Chapter 49
Opuntia spp. in the Textile Industry

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Abstract The textile industry is one of the anthropogenic activities that most consume/pollute drinking water. Some of the synthetic dyes are highly toxic, mutagenic, and causing oxygen debilitation in water. The use of biocompatible chemicals started to be increased substantially over the last few years for its safety and health benefits to reduce environmental pollution. The use of eco-friendly dyes for textiles is highly appreciated because of the sustainability, biocompatibility, biodegradability, and various therapeutic advantage such as antibacterial, antifungal, and antioxidant properties. Plants belonging to the *Opuntia* genus are important natural sources that contain important natural dyes that can be used in the textile industry. The fruit contains dyes that belong to betalain skeletons and are responsible for yellow and red colours. These pigments can be used as a natural and safe alternative to synthetic colours. This chapter discusses the potential of *Opuntia* fruits' different constituents in the textile industry.

Keywords Natural colour · Indicaxanthins · Betanin · Textile · Antibacterial

1 Introduction

The textile industry is one of the biggest industries, globally worth over $450 billion, in terms of nominal sales (Euler Hermes Economic Research, 2016). It is one of the most jobs creating industries in the world, contributed nearly 7% of world production among all manufacturing sectors globally (Allwood et al., 2006; Herva

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et al., 2008; Desore & Narula, 2018). On the other hand, it is regarded as one of the biggest global polluters (De Brito et al., 2008; Hossain et al., 2018).

The production processes intensively use chemicals and water, which generate a high environmental negative impact. The chemical compounds such as dyes are partially utilized and washed away with the effluents (Islam & Khan, 2014). The non-biodegradable and carcinogenic components of these chemicals directly contaminate the water resources and affect the biological system (Hessel et al., 2007). Over 10.000 dyes and pigments are used in different industrial sectors, including textiles. The resulted washing of the dyeing process produces a large volume of liquid effluents contain (10–60%) the unreacted portions of these dyes and pigments with other chemical products and auxiliaries such as phosphates, nitrates that affect aquatic fauna, flora and human health (Berradi et al., 2019).

During the last years, the demand for environmental-friendly textiles and clothes, manufactured and distributed, minimizing the negative impacts of the toxic chemicals on the environment, has vigorously emerged from many stakeholders, including consumers (Lellis et al., 2019). Subsequently, steps have been implemented to make textile materials and processing more environmentally friendly, including dyes and their applications (Roy Choudhury, 2013). Green chemistry has emerged as a useful tool to make textile wet processing sustainable and develop alternative green and biodegradable chemicals such as biodegradable natural dyes (Gulzar et al., 2019).

2 Natural Dyes from *Opuntia*

Color is the main attraction of any fabric. The use of natural dyes for fabric dyeing has been known to humankind since immemorial times. Natural dyeing was found very encouraging, with its non-toxic, non-allergic, and non-carcinogenic soothing harmless effects. Moreover, these dyes are cost-effective and eco-friendly. Natural dyes work on cotton, silk, and wool. Their colours are relatively stable and eco-friendly because of no irritating effects on human skin. Different plant such as madder, walnut, weld, white onion, red onion, pomegranate, turmeric, barberry, and cactus have been reported as a source for natural dyes (Kasiri & Safapour, 2015).

The prickly pear or cactus pear are general names for different *Opuntia* species. It belongs to the Cactaceae family, which is native to the new world. Some species, mostly *O. ficus indica* cultivated in different regions of the world. The red pigments in *Opuntia*, known as betalains pigments, have a different range of colours, such as red-violet (betacyanins) and yellow-orange (betaxanthins) (Scarano et al., 2020). The relative proportions of betacyanins and betaxanthins are responsible for the enormous range of colors of the flowers of bougainvillea vines. There are 75 compounds described so far in the literature, 17 of them identified in different *Opuntia* species. However, betanin and indicaxanthin are the most common dyes distributed among *Opuntia* species (Khan & Giridhar, 2015; Check Aruwa et al., 2018).

