Chapter

Review on Natural Dyes for Textiles from Wastes

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Abstract

Agriculture and food processing industries generate a large amount of organic waste that still contains colouring pigments. Their sustainable use in dyeing textiles will expectedly solve the problem of their disposal. Some studies involving the use of agro and industrial waste have been documented in this chapter to provide a guideline for further research. Information on some selected wastes that have been used in dyeing of textiles by several authors have been listed, their composition and production details highlighted and their use is explained systematically. The documented studies have been placed in the form of case studies to highlight the different approaches of the authors for explaining the effectiveness of such wastes as a source for textile colourants. As reported in some studies, the extracted dye from the waste doubled up as a mordant itself. Most studies also indicate good dye-ability with appreciable fastness with respect to the textiles dyed with such wastes. Some wastes have also reportedly rendered antibacterial and sun/light protective properties to the dyed fabric.

Keywords: natural dyes from agro-waste, natural dyes from industrial waste, natural dyes from flower waste, natural dyes from fruit waste, sustainable natural dyes, waste utilisation

1. Introduction

Natural dyes have the advantage of having a renewable source and are biogradable in nature having low environmental impact [1], but are still associated with problems of poor to moderate colour fastness [2], absence of standardised procedure for extraction and application [3], non-reproducibility of shades [2], pollution caused by use of metallic mordants [4], high energy consumption during extraction and exhaust dyeing [5] and, high cost [6]. Natural dyes also have a significantly lower affinity for fibres resulting in lower dye-exhaustion from the dye-bath on to the fibre surface [7]. Several efforts have been undertaken all over the world to address these shortcomings of natural dyes and find suitable alternative sources in view of the tremendous environmental advantages that they offer. Moreover, the content of the colour component in most natural dyes is a tiny percentage of its total solid weight and large quantities of the dye source are needed to colour small quantities of the textile material. For this, enormous amounts of the dye source has to be procured, which may lead to overexploitation of natural resources; specifically, if they are from the vegetable or animal origin. This would also threaten some endangered vegetal and animal species [8]. In this respect, use of a waste material that are available at no or little cost to dye textiles would expectedly make the process of natural dyeing cost-effective and at the same time bring about increased sustainability in the textile dyeing operations.

Natural dyers have long used numerous parts of trees for dyeing textiles due to their rich tannin content, which can act as a dye or mordant [9, 10]. The use of different parts of a plant/tree to get dyestuff leads to the question of sustainability particularly if the dyestuff-source needs to be renewed and made viable for recurring usage. Production of the plant material for extraction of natural colourants must not compete with farming of crops for food production. Also, at present the production of natural dyes by direct farming, results in substantially high specific cost per kilogram of plant materials and correspondingly per kilogram of the dyed material [11]. The cost can be lowered by the use of by-products from agriculture and food industry, and wastes from forestry. Fruit of trees could be considered the most sustainable in this respect, with new produce grown each year and processed for value-addition and uneatable parts of the fruits like peels and seeds thrown away as waste. Agro-waste such as peels, shells, seeds, etc., are rich sources of pigments and natural dyes can be obtained from them. Sources such as pomegranate peels [12, 13], onion peels [14], leaves of eucalyptus [15], walnut husks [12, 16], walnut shells [6, 17, 18], almond shells [19], peanut shells [20], beetle nut [21], indigo seeds [22], mango seeds [23], tamarind seeds [24], safflower seeds [25], strawberries [26], beetroot [27], discarded/used marigold flowers [28], etc., have been explored for their use as natural colourants. Such studies are however limited and inadequate.

Use of fallen leaves and branches having a rich dye content is another sustainable approach. While it's possible to collect roots and bark from trees without harming the plant, it requires skill and detailed knowledge of a tree's structural and nutritional needs and thus, is not recommended. A healthier practice would be to collect these items from trees that are already harvested for manufacturing or for timber control.

At the same time, disposal of waste generated as by-products by the agriculture, forestry and industries pose a serious threat to the environment. With the environmental awareness and scarcity of space for landfills, wastes or by-product utilisation becomes an attractive alternative to direct disposal.

Thus, a promising concept for production of natural dyes with lowered cost could involve the use of plant materials discarded as waste/by-products from different sources.

1.1 Waste from agriculture

Agricultural wastes are non-product outputs resulting from various *agricultural* operations. Most of these wastes are discarded by burning and dumping or piling into landfills. This on one hand leads to wastage of resources, and on the other hand their disposal poses a serious threat to the environment. Effective utilisation of this agro-waste utilisation would not only address disposal issues, but also provide additional income to the farmers or processing industries that generate the waste [29].

Agro-wastes may include, among other materials, harvest-*wastes* including barks, flowers, fruits, leaves, roots, woods and seeds yield dyes.

Some studies in this area using agro-waste as natural dyes have already been undertaken. Wool yarns have been dyed with sawmill waste of regional dyewoods like Kansas black walnut (*Juglans nigra*), osage orange (*Maclura pomifera*) and eastern red cedar (*Juniperus virginiana*) [30]. Extracts from plants such as *Albizia coriaria*, *Butea monosperma*, *Callistemon citrinus*, *Tagetes erecta* L. and *Camellia sinensis* (oolong tea) have been studied and recommended as sources of natural dye for dyeing cotton fibres [7, 30–35]. Agricultural waste such as eggplant and mangosteen have also been reported as effective raw materials for the production of natural dyes [16, 36]. A wide range of shades with good fastness have been obtained using natural dye extracted from pomegranate peel extracts [4, 12–14, 36, 37]. Such peels have also been reported to exhibit antimicrobial properties [38, 39] and good UPF properties [40]. Onion peels/scales have been used for dyeing [11, 41–45]. Dyeing textiles with turmeric rhizome is well known, but use of the turmeric leaves discarded as waste to dye silk and cotton has also been explored in search of newer alternative and novel source for natural dyes [46]. Banana peel, an inexpensive by-product/waste has been used to dye cotton with excellent antibacterial activity and high UV protection properties [47, 48]. Banana floral stem sap has been used for dyeing cotton knitted fabric with good colour fastness properties barring light fastness [49]. Cotton has also been dyed with banana leaves [47]. Extracts from banana fruit peel has been used to dye mordanted cotton and silk [48]. Natural dyes from orange peel extract applied to wool fibres have a UV protective effect that is six times higher than that achieved with synthetic dyes [50]. Pashmina shawl with very good washing and light fastness has been dyed with petals of the saffron flower, which is thrown as a waste after harvesting [18].

1.2 Waste from forestry

Forestry operations can generate large quantities of brash wood chip, bark, fallen branches and leaves due to deforestation, woodland clearing and firebreaks from plants such as deodar, cedar, jackfruit, walnut and eucalyptus, etc., that can be utilised for textile colouration. Waste is also generated from timber industry in the form of saw dust, timber slash, extracted liquor from timber control and mill scrap. These residues are mostly used as a renewable energy source and in the making of bio-mass fuel and contain natural colouring pigments. Fallen leaves enrich soil, they can pose a threat to the environment as they can decay into harmful methane gas. Many of these leaves contain pigments that can dye textiles.

