantly on the surface fibers. Once the leuco indigo has adsorbed in the fibres, it is oxidized into the original insoluble form by the air or any oxidizer used. This insoluble dye element is stuck within the chemical structure of the fiber, results in permanent coloration of the fibres. Unlike most of the textile dyes, the bond formed between indigo and fibres is mechanical and not chemical bond [33].

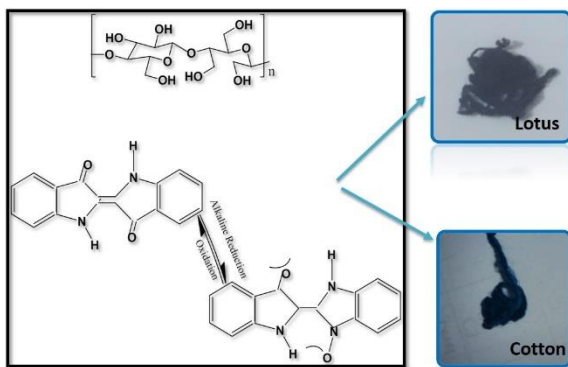


Fig. 7: Indigo dye interaction mechanism with Lotus fibres.

Washing fastness

The washing fastness results for lotus and cotton fibres dyed with Henna, reactive and indigo dye are shown in Table 3. The lotus and cotton fibres both gave good washing fastness and can be satisfactorily used as a textile raw material. Lotus and cotton fibres both are cellulosic in nature, make covalent bond with reactive dye and strong mechanical bond with vat dye. Moreover, the lotus fibres have 55-60 % amorphous in nature with larger surface area [23], which make it highly absorbent for water as well as dyes. Similarly cotton fibers also exhibit enough absorbency for good dye absorption due to 35-40% amorphous region. This absorbency of both lotus and cotton fibres make dye to absorb deeply in the chemical structure and resulted in good washing fastness.

Table 3: Washing fastness of Lotus and Cotton fibers.

	Lotus fibres	Cotton fibres
Henna dye	4-5	3-4
Reactive dye	4-5	4-5
Indigo dye	4-5	4-5

Conclusion

Lotus fibers were successfully extracted and converted to yarn from the wasted lotus roots. The lotus fibers are hollow fibers with circular/irregular cross section. Lotus fibers were successfully dyed with chemical reactive and indigo dyes as well as natural Henna dyes. The higher color strength and color attributes of lotus fibers as compared to cotton fibers is mainly due to its high moisture absorption and low crystallinity. Lotus fibres showed high elongation at break as compared to cotton fibres which make it more flexible and elastic fibre than cotton fibres. Moreover, lotus fibres showed high moisture absorption than cotton fibres which is due to its low crystallinity. The whiteness index and length of lotus fibres are enough to be used as a textile fibres for many applications.

Furthermore, chemical resistance of lotus fibres towards alkali and degradation with acid showed that lotus fibres are cellulosic in nature like cotton fibres. The present study showed that the lotus fibers can be used as one of the promising sustainable future raw materials to add some luxury to the products. The outcomes in the current study are likely to extend the knowledge of coloration behavior and mechanism of dye fibre interaction of new and innovative sustainable lotus fibers/products.

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