Betaxanthins (Latin: beta = beet; Greek: xanthos = yellow) are condensation products of betalamic acid and amino acids or amines, respectively. Depending on the amino compound's particular structure, the maximum absorption of betaxanthins varies between 460 and 480 nm (Stintzing et al., 2002). Indicaxanthin is the most common and predominant pigment in yellow cactus pears (*Opuntia* ssp.). Betaxanthins-related structures are more diverse than the betacyanins (Castellanos-Santiago & Yahia, 2008; Cejudo-Bastante et al., 2014; Aruwa et al., 2018). In addition to visible variable colors range from yellow to reddish violet, betalains also showed interesting fluorescent properties. Most betaxanthins are weakly fluorescent in aqueous solution and are responsible for the fluorescence of the yellow flowers of *Mirabilis jalapa* and *Portulaca grandiflora*. On the other hand, betacyanins are non-fluorescent (Gonçalves et al., 2015). The discovery of the fluorescence phenomenon of betalains' in 2005 (Gandía-Herrero et al., 2005) opens up new applications in different fields, including the textile industry (Guesmi et al., 2012a). Metal cations can bind to betalains *via* complexation with the 1,2,3,4-tetrahydropyridine 2,6-dicarboxylic acid moiety of the betalamic acid portion molecule. The complex formation between betalain with Eu^{3+} forms a stable orange-color (Quina & Bastos, 2018).

Betalains are chemically labile in highly acidic aqueous media (pH < 1), and alkaline hydrolysis occurs at pH > 8, restricting most studies in aqueous media to slightly acidic conditions (4 < pH < 6). Even at pH 5, most derivatives' thermal decomposition occurs in a few minutes at 100 °C (Herbach et al., 2006a, b). Betalains are also subject to metal-ion catalyzed decomposition (Belhadj et al., 2017).

3 Dye extraction

Betalains are hydrophilic compounds and can be easily extracted using aqueous-alcoholic solutions or water. Due to the stability issues, usually adjusting the pH to acidic media and/or antioxidants like ascorbic acid be added during the extraction procedures. Ultrasonic-aided extraction was applied to improve the dye's extraction (Sivakumar et al., 2009; Guesmi et al., 2012b, 2013a, c; Yaqub et al., 2018; Salem et al., 2020). Highly pure betanin was extracted using ultrasonic from the reddish-violet fruit homogenate using water followed by fractional crystallization (Guesmi et al., 2013c). From the orange-yellow fruits, yellow pigments (indicaxanthin) was extracted with 80% aqueous ethanol, the partial purification completed using acidified C18 cartridge and ethanol as eluent (Guesmi et al., 2012a, 2013a). Acidified water was also used to enhance the dye stability during the extraction process (Guesmi et al., 2012b; Salem et al., 2020). Comparing the maceration extraction with the Naviglio extraction method, under the same extraction conditions (solvent, time, pH, ratio sample: solvent), the measurements indicated that the Naviglio method had higher dye content a short time. The extractor Naviglio® is a new solid-liquid extraction technology based on the suction effect (Salem et al., 2020).

Supercritical fluid extraction (SFE) with CO_2 as a solvent and EtOH/water as co-solvent was used and optimized for the extraction of betacyanins from red pitaya fruit (*Hylocereus polyrhizus*) peel. At optimal condition, the extraction yield of 4.09%, total betacyanins content of 25.5 mg/100 mL was recovered (Fathordoobady et al., 2019; Luo et al., 2014).

Antioxidants compounds such as ascorbic and isoascorbic acids as antioxidant agents and citric acid as a chelating agent were reported to enhance betalain stability and prevent degradation during the thermal process (Attoe & Von Elbe, 1982; Han et al., 1998; Herbach et al., 2006a, b; Wendel et al., 2015). EDTA was proven to increase the half-life time of betanin by 1.5 times (Pasch & Von Elbe, 1979),

4 Factors Affecting the Dyeing Process

During the dyeing process, the textile fibers are in continuous contact with all the dye liquor, and therefore the fibers gradually absorb the dyes. To obtain well-penetrated dyeing, some variables such as the dyeing temperature, pH, and the concentration of auxiliary chemicals must be carefully controlled. Different factors affect the quality of dye application to the textile materials, such as pH, temperature, tissue type, salt concentration, and mordants nature.