Some studies have been undertaken to dye textiles with forest wastes. Eucalyptus liquid waste from lumber steaming has been used by to dye cotton [51]. Sawmill waste from Kansas black walnut, osage orange, and eastern red cedar timber have been used as dyestuffs for textiles [29]. Waste bark extracts from Turkish red pine (*Pinus brutia* Ten) have been applied on cotton, flax, wool, tencel, polyamide and acrylic [46]. Silk and wool yarns have been dyed with extract from mango tree bark [52]. Cotton was dyed with colour extracted from eucalyptus bark [53, 54]. Silk and wool fibres have been dyed with using eucalyptus leaves [15].

1.3 Waste from industry

The food industry produces large quantities of liquid and solid wastes that are potential pollutants with associated disposal problems. These residues are used either as animal feed or are disposed or composted. However, wastes from pressed berries, grapes, distillation residues, wastes and peels from vegetable processing containing dyestuff are available at almost free cost.

Some studies on the use of plant waste from the food and beverage industry could be suitable as a source for the natural dyeing of wool fibres [11, 55] have been reported.

There is a need to explore the new sources of natural dyes. Although literature has reported use of agro, forest and industry wastes in dyeing of textile, research in this field is limited, scanty and sporadic. Also a common database on wastes used as natural dyes in dyeing textiles is not available. This chapter thus deals with few wastes that can be used as potential sources for textile colourants. Many of them are associated with multi-functional properties. Research already undertaken by people have been discussed in the form of case studies to provide a clear understanding of the waste source, its related composition and chemistry and effectiveness in imparting colour to textiles, sometimes with additional functional features.

2. Case studies for specific natural dyes from waste

2.1 Grape

Kingdom: Plantae; Family: Vitaceae; Botanical Name: *Vitis vinifera* L.; Common Name: Grape or *angoor* (Hindi); **Part used for dyeing**: Grape pomace, comprising of seeds, stalks, grape marcs, lees and stems.

2.1.1 Occurrence

Grapes belong to Vitaceae family and is cultivated mainly in the Mediterranean region, central Europe, south-western Asia, north and south Germany and eastern and northern Iran. About 5000–10,000 varieties of grapes are known of which only a few are of commercial significance and are used for eating (as a fruit) and for wine making. The fruit is classified as a berry and in the wild variety, are 6 mm diameter that ripen to give dark-purple to blackish colour. In cultivated plants, the berries are usually much larger, up to 3 cm long, and can be green, red, or blackish-purple.

2.1.2 Chemical constitution

Grape pomace potentially constitutes an abundant and relatively inexpensive source of polyphenols like phenolic acids, flavonols and anthocyanins and the colouring component in it is tannins (procyanidin, prodelphinidin, glucosylated procyanidin and gallated procyanidin) [56, 57].

2.1.3 Waste generated for grapes

Grape is one of the world's largest fruit crops and the world wine manufacturing converts currently 10–25% of raw grapes into residues, known as pomace, mostly comprising of seeds, stalks, grape marcs, lees and stems [58]. Because of a low extraction during wine making, the solid residues still retain high levels of condensed tannins [57]. Though rich in polyphenolic compounds; the use of grape pomace or waste has been restricted to animal feed and fertilisers and in some cases they are simply dumped.

Case study 1

Objective of the study: Dyeing of wool, silk, acrylic and cationized cotton and polyamid fibres with aqueous-extract of grape pomace [58].

Baaka et al. [58] have explored the use of a by-product, pomace, abundantly available at relatively inexpensive costs, to dye natural fibres like cotton, wool and silk; and synthetic fibres like polyamide and acrylic using sulphate salts of alum and ferrous as mordants. Since cotton and acrylic are associated with poor affinity to dyes, they were subjected to additional pre-cationization process prior to mordanting for improving dyeability. Surface colour strength of the dyed fabric was found to improve with use of metallic mordants, and good fastness properties to rubbing (4–5), wash (4–5) and light (4) were obtained. Authors have concluded remarkable improvements in the UPF of the fabric dyed with grape pomace in the presence and absence of metallic mordants (**Table 1**).

Case study 2

Objective of the study: Dyeing of silk with anthocyanins extracted from grape pomace [59].

Mansour et al. [59] have optimised the method for extracting natural dye from grape pomace, a winery waste and exhaust-dyeing silk with it using tannic acid ($C_{76}H_{32}O_{46}$, Aldrich) as a natural mordant using the meta-mordanting process.

Reporting that hydration of plant materials is important to enhance the overall extraction rate, the authors have used ethanol containing a weak acid like acetic acid to extract the more labile acylated anthocyanin pigment from *Vitis vinifera* L., leaves and have reported 60:40 as the optimal ratio of water-acidified ethanol mixture. Noting that anthocyanins are sensitive to temperature, a liner increase in yield was seen with rise in temperature and 32°C was found to be optimal for extraction. A time of 38 h was sufficient to swell and hydrate grape pomace permitting diffusion of the solvent into the plant matrix to leach out the pigment.

Mordanting with tannic acid at 95°C showed an increase in K/S value, but it lowered the L^* value. Positive a^* and negative b^* coordinate values of silk dyed with pomace extract indicates redness and blueness respectively and thus the extract rendered a purple and violet shade to the dyed silk fabric. The use of tannic acid at 85°C however increased the b^* coordinate values giving a bronze shade. Due to the ionic character of the –OH groups in anthocyanine, the dye from pomace extract probably reacted ionically with the pronated terminal amino groups of the silk fibre in an acidic pH, giving high surface colour strength. Thus, dyeing at pH 2.5 was considered optimum. The authors have also found that dyeing without any salt gives highest surface colour strength.

Through the use of Box–Behnken model of experimental design in response surface methodology (RSM), the authors have concluded that the optimal depth of shade on silk can be obtained at (a) 2.62 (pH), (b) 83.6°C (dyeing temperature), and (c) 0 gL^{-1} (salt concentration).

The dyed silk showed good light, wash and rubbing fastness that improved through the use of a mordant. The use of tannic acid as a mordant specifically improved the light fastness of silk fabrics dyed with grape pomace and gave darker shades probably due to the effective cross-links formed between the silk fibres and anthocyanin of the dye facilitated by the high molecular weight phenolic hydroxyl group contained in the tannin (mordant).

Fibres	Control (with dyeing and mordanting)	Fabric dyed with grape pomace extract without the use of any mordants	Fabric simultaneously dyed with grape pomace extract and mordanted with aluminium sulphate	Fabric simultaneously dyed with grape pomace extract and mordanted with ferrous sulphate
Cotton	5	25	40	35
Wool	5	30	50^+	50^+
Silk	2	18	24	40
Polyamide	20	26	40	35
Acrylic	25	30	30	50^+

Table 1.
UPF rating of fabric composed of different fibres before and after dyeing with grape pomace extract with and without metallic mordants [58].

2.2 Pomegranate rind/peels

Kingdom: Plantae; Family: Lythraceae; Botanical Name: *Punica granatum* L.; Common Name: Pomegranate or *anar* (Hindi); **Part used for dyeing**: Outer rind/peels of the fruits.

2.2.1 Occurrence

Pomegranate belongs to Punica Genus which has two species, the most important being *Punica granatum* L. and is found in the region extending from Iran to northern India. It is a fruit bearing deciduous and thorny plant which bears cone-shaped red flowers singly, or in clusters.

The edible part of the pomegranate fruit (50%) consists of arils (40%) and seeds (10%). The remaining 50% of the total fruit weight comprises of tough, leathery and yellow or deep pink/red coloured peels.

2.2.2 Chemical constitution

The colouring matter from pomegranate peels consists of flavonoids (mostly flavan-3-ols or granatonine) and condensed tannins (26% of the total chemical composition) [60]. It also contains complex polysaccharides and many more compounds such as phenols, proanthocyanidin and ellagitannin (punicalagins and its isomers), low amounts of punicalin, gallic acid, ellagic acid, and ellagic acid glycosides [61].

2.2.3 Waste generated for pomegranate

The world pomegranate production amounts to approximately 1,500,000 tons [62]. The peel of the fruit consisting of pericarp/rind/hull comprises approximately of 60% of the weight of the fruit and is considered as an agro-waste though it is

Case study 3

Objective of the study: Dyeing of khadi cotton with aqueous extract of pomegranate rinds [40].

Sinnur et al. [40] have used pomegranate rinds as a natural colourant to dye cotton khadi fabric using single and double mordants. They have found that 15% myrobolan (*harda*) along with potash aluminium sulphate when used as mordants in 50:50 ratio offers maximum K/S value and renders good overall good colour fastness than any other combination of the two mordants. The authors have also reported the standardised conditions of dyeing khadi cotton fabric with aqueous extract of pomegranate rinds to be (a) 60 min (dyeing time), (b) 80°C (dyeing temperature), (c) 1:30 (dye-bath MLR), (d) 9.0 (dye-bath pH), (e) 20% (dye concentration) and (f) 3% (common salt).

Through the results of the FTIR and AAS studies, the authors have postulated the possible corresponding reaction mechanism resulting in the formation of large complexes between the mordant, fibre and the dye. The FTIR studies have indicated the presence of aluminium in dyed cotton khadi fabric, thereby confirming the formation of a complex between the cotton fibre, aluminium sulphate mordant and dye from pomegranate rinds together with the presence of some zinc and sulphur.

The dyed cotton khadi fabric shows medium to good level of ultraviolet protection as the UPF rating increases from 5 for the control untreated sample, to 15–20 for the cotton dyed with pomegranate rinds.

Case study 4

Objective of the study: Precipitate of dye extract from pomegranate peels has been used to dye wool yarn, cotton jersey and flax fabric [14].

For ease in use, transportation and storage, extracts can be concentrated either by evaporation of the aqueous extract or by membrane concentration techniques. The former evaporation technique uses considerable amounts of energy and the latter membrane concentration technique calls for a huge capital investment.

To overcome both the disadvantages of source-substitution and poor dye-concentration, Ali et al. [14] have used agro-waste like pomegranate peels, onion peel and Canadian goldenrod in the form of concentrates prepared by energy-friendly precipitation method to dye wool yarn, cotton jersey and flax fabric. Extracts of these dyes from agro-waste have been precipitated as Al-based dye lakes/concentrates using alumnium sulphate. On an average, the precipitate yielded 5% of the original plant weight as dry lake, the concentration of total phenolics (TPH) in this dry solid mass ranged between 20 and 40% and aluminium content varied between 3 and 5% (w/w).

The authors have shown how these Al-based dye lakes that are soluble in oxalic acid, can be used as direct dyes in the presence of aluminium-salt or iron-salt as mordants. Though the associated colour strength/depth of colour of the lakes was found to be lower, but the chroma of the dyeings (chromaticity) was higher. The authors have assigned the reason for the production of darker and more brilliant dyeing (higher b* coordinate values) by the direct use of precipitated extracts to the use of purified condition of the extract that contains more colour in the same amount of plant material.

a rich source of antioxidants, phenols, flavanoids [63]. Reportedly the peels also possess antibacterial, antifungal, anti-cancer and anti-inflammatory medicinal properties and have mostly been used as cattle feed.

2.3 Onion Peel

Kingdom: Plantae; Family: Amaryllidaceae; Botanical Name: *Allium cepa* L.; Common Name: Onion or *pyaz* (Hindi); **Part used for dyeing**: Outer papery skin of the fruit.

2.3.1 Occurrence

Onion, botanically known as *Allium cepa* L., onion belongs to the Liliaceceae family. The plant originated in the middle-east and in India it is cultivated extensively in South India and Bengal. The onion plant is a perennial herb growing to about 1.2 m in height, with 4–6 hollow cylindrical leaves. The underground bulb of onion plant comprises of fleshy leaf sheaths forming a thin-skinned capsule, and varies greatly in size (2–20 cm), shape (flattened, spherical, or pear-shaped) and colour depending on the variety.

2.3.2 Chemical constitution

The main colouring component found in the outermost dry skin of onion is quecertin ($C_{15}H_{10}O_7$), a flavonoid, proto-catechuic acid, kaempferol, anthocyanadine and some tannins [64].

2.3.3 Waste generated for onion

The outer layer of onion, i.e., the onion peel accounting for 10–25% (w/w) of the total weight are essentially removed before usage and thus forms a natural by-product of the food industry. With 22.43 million tons of onion produced by India as the second largest producer in the world [26], significant quantities of onion peel is generated as a waste.

Case study 5

Objective of the study: Dyeing of silk with a combination of a purified soxhlet extract of onion peels and pomegranate rinds [65].

Singh and co-workers [64] have used extract of peels of fruits and vegetables discarded as wastes (outer scaly peels of onion and rinds of the pomegranate fruit) to impart antibacterial functionality to degummed silk fabric dyed with the extract. Aqueous extraction of colour, from both the sources was done separately, and each solution was further purified through soxhlet distillation method. About 3 and 5 g/l of the both the extracts were applied on silk fabric as a mixture in a combination of 50:50, 25:75 and 75:25.

About 5 g/l concentration in the combination of 50:50 of the both the colourants gave the most optimised results and rendered a reduction of 98.1% in the bacterial count as evident from **Table 2**. Higher concentration of the dye when applied singly increases the antibacterial effect and higher proportion of pomegranate peel in the mixture has a slight improved edge over onion peel in rendering this effect.

2.4 Walnut shells

Kingdom: Plantae; Family: Juglandaceae; Botanical Name: *Juglans regia* L. (Persian Walnut) & *Juglans nigra* L. (American Black Walnut); Common Name: Walnut or *akhrot* (Hindi); **Part used for dyeing**: Outer hard shells enclosing the fruit.