4.1 pH

The presence of nitrogen-containing and carboxylic groups in betalains chemical structures represent proton donor/acceptor centers. Protonation of the nitrogen atoms could occur in an acidic solution. The charge alteration of betanin upon pH changes was proposed (Simon et al., 1993; Filgueiras et al., 2000). The adsorption of the dye is mostly affected by the pH of the dyeing solution. The lower pH could form positive charges on the surface of fabrics such as wool and cause ionic interaction with the dyes' carboxyl groups. On the other hand, the dyes' thermal stability at lower pH may negatively affect the stability of the dyes (Guesmi et al., 2012b, 2013a, c). The optimum pH was recorded between 3.0–5.0. Fabrics such as wool, silk, and acrylics can be directly affected by changing the pH (Savolainen & Kuusi, 1978; Ganesan & Karthik, 2017; Scarano et al., 2020).

4.2 Salt Concentration

During the dye diffusion into the textile fibers, an electrostatic charge barrier that develops on the surface of the fibre must be overcome. In some cases, salt in large amounts is added to the dyeing bath to reduce the electrostatic charge on the surface of the fibre and even promote the dye's penetration. The addition of NaCl to the dyeing bath at different concentrations showed adverse effects. Increasing the salt (NaCl) concentration decreases the color strength of the dyed wool and acrylic fabrics (Guesmi et al., 2012b, 2013a, c).

4.3 Temperature

The dye/fibre interaction depends on many factors that control the dye diffusion into the fibre. The temperature is one of the important factors that control the diffusion process (Schönberger & Schäfer, 2003). Cotton and wool's dyeing process was reported to be effective at 80 °C for 60 min in the presence of mordants (Scarano et al., 2020). On the other hand, the color strength of dyed wool increase as the temperature increases up to 40 °C then it decreases drastically after 50 °C (Guesmi et al., 2013c). In modified acrylics, the optimum temperature of dyeing with betanin was found to be 50 °C (Guesmi et al., 2012b). The result can be a consequence of the losses of betanin stability or dye molecules' desorption from the fibers at high temperatures. The dyeing with pure indicaxanthin showed dye stability and increasing the color strength by increasing the temperature up to 80 °C (Guesmi et al., 2013a).

4.4 Ultrasound Irradiation

The use of ultrasound in textile dyeing showed color strength enhancement and reduced dyeing time (Vankar et al., 2007). The use of ultrasound in dyeing wool with betanin dye demonstrated that using a lower frequency of ultrasound irradiation increases dye exhaustion (25 kHz > 40 kHz > conventional heating) till 30 min and then it decreases slowly (Guesmi et al., 2013c, b). The ultrasound assisted in uniform dispersions in the dye on the fabrics and resulted in a higher depth shade (Ghorpade et al., 2000). The dyeing of the modified acrylic fiber with ultrasonic also improved indicaxanthin dye uptake (Guesmi et al., 2013a).

4.5 Effect of Bio/Mordant

The use of natural dyes has always been bounded to the use of the metallic salts as mordants to improve fastness properties (Cristea & Vilarem, 2006). Dyeing of fabrics with natural dyes often leads to problems such as narrow shade range and lower colorfastness of the dyed textiles. Attempts to overcome these problems have been mainly focused on the use of mordant. The term "mordant" is often used for chemicals, including metal with at least two or more valence that increases affinity between the dye and the fiber and is also used to change the hue of dyes (Shahid & Mohammad, 2013). The mordants' main function in textiles is to fix a dye to the fibers provide and obtain different colors. They also improve the take-up quality of the fabric and help improve color and light-fastness. In the mordanting process, firstly, the fabric is impregnated with the mordant, and then the dye reacts with the mordant for forming a chemical bond and attaching it firmly to the fabric during the dyeing process (Abuamer et al., 2014). The salts of di- and trivalent metals are the most common mordants, however in the last few years, biomordants such as chlorophyll a (Guesmi et al., 2013c) are tannic citric, tartaric, and acetic acids have been reported (Yaqub et al., 2018).

Different types of mordents such as potassium dichromate, copper sulphate, ferrous sulphate, and tannic acid were used as mordants during dyeing wool with an aqueous extract of red prickly pear fruits. The use of potassium dichromate and tannic acid improved the lightfastness of wool dyed with betalains pigments more than copper sulphate and ferrous sulphate. On the other hand, using tannic acid improved the washing fastness than other mordents. Also, the post-mordenting gives maximum colour strength when compared with pre-mordenting (Ali & El-Mohamedy, 2011).