Conditions of dyeing of silk	Concentration of the extracts	
	3 g/l	5 g/l
Only pomegranate peel extract	2.4×10^6 (96.1%)	2.1×10^6 (96.6%)
Only onion peel extract	2.8×10^6 (95.5%)	2.5×10^6 (96.0%)
Combination of pomegranate peel and onion peel extracts in the following ratios		
25:75	2.0×10^6 (97.1%)	1.9×10^6 (97.0%)
50:50	1.4×10^6 (97.7%)	1.2×10^6 (98.1%)
75:25	1.7×10^6 (97.3%)	1.6×10^6 (97.4%)

Data in parenthesis corresponds to the percentage in reduction.

Table 2.
Bacterial count (CFU/ml) and percentage reduction of E. coli *on silk fabric dyed with a single and combination of purified extracts of pomegranate peel and onion peel [65].*

Case study 6

Objective of the study: Dyeing of silk with onion peels discarded as agro-waste using unconventional energy effective methods of pad-dry-cure and pad-batch-dry techniques [44].

Natural dyeing is generally done at higher temperature that adds to the cost of the final dyed product. Singhee and Dhanania [44] have explored the challenge and provided solutions.

The authors have optimised conditions of aqueous extraction of colour from onion skin at (a) 5 (pH), (b) 1:20 (MLR) and (c) 80 min (time) and (d) 90°C (temperature). Use of 10% (owf) alumnium sulphate (mordant) by the pre-mordanting process was found optimum with respect to minimum strength loss maximum surface colour strength of the treated silk fabric. 80% (owf) dye concentration at pH 4 using MLR 1:40 at 60°C for 40 min gave optimum results with respect of surface colour strength and fastness properties of the dyed silk fabric. Strict control of pH, temperature of the dye-bath and dye concentration would result in uniform dyeing of silk with onion skins extract as indicated by high dispersion in the CDI values of silk dyed under variable conditions. These three have been identified as the predominating dyeing process parameters for dyeing silk fabric with onion peel.

To bring down energy costs, dyeing was carried out are room temperatures by the pad-dry-cure and pad-batch-dry methods. In the pad-dry-cure method, silk was treated with purified onion peel extract (extracted in ethyl alcohol and toluene solvents using soxhlet apparatus) at room temperature and then padded on a two-bowl padding mangle at 80% pick-up by 2 dip-2 nip process followed by drying at 90°C for 5 min. For the pad-batch-dry method, the padded silk fabric was rolled on a glass rod, covered with a plastic sheet and kept at room temperature for 24 h. Among both these two simpler dyeing processes that uses less water and fuel, the pad-batch-dry method gave highest K/S values which was higher than even that obtained when aqueous extract of the dye is used (**Table 3**). Colour fastness properties to wash and rubbing though were found be lower for pad-batch-dry method.

Samples dyed with aqueous extract of onion peel were subsequently treated with UV-absorbers (benztriazole, benzophenone and MEK) for improving light fastness, and dye fixing agents (Tinifix WS Conc., cetrimide and CTAB) for improving wash fastness properties by both exhaust process and pad-dry-cure method. UV-absorbers like benztriazole and benzophenone gives ½ to 1 grade improvement in the light fastness; while among the different dye fixing agents used, CTAB (cetyl trimethyl-ammonium-bromide) shows higher degree of improvement in the wash fastness when applied by the pad-dry-cure process on silk fabric dyed with aqueous extract of onion peel than by the exhaust method.

2.4.1 Occurrence

The walnut is edible fruit and belongs to the Juglandaceae family. The two most commercially important species are *J. regia* L. for timber and nuts and *J. nigra* L. for timber. The deciduous tall trees (10–40 m) are native to central Asia and the western Himalayas and have been cultivated in Europe as early as 1000 BC. Walnut is single-seeded stone fruit. The kernel or meat, which is usually made up of two halves separated by a partition and is encased in ligno-cellulosic shell that forms the thin endocarp or husk of the fruit.

Dye Concentration	K/S at λ_{max}	LF	Wash Fastness (ISO-II)		Rubbing Fastness	
			LoD	ST	Dry	Wet
Exhaust method (using 80 gpl of aqueous extract at standardise condition of dyeing)	8.2	3–4	1–2	4	3	4
Pad-Dry-Cure (using 5 gpl of purified dye)	2.7	2–3	1	4	4	4
Pad-Batch-Dry (using 5 gpl of purified dye)10.9	2–3	2	3	3	2–3	

LF—light fastness, LoD—loss in dept. of shade, ST—extent of staining on cotton

Table 3.
Surface color strength and color fastness properties of pre-mordanted silk fabric exhaust-dyed, dyed by the pad-dry-cure and pad-batch-dry method with purified extract of onion peel.

2.4.2 Chemical constitution

The colouring component present in walnut shell is juglone (CI 75500), a napthoquinone (5hydroxy-1, 4-napthoquinone) which imparts brown shade to textile substrate [66].

2.4.3 Waste generated for walnut

The walnut shells comprises 67% of the total weight of the fruit and as of 2009, the worldwide production of walnut was approximately 2.2 million tons. With this,

Case study 7

Objective of the study: Wool has been dyed with tannin-rich extract of walnut shells and pomegranate peel (agro-wastes) in combination with metallic mordants [66].

Shahmoradi et al. [65] have exhaust-dyed wool fabric pre-mordanted with metallic mordants with aqueous extracted walnut shells and pomegranate peels. The effect of the mordants, alumnium sulphate, copper (II) sulphate, iron (II) sulphate, iron (III) sulphate, potassium dichromate, tin (II) chloride and tannic acid, on colorimetric and antibacterial properties has been studied. Assigning the occurrence of –OH and C=O in the tannins present in the dye, the authors have reported improved colour strength in the dyed and mordanted wool fibre due to formation of a complex between the metal salt of the mordant and the dye.

K/S values appeared to be dependent on the concentration of the colourants in the solution from the two sources, and lower dye concentration though resulted in brighter shades (high L^* value), but the shades were less red (lower a^* coordinates). Among the various mordants studied, iron (II) and (III) sulphate and copper sulphate significantly improved K/S values of the dyed fabric.

Washing fastness of all samples treated with both the colourants was high, regardless of the type of mordant used, though light fastness ranged from excellent to poor. The authors have assigned the cause of poor light fastness to oxidation reaction of tannins in the dyes in the presence of light. If the dye-fibre bond promotes transfer of excitation energy from dye to fibre macromolecule, it makes it stable to light thereby improving light fastness. The authors have used this explanation to elucidate the role of chrome and copper salts as mordants in imparting dyeings with higher light fastness for both the dyes.

Pomegranate peel rendered better antibacterial activity against gram-negative bacterium (*E. coli* and *P. aeruginosa*) to the dyed wool irrespective of the mordant used compared to walnut shell extract. Increase in dye concentration improved the activity significantly and 5% concentration of dye in the presence of mordants dramatically enhanced the antibacterial properties. The antibacterial properties against *S. aureus*, *E. coli* and *P. aeruginosa* reduced significantly after washing and exposure to light due to breaking of the metallic complexes as a result of oxidation of tannins in the dye to hydroxyls and ketones in the presence of light. One cycle in a Launder-O-Metre was taken as equivalent to five home washing. Copper, aluminium and tin salts were found to be the most effective mordants for improved antibacterial activity that was durable to washing and light (could sustain five home washing). Interestingly the authors have also reported no significant antibacterial effect on the dyed fabric when chrome was used as a mordant.

Case study 8

Objective of the study: Dyeing of polyamide fibres using purified dye from walnut shells [16].

Mirjalili et al. [16] have used soxhlet extract of walnut shells to dye polyamide fabrics using 3% (owf) of different mordants (potassium aluminium sulphate, cupric sulphate, and ferric sulphate) by simultaneous mordanting and dyeing process. They have reported considerable increased in dye absorption leading to higher K/S values in case of mordanted samples compared with unmordanted ones, with ferric sulphate giving the highest colour strength.

Use of mordant also imparted higher antibacterial activity and here again ferric sulphate was more effective against *S. aureus* than other mordants; while cupric sulphate was better against *E. coli*. An increase in the dye concentration resulted in a tangible effect on antibacterial activity of dyed fabrics indicating that increased presence of phenolic and naphthoquinone compounds in the higher concentration of the dye extract is responsible for the improved antimicrobial activity. Use of metallic mordants also improved the wash durability of antimicrobial finish.

around 1.5 million tons of walnut-shells is generated as agro-waste [67]. Farmers mostly dispose the agro-waste residues including the shells, husk, stalks, etc., by burning them. But this is associated with serious environmental problems.

2.5 Almond shell

Kingdom: Plantae; Family: Rosaceae; Botanical Name: *Prunus dulcis* L.; Common Name: Almond or *badam* (Hindi); **Part used for dyeing**: Outer green hull and the woody hard shells enclosing the fruit.

2.5.1 Occurrence

Almond, a shrub or tree of the *Rosaceae* family belongs to the same genus, *Prunus* as the apricot, cherry, etc., but it differs from them in having a leathery fruit, which can only be eaten when immature, and has a comparatively large stone and kernel. This deciduous tree grows best in warm temperature where there is no spring frosts or tropical humidity and thus it is common to areas extending from West Asia to the West Mediterranean region. When the fruit ripens, its husk or flesh splits open, exposing the drupes (nut) which is rugged and pitted with irregular holes. The almond fruit consist of four parts, the kernel or meat covered by thin leathery brown seed-coat and enclosed in wrinkly, hard and woody endocarp shell. The shell is encased in the outermost green shell cover called the hull that becomes brittle as the fruit ripens.

2.5.2 Chemical constitution

Almond shells are rich in lignin and other phenolic compounds and the major flavonoids present are (+)-catechin, (−)-epicatechin, kaempferol, and isohamnetin. HPLC analysis has also revealed presence of quercetin, isorhamnetin and morin [30].

2.5.3 Waste generated for almond

Almonds are consumed as a valuable food and its production generates millions of tons of residues (shells, hulls, pruning, leaves, skin and inedible kernel disposition), the bulk of which are the almond shells (35–75% of the total fruit weight). Three million tons of almonds was globally produced annually as of 2014 and with this about 10.5–22.5 million tons of shells are generated and generally discarded [68].

Case study 9

Objective of the study: Wool has been dyed with extract of the outer green hull of the almond fruit [17].

Use of natural renewable and sustainable sources to dye textiles is one of the solutions as an environmentally safe technology that will expectedly conform to ecological legislations and limits. Keeping this in view, Ismal and co-workers [17] have dyed wool with extract of the outer green hulls of the almond fruit using metallic mordants as well as natural bio-mordants. Valex (acorn of *Quercus ithaburensis* ssp. *macrolepis*), pomegranate rind (*Punica granatum* L.), rosemary (*Rosmarinus officinalis*) and thuja leaves (*Thuja orientalis*) have been used as bio-mordants; while alum, iron (II) sulphate, copper (II) sulphate and potassium dichromate have been used as metallic mordants. Wool was dyed simultaneously with 2 g/l of aqueous extract of dried almond hull and varying amount of the bio-mordant (3–20 g/l) at 100°C using 1:50 MLR for 1 h. In case of the metallic mordants, pre and post mordanting techniques at 100°C using 1:50 for 1 h was also used. Unique and rare colour gamut of rose, brown, cinnamon and burgundy/reddish hues were obtained with the metallic mordants and the bio-mordants yielded completely different hues.

Among the metallic mordants, copper (II) sulphate gave highest K/S in case of all of mordanting process types (pre, post and simultaneous). Also, simultaneous-mordanting with any of the metallic mordant studied followed by dyeing with almond hull extract rendered lowest K/S value to the wool fibre. Baring valex, all bio-mordants increased the corresponding K/S values of the wool simultaneously mordanted with the bio-mordant and dyed with almond hull extract in contrast to when they were used individually in comparable concentrations. Valex when used alone, gave the highest colour yields among the bio-mordants and its K/S value for all concentrations was higher than that of the control sample. Relatively higher b^* value for valex resulted in a significant increase in yellow nuance of simultaneous bio-mordanted and dyed wool. The authors concluded that colour yields with bio-mordants can compete with metallic mordants barring copper sulphate when used through one-bath simultaneous dyeing and mordanting process.

Most bio-mordants can themselves be a source of colour and can dye fibres. The authors have also tried to study the synergistic effect on shades developed by natural bio-mordants when used in conjunction, at various concentration levels, with the dye from almond hull. Rosemary, valex and pomegranate bio-mordants show a synergistic effect with respect to the redness of the combined shade; their individually low redness value expectedly reduces the redness of the combined shade. Contrarily in case of thuja, the redness value of the combined shade is higher in spite of the lower redness value of thuja itself. Synergistic effect with respect to the blueness value can also be observed when valex, pomegranate and thuja are used in combination with almond hull extract; the blueness of the combined shade is higher than that of the control sample dyed only with almond hull extract or with the individual bio-mordant. L values of combination dyeing are always higher as compared to individual dyeing with almond hull extract for any of the bio-mordants under study, barring rosemary.

Wash fastness properties of wool simultaneously dyed with almond hull extract and bio-mordants improves slightly, but the corresponding values are lower when wool is mordanted with only with the bio-mordant without dyeing. Among the bio-mordants, valex and pomegranate rinds improve the light fastness of the wool when applied simultaneously with the dye from almond hulls.

Case study 10

Objective of the study: Silk has been dyed with an aqueous extract of almond shells [19].

Deepali and Shraddha [19] have used almond shells waste that is available abundantly and at almost no cost, to dye crepe silk fabric and have optimised process condition parameters (time, temperature, pH, material to liquor ratio and dye concentration) for pre-mordanting with 25% (owf) aluminium sulphate and exhaust-dyeing degummed crepe silk fabric with aqueous extract of the almond shell. The optimised extraction condition with respect to the highest optical density at maximum wavelength for onion peel has been established at (a) 25% owf (mordant concentration), (b) 80°C (extraction temperature), (c) 15 min (extraction time), (d) 1:30 (MLR), (e) 11 (pH).