The metallic mordant concentration has a negative effect on the colour strength (Ali & El-Mohamedy, 2011). This was supported by the results that showed metal cations (Fe^{2+}, Fe^{3+}, Sn^{2+}, Al^{3+}, Cr^{3+}, Cu^{2+}) accelerate betanin degradation (Attoe & Joachim, 1984). On the other hand, increasing the concentration of biomordant such as chlorophyll a was found to enhance the dyeability due to the release of Mg cation liberated at acidic pH (Diop Ndiaye et al., 2011; Guesmi et al., 2013c).

5 Natural Fibers

5.1 Cotton

Cotton fibers are the purest form of cellulose. The hydrophilic nature of the cellulose fibers is reflected from the abundant hydroxyl groups, which allow extensive intermolecular and intramolecular hydrogen bonding (Fig. 49.1), which result in various ordered crystalline arrangements. These hydroxyl groups are also critical for the chemical interaction with dyes (Hsieh, 2007; Park et al., 2010). The cellulose fibers usually exist in crystalline form. However, the amorphous structure also present. The diffusion of the dyes through the crystalline cellulose is difficult, and the final dyeing results depend on the amount of the amorphous phase present.

Different natural dyes such as Madder (*Rubia tinctorum*), weld (*Reseda luteola*), and coreopsis (*Coreopsis tinctoria*), have been used to dye cotton (Haar et al., 2013). The application of natural dyes to cotton usually associated with poor fastness (Cristea & Vilarem, 2006), and the use of mordant such as aluminum acetate, copper sulfate, and potassium dichromate are highly recommended (Haar et al., 2013).

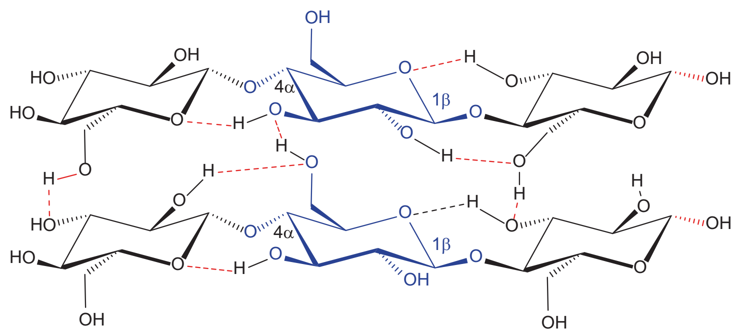

Fig. 49.1 Cellulose linear structure shows inter- and intera-hydrogen bonding (dashed line)

The green synthesis strategy was established for textile finishing, aiming at the development of antimicrobial-printed cotton. A natural dye extract obtained from red prickly pears was applied with or without mordants (tannic acid) to chitosan-treated cotton. The result that cotton fabrics containing chitosan had higher color strength than untreated fabrics with or without the mordant. The printed fibres also showed good antibacterial activity (Hebeish et al., 2011, 2012).

5.2 *Wool*

Wool has a typical protein structure build-up from amino acids linked together through peptide bonds. Parts of the protein chain exist in crystalline form and have an α-helical structure stabilized by intramolecular hydrogen bonding between NH and CO groups. The similarity in chemical structures between the wool and natural dyes from *Opuntia* proposes strong intermolecular hydrogen bonding and electrostatic interactions, and these are reflected from the presence of donor and acceptor active groups among each structure (Fig. 49.2). The dye's affinity to the wool fabrics is expected to be good and can be improved using temperature, sonication, mordant and low pH.

The colour strength and the dye uptake by the wool fabrics have exhibited high values. Good fastness properties of the dyed fabric were achieved using different mordants. This depends on the groups capable of forming hydrogen bonding and a metal complex. The higher the number of these groups, the lower the magnitude of the dye removal. Thus, mordanting with tannic acid gave the highest washing fastness, followed by potassium dichromate. The dyeing process's optimum conditions were observed at pH 5 and temperature 100 °C (Ali & El-Mohamedy, 2011).

Rubbing fastness was high in the mordanted samples. The lightfastness of samples mordanted with potassium dichromate and tannic acid was found to be more than samples mordanted by copper sulphate and ferrous sulphate comparing, while the unmordanted samples showed inferior fastness (Ali & El-Mohamedy, 2011).