The optimised dyeing condition with respect to both surface colour strength and fastness properties for dyeing silk crepe fabric using almond shell extract was established at (a) 100°C (dyeing temperature), (b) 60min (dyeing time), (c) 1:40 (MLR), (d) 2 (pH) and (e) 800% (dye concentration). CDI values of the dyed samples are widely dispersed for variations in temperature and pH indicating that special care should be taken with respect to the control these dyeing process parameters while dyeing protein fibres like silk with almond shell extract (**Table 4**). Dyeing under acidic pH gives darker shades and the fastness (wash and rubbing) properties of the dyed samples range from moderate to good.

Varying parameters	*K/S* at λ_{max}	CDI	RCR (CDI_{max} - CDI_{min})
Degummed and alum pre-mordanted silk (**CONTROL**)	1.00	—	—
Variation in **Time**			
15 min	1.8	55.8	49.44
30 min	1.6	11.3	
45 min	1.8	26.2	
60 min	1.8	17.5	
75 min	1.8	23.1	
90 min	1.8	6.3	
Variation in **Temperature**			
RT °C	1.2	67.6	66.37
40 °C	1.5	3.2	
60 °C	1.5	1.3	
80 °C	1.6	3.6	
100°C	2.0	4.4	
Variation in *p***H**			
2	5.9	9.6	109.01
4	5.2	110.1	
7	2.0	101.9	
9	1.9	3.2	
11	1.3	1.1	
Variation in **MLR**			
1:10	1.2	0.0	5.53
1:20	1.3	0.3	
1:30	1.5	3.3	
1:40	1.9	1.9	
1:50	1.7	5.6	
Variation in **DYE CONCENTRATION**			
25%	1.2	0.1	4.02
50%	1.2	0.8	
100%	1.6	4.1	
200%	1.8	0.0	
400%	2.16	0.1	
800%	2.39	0.5	

CDI—colour difference index postulated by Samanta et al. [69].

Table 4.
Color strength and color difference index of crepe silk fabric pre-mordanted with 25% (owf) aluminium sulphate and dyed with aqueous extract of almond shell using variable conditions of dyeing [19].

2.6 Groundnut/Peanut skin

Kingdom: Plantae; Family: Fabaceae; Botanical Name: *Arachis hypogaea* L.; Common Name: Peanut or *mungphali* or *chinia badam* (Hindi); **Part used for dyeing**: Outer hard shells enclosing the fruit and the thin papery skin (seed-coat) covering the fruit.

2.6.1 Occurrence

Peanut is a legume and oil crop grown worldwide and classified as *Arachis hypogaea* L. and belongs to the Fabaceae family. Originating in South America, it is grown also in China, Africa, Indian, Japan and United States of America. The fruit is enclosed in a pod that consists of outer covering (shell/hull). The veined brown shell or pod of the peanut contains two or three peanut kernels. Each oval-shaped kernel or seed is comprised of two off-white lobes/cotyledons that are covered by a brownish-red paper-like covering called the seed-coat.

2.6.2 Chemical constitution

Peanut skin have high content of phenolic compounds and contains eight different types of flavonoids along with and two alkanoids [70], phenolic acids, procyanidins dimmers, oligomers and some tannins. The main colouring components in peanut skin are vanillin, catechin and epicatechin (polyphenolic compounds) [71].

2.6.3 Waste generated for peanut

Substantial amounts of by-products (peanut meal, skin, hull and vines) are generated in the process of peanut harvest, peanut oil extraction and making of peanut butter, peanut oil, peanut confections, roasted snack peanuts, extenders in meat product formulations, soups and desserts [72]. Though they contain appreciable amount of polyphenols. Only a portion of these by-products are used as animal feed or as fertilisers. A large portion of the waste is discarded, incinerated or lands up into landfills.

The peanut shells are obtained as agricultural by-product when graded peanuts are passed through shelling machines to obtain the kernels. The production of peanut shells has been estimated to be 230–300 g of peanut shells per kg of peanut [73].

About 35–45 g of peanut skin being generated per kg of shelled peanut kernel and the annually worldwide production of peanut skin, as a by-product of the peanut processing industry is over 0.74 million metric tons [74]. In India, 3.0 lakh kg is available annually and can approximately dye 4.0 lakh kg of the fabric. Thus, a good source of dye from agro-waste associated with good sustainability is available at lost cost.

Case study 11

Objective of the study: Microwave energy has been used to dye cotton, silk and wool with aqueous extract of peanut skin (seed-coat) [20].

Peanut skin discarded as waste from food processing industry contains tannins and thus Pandey et al. [20] have dyed cotton, silk and wool fabrics with aqueous extract of peanut skin without using any mordants by both the conventional exhaust method and new method using microwave energy. Peach shades were obtained on all fibres dyed with peanut skin extract and the colour yield on silk was higher than wool; it was least on cotton. Increase in temperature of dyeing yielded better K/S values and dyeing at 90°C gave highest results on all fibres. Microwave dyeing gave good results, but the surface colour strength of the dyed fabric was less than exhaust dyeing at 90°C except in case of wool. The tenacity of the fabric decreased notably when dyeing was carried out using microwave energy.

Good light (6) and wash (4) fastness, excellent rubbing fastness (4–5) and good perspiration fastness (4) was observed on most dyed fibres. Peanut skin extract rendered excellent sun-protective function (UPF) to the dyed silk and wool fabric.

2.7 Tamarind seeds

Kingdom: Plantae; Family: Fabaceae; Botanical Name: *Tamarindus indica* L.; Common Name: Tamarind or *imli* (Hindi); **Part used for dyeing**: Shells and seed-coat of the fruit.

Case study 12

Objective of the study: Cotton has been dyed with extract from peanut pods (including shells and edible seeds) using metallic mordants [75].

Chhipa et al. [73] have used peanut pods to dye cotton using 10% (owf) metallic mordants (alum, copper sulphate and ferrous sulphate) using pre, post and simultaneously mordanting and dyeing processes. Authors have reported higher surface colour strength with increase in dye concentration irrespective of the process of mordanting used except in case of the pre-mordanting process, which indicates a decrease in the K/S values. In all cases, alum as a mordant gave better colour yield (K/S) and ferrous sulphate the least. Simultaneous mordanting and dyeing gave higher colour strength on cotton in all cases except when the cotton fabric was dyed by the post-mordanting process using copper sulphate.

Irrespective of the sequential process of dyeing and mordanting used, light, wash and rubbing fastness was reportedly good for cotton dyed with aqueous extract of peanut pods.

2.7.1 Occurrence

Tamarindus indica L. belongs to Fabaceae family, and is one of the most important multipurpose evergreen tree cultivated throughout the Indian sub-continent except in the Himalayas and western dry regions. The fruit of this tree is enclosed in a pod that comprises of pulp (55%), seed (34%) and hard brown shell (11%). The seed comprises of a brown coloured hard coat/testa (20–30%) and a kernel (70%). The kernels are separated from the seed-coat either by roasting or by soaking the seeds in water.