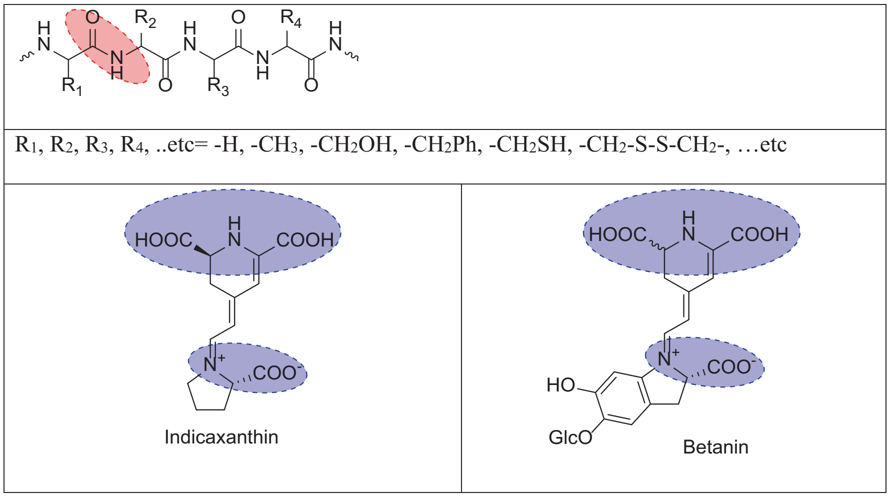

Fig. 49.2 Active chemical sites in indicaxanthin and betanin

Antimicrobial wool fabric activity showed a high inhibition zone at 2% of the dye concentration against *Bacillus subtilus* and *E. coli* (Ali & El-Mohamedy, 2011).

A stable color was achieved by the addition of ascorbic acid and different mordants. The dry cleaning fastness was excellent, while the wash fastness in neutral detergent was more effective than alkaline detergent. The perspiration fastness was increased by the mordanting method. On the other hand, the lightfastness was inferior. The *Opuntia* extracted dye improved the antibacterial and UV-B protection properties of wool fabrics (Lee et al., 2006).

Due to the natural variation of natural products among natural resources, the dyeing with total extracted dyes showed a reproducibility problem (Türkmen et al., 2004) using single pure compounds such as betanin or indicaxanthin is preferable. The dyeing of wool fabric using purified betanin as natural dye and chlorophyll a as biomordant showed an increase in the colour strength values with increasing the concentration of biomordant. The depth of shade reached its maximum value at 45 min. On the other hand, the optimum temperature for dyeing was found to be 40 °C, and the optimum pH was 4.5, at acidic conditions, the Mg is released from the porphyrin ring of chlorophyll a and enhance the dyeability of the betanin. The fastness properties of dyed wool against washing, light, dry and wet rubbing showed an improvement. The mordant samples showed good fastness properties than the un-mordanted ones (Guesmi et al., 2013c). The same group studied the dyeing of wool with indicaxanthin. The dye's adsorption on the wool was found to be optimum at pH 4 and temperature between 70–80 °C. The use of mordant directly affected the dyeing process. The colour strength increased in the order of the dyeing using $KAl(SO_4)_2 > MnSO_4 > CoSO_4 > FeSO_4 >$ none $> ZnSO_4 > CuSO_4$. Un-mordanted samples have good properties of water and washing fastness.

Mordants KAl $(SO_4)_2$ and $CoSO_4$ were found to give good lightfastness (Guesmi et al., 2012a).

The wool fabric dyed with indicaxanthin showed interesting fluorescence properties. Photoluminescence was recorded at 576 nm. The samples showed a weak response to light irradiation but open up a new field of the corporation of natural fluorescent dyes into textile fabrics (Guesmi et al., 2013d).

5.3 Silk

Fibroin is the structural protein of silk fiber; it has a typical protein structure where amino acids are linked together by peptide bonds. The presence of active carboxylic and amino groups enhances the interaction with dyes and improves the fastness. The aqueous extract of the red prickly pear fruit was used to dye silk fabric. Myrobalan is a rich tannin extract used as a biomordant and copper sulphate (Ashrafi et al., 2018; Jain & Vasantha, 2016). The post-mordant-processed fabric with $CuSO_4$ showed higher colour strength when compared to myrobalan. The superiority of CuSO4 in improving the dyeing process may reflect the metal complex's importance than tannin effects. The antibacterial properties of dyed fabric without mordant showed better results than pre-mordant and post-mordant samples, and the antibacterial activity decreases gradually during further washing cycles (Ganesan & Karthik, 2017). Treatment of Silk fabric with chitosan improved *Opuntia* dye interaction and displayed a good colour strength and high antibacterial activity (Julia & Pascual, 2000; Hebeish et al., 2012).