The tamarind fruits are bean-like, irregularly curve and bulged pods that usually vary from 2 to 7 cm in length. The pods are initially green and turn brown or greyish-brown as the fruit ripens. The pod-skin becomes brittle and the pulp inside dehydrates naturally to a sticky paste that encloses a few coarse strands of fibre extending along the length of the stalk.

2.7.2 Chemical constitution

The tamarind seed-coat contains 38–40% water soluble matter, 80% of which is a mixture of tannins, polyphenolic compounds [76] as condensed tannins and colouring matter (procyanidin trimer, procyanidin B2 and epicatechin) [77].

Case study 13

Objective: Cotton and silk has been dyed with tamarind fruit pods [78].

Umar et al. [76] dyed cotton and silk with aqueous extract, and purified methalonic and ethanolic extract of dried pod-skin of the tamarind fruit using alum, copper sulphate, ferrous (II) and (III) sulphate as mordants. All three methods of pre, post and simultaneous mordanting were used for comparison purpose.

Cotton and silk dyed with the aqueous extract of the tamarind pods, showed excellent to good light except when the sample was dyed with alum by the simultaneous mordanting process. All the dyed samples showed fair to good wash fastness with negligible staining. Rubbing fastness was also found to range from excellent to good, except when post mordanting process was used. Fastness to alkaline perspiration fastness was good (4–5) except in case of silk when iron (II) sulphate was applied by the simultaneous mordanting process or in case of cotton when copper (II) sulphate was applied by the post-mordanting process.

For cotton and silk dyed with methanolic extract of the purified dye, excellent to good light fastness was observed in all cases except when both cotton and silk were pre-mordanted with alum. The wash fastness of the dyed samples was found to be fairly good and negligible staining was observed. Rubbing fastness was found to range from excellent to good, while the perspiration fastness was fair.

For cotton and silk dyed with ethanolic extract of the purified dye, excellent to good light fastness was observed negligible colour staining. The wash fastness was fairly good (3–4) for cotton and silk. Perspiration fastness test was fairly good (3–4) in both acidic and alkaline medums, except in case of pre-mordanting process using iron (II) and iron (III) sulphates, where some loss in colour depth was visible specially when treated in an acidic medium. The extent of staining for both acidic and alkaline mediums was negligible.

Case study 14

Objective of the study: Sour and sweet tamarind seed husks or seed-coat has been used as a colourant to dye cotton and silk along [79].

Tepparin et al. [77] have dyed cotton, *Bombyx mori* (mulberry) silk and eri silk fabrics with aqueous extract of both sour and sweet tamarind seed husk/seed-coat (*Tamarindus indica* L.) varieties using zinc and iron sulphate and potassium dichromate as mordants. K/S values of silk dyed with tamarind seed husk in all cases was better than cotton with eri silk showing higher colour strength than mulberry silk under comparable conditions. Among the mordants, potassium dichromate gave highest colour strength on cotton and mulberry silk; while eri silk showed highest colour uptake by iron sulphate.

The colour fastness to washing of all the dyed fabrics ranged from good-excellent level and was stable even after five washing cycles. Colour extracted from sour tamarind seed-coat rendered a higher colour strength on cotton and silk than that obtained when sweet tamarind seed-coat extract was used under comparable conditions. Sour tamarind seed-coat yield redder, yellower and more intense shades than the sweet tamarind seed-coat.

2.7.3 Waste generated for tamarind

The reddish or purplish brown seeds rich in tannins [76] are generated as a by-product by the tamarind pulp industry. Also after removal of the pulp, the pods are mostly discarded. India alone produces about 0.3 million tons of tamarind annually.

2.8 Banana

Kingdom: Plantae; Family: Musaceae; Botanical Name: *Musa acuminate Colla*; Common Name: Banana; **Part used for dyeing**: Banana psuedostem, leaves and fruit peel.

2.8.1 Occurrence

Musa acuminata is an evergreen plant. The plant grows horizontally or obliquely from a false trunk also known as pseudo-stem and comprises of leaf sheaths packed in layers. The leaves are composed of a stalk called petiole and a blade or lamina. The individual flowers are white to yellowish-white in colour and grows upwards that is away from the ground. The fruits are slender berries whose size depends upon the number of seeds they contain. Each fruit bunch has about 160 fingers. As each plant produces only one bunch of bananas and the bare pseudo-stems cannot be used in the second harvest, the banana cultivation generates large amount of residues that are considered as organic wastes [49].

2.8.2 Chemical constituent of the dye

The holocellulose of banana pseudo-stem contain tetrahydroxyflavone (flavonoids) also known as luteolin and some tannins as the colouring components [49].

2.8.3 Waste generated for banana

Banana accounts for about 16% of the total world organic production [80]. After harvesting, almost 89% of banana plant (floral stem, outer part of pseudo-stem, peels, peduncle, underground parts, leaf stalk and leaves) remain as a waste.

2.9 Flower waste

Kingdom: Plantae; Family: Asteraceae; Botanical Name: *Tagetus erecta* L.; Common Name: Marigold; **Part used for dyeing**: Flower petals.

Case study 15

Objective of the study: Cotton has been dyed with banana floral stem sap without the use of any mordants [49].

Repon et al. [49] have retrieved sap from fresh banana floral stem using roller squeezer machine and used it to dye cotton by the exhaust method at varying temperatures. 100°C gave best result in term of the colour yield (K/S), colour levelness (ΔE), brightness index and colour fastness to wash, rubbing and perspiration. Assigning the role of temperature in boosting swelling of fibres to promote easier penetration of dye into interior of the fibre, the authors have explained the achievement of colour levelness at higher temperatures.

Except light fastness, all other colour fastness (washing, rubbing and perspiration) value ranged between 3 and 5, i.e., good to excellent. Wet rubbing properties were found to be lower than dry rubbing. Increase in temperature slightly improved the overall colour fastness properties. With increase in temperature, specimens showed a slight improvement in overall colour fastness properties. Authors have assigned poor light fastness of the dye to both lower photo stability of the natural pigments and also to the weaker dye-fibre interaction between the cotton fibre and the natural pigments in the banana floral stem sap.

Case study 16

Objective of the study: Alkaline and acetone medium have been used to extract colour from banana leaves to dye Egyptian cotton using metallic mordants [81].

Saleh [79] have extracted pigment from banana leaves using alkaline and acetone medium to dye Egyptian cotton fabrics in the presence of ferrous sulphate, copper sulphate and potassium dichromate as mordants. Dyeing of pre-mordanted cotton with the alkaline extracts of banana leaves was done at 80°C for 90 min at pH 9 using MLR 1:40; while dyeing with acetone extracted solution was carried out at 56°C for 5 min using MLR 1:40.