6 Synthetic Fiber

Acrylic fiber is a synthetic polymer composed mainly of acrylonitrile unit. It has a strong hydrophobic character and low dye affinity. Pre-treatment with hydroxylamine hydrochloride (Fig. 49.3) was proposed to improve the fibers' dye-ability (El-Shishtawy & Ahmed, 2005). The modified acrylic fibers were subjected to dyeing with indicaxanthin and betanin dyes purified from *Opuntia* species (Guesmi et al., 2012b, 2013a). The optimal conditions for dyeing with indicaxanthin dye were found at 80 °C for 30 min and pH 3. Sonicator improved the dye uptake and

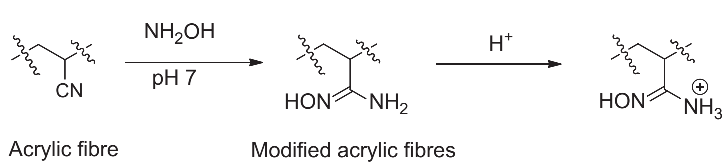

Fig. 49.3 Introducing active sites in acrylic fibres by reaction with hydroxylamine

color strength and gave a higher depth shade at a lower dyeing temperature (Guesmi et al., 2013a). On the other hand, the optimal conditions for betanin dye were found at 50 °C for 45 min and pH 5. Using $CoSO_4$ was found to give good lightfastness. Nevertheless, the un-mordanted samples showed good properties of water and washing fastness (Guesmi et al., 2012b).

7 Functional Textiles

Cellulose and protein derived natural fibres provide suitable conditions for bacterial growth and multiplication and result in unpleasant consequences such as foul odors, mold and mildew strains, discoloration, and loss of functional properties (Hebeish et al., 2011). The control of microorganisms' growth on textile fabrics using non-toxic chemicals, environmentally benign and renewable materials is one of the key issues that merit important consideration of the green synthesis strategy (Singh et al., 2005). The use of natural dyes is an important source of antibacterial agents and can play dual roles.

Dyeing of wool fabric with prickly pear 2% dye showed antibacterial and anti-fungal activities, the highest inhibition zones of 27.9, 18.3, 15.0, and 13.5 mm against *Bacillus subtilus, Pseudomons aeruginosa, Staphylococcus aureus, and Escherichia coli* were recorded (Ali & El-Mohamedy, 2011). The antibacterial activity of silk fabrics dyed with betalains from *Opuntia* showed antimicrobial activity even after 15 washes against *S. aureus* and *E. coli* (Ganesan & Karthik, 2017). Dyes from *Opuntia* were also used for printing cotton and silk that were pretreated with chitosan and tannic acid. The results showed a high growth reduction of microbes, and the growth of *S. aureus* and *E. coli* was inhibited completely (Hebeish et al., 2011).

8 Conclusion

In addition to high safety margins, affordability, and availability, natural dyes have multifunctionality, i.e., UV-protection and antibacterial activity. Total extract of *Opuntia* dyes and purified constituents such as betanin and indicaxanthin dyes were found to compete with synthetic dyes in color depth shade and fastness properties. The chemical structure of betalains is unique, where carboxylic and amino functional groups are present. This feature gives an advantage of strong and direct interaction with textile fibers. The dyeing with *Opuntia* dyes can be improved using different factors such as pH, ultrasonic, temperature, and bio/mordants, proper adjustments of these factors enhance the dyeability and fastness of the dyes on the different type of textiles. The nature of the natural dyes' chemical structures and stability is one of the major drawbacks reflected from the poor wash and light fastness of textile fibers. More research will be highly appreciated in this regard, considering the importance of using a standardized total extract. Although the industrial application of natural dyes, including *Opuntia*, is not among the major textile industry, makers' sco lightfastness scale and handmade industry look for more safe and affordable natural dyes.

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