High performance thin layer chromatography (HPTLC) of the extracts identified pigment compounds, luteolin and apigenin in the alkaline extract and chlorophyll a, b and β-Carotene in the acetone extract. Though the K/S values for all dyeing were high, mordanted cotton displayed higher K/S value than unmordanted cotton irrespective of the mordant used. Mordanting with iron (ferrous sulphate) gave higher colour difference (ΔE) with yellowish brown colour probably due to a stable complex formed between iron and luteoline, a phenolic compound. Copper sulphate rendered a greener shade, while chromium produced pale yellow shades on cotton. Light fastness of the dyed samples was moderate, but good wash and rubbing fastness were obtained which improved when mordants were used.

Case study 17

Objective of the study: Alkaline fractions of banana fruit-peel have been used to dye cotton fabrics [47].

Salah et al. [47] extracted colour from banana peels using 0.1% NaOH. The extracted dye was applied to the ferric sulphate pre-mordated bleached and mercerized Egyptian cotton fabrics. The HPTLC analysis indicated the presence of pigment, Luteolin, in crude alkaline extract of banana peels.

Fe is known for its ability to readily chelate and form coordination complexes and several natural dyes. The authors have assigned the formation of a stabile complex between Fe of the mordant and luteoline of the colouring pigment in banana peel as the reason for maximum ΔE and corresponding lower L^* values of the pre-mordanted cotton fabric dyed with the alkaline extract of the peel. Interestingly they have denoted the cause of higher dye uptake by the pre-mordanted cotton fibre to the formation of a ternary complex between the mordant, dye and the fibre. The coordination numbers of iron causes some coordination sites to remain unoccupied when it interacts with cellulose of cotton and these unoccupied sites are taken over by the colour component in the dye thereby enhancing the interaction between the fibre and the dye.

Transformation of cotton from cellulose I to cellulose II during mercerization involves partial destruction of intermolecular bonds due to alkali-swelling. This allows the dye to penetrate the swollen amorphous region of the cellulose where they form hydrogen bonding with the fibre cellulose. This explains the better dyeability and corresponding higher dye uptake of the mercerized cotton compared to unmercerized cotton. Iron from the mordant also plays a role in increasing the dye uptake and shifting of the colour for mercerized cotton due to respective formation of a chelate with the sodium cellulose and sodium alcoholates and a complex formation with luteoline of the dye. Correspondingly the L^*, a^*, and b^* change for mercerized cotton.

The data obtained showed that the mercerized cotton have excellent antibacterial activity both in terms of zone of inhibition and quantitatively in terms of percentage reduction in bacteria. Swelling of cotton with its resultant shrinkage during mercerization causes reduction in the fabric interstices/porosity and this restricts the consequent UV transmittance giving high UV protection in terms of UPF to mercerized cotton. The UPF of the mercerized cotton fabrics was found to 64 compared to 18.0 for the unmercerized cotton.

The obtained results were also subjected to statistical analysis according to Sendcor and Cochran [82]. The experiments were in randomised complete design (RCD) with three replicates. Means were compared using the significant difference (LSD) at 5% level probability.

2.9.1 Occurrence

Marigold plant, a small perennial shrub that can grow up to 60 cm height belongs to Asteraceae family. It abundantly bears flowers varying in colour from yellow and gold to orange, red and mahogany depending on the species during the flowering season. Marigolds have been cultivated all over the world mostly South Asia [83].

2.9.2 Chemical constituent of the dye

Marigold flowers contain compounds called carotenoids containing lutein ($C_{40}H_{56}O_2$) and patulinin, a flavonoid. Lutein and patulinin are the two natural pigments responsible for colouring of textiles. Lutein is present along with its isomer, zeaxanthin also known as oxycarotenoids [84].

2.9.3 Waste generated for marigold

Marigolds, yellowish-orange coloured flowers are used in many religious ceremonies and offered in huge quantities to deities in temples throughout India.

Case study 18

Objective of the study: Cotton, silk and wool have been dyed with purified ethanolic extract of waste flowers collected from temples using metallic mordants [28].

Vankar et al. [28] have used temple waste comprising of tagetus flowers collected from the Kanpur city to dye cotton, silk and wool. NMR and mass spectra analysis of the crude extract from the flower identified the presence of patuletin as the flavanoid as the colouring matter present in *Tagetus erecta* L. Cotton was dyed, with colour extracted in an aqueous medium and also separately with colour extracted in ethanolic medium. Exhaust-dyeing of cotton fabric with ethanolic extract of the dye gave better colour strength (K/S of 141.73) than when dye extracted by the conventional method of aqueous extraction was used (K/S of 77.59).

Cotton (additionally pre-treated with tannic acid), silk and wool were pre-mordanted with different mordants (alum, copper sulphate, ferrous sulphate, stannic chloride and potassium dichromate) and then dyed with the purified ethanolic extract of the dye. Though the fastness properties improved with the use of metallic mordants, it rendered a slight change in the hue of the dyed fabric from yellow to yellowish-green to brown. Among the mordants used, ferrous sulphate gave the highest K/S on all fibres. Also the percentage of dye exhaustion due to metal mordanting varied with the different fabrics used; for cotton it varied between 45 and 52%; for silk it was 38–46% and for wool it ranged from 37 to 51%.

Case study 19

Objective of the study: New protein fibre derived from soyabean has been dyed with marigold flowers collected as temple waste [82].

Teli et al. [82] have dyed soyabean protein fibre (SPF) with marigold extract using natural mordants derived from tamarind seed-coat, *amla* (Indian gooseberry), *harda* (myrobolan) and compared the results with those obtained by using the more commonly used metallic mordant, alum, through pre-mordanting process. The K/S values of pre-mordanted SPF was higher than the unmordanted one. Alum rendered higher K/S value compared to the natural mordants and among the natural mordants, *amla* gave highest increase in the K/S values. Reportedly, increase in K/S values was observed with increase in concentration of mordants, and this was more so in case of the natural mordants. Results also indicated an increase in redness of the dyed fabric due to use of tannin-based natural mordants.

The concentration of the dye also impacted the K/S values, which improved with increase in the dye concentrations. Results also indicated that use of mordant improves fastness properties that were comparable for all natural and metallic mordants used in the study. Though the antibacterial properties of the samples dyed with marigold extract without mordanting was low, it improved after mordanting and all the three natural mordants rendered similar overall antibacterial activity. The antibacterial finish reduced on washing, but it was within the acceptable limit of 70% after 30 washes. Among the natural mordants, tamarind seed-coat and *amla* gave wash-durable antibacterial finish compared to *harda*.

Most of them are thrown in to the neighbouring ponds, lakes and rivers or simply dumped around the temple to decay naturally. This adds to pollution in and around most temples and has an undesirable effect on the river ecology [28, 81].

3. Conclusion

As ecological considerations become important factors in the selection of consumer goods all over the world consumers want quality product with no harm to the ecology, use of wastes from various sources for dyeing textiles can prove to be attractive for two reasons. One, they provide novel sources for dyeing textiles at lower costs. Two, they provide sustainable solution to the management of waste disposal.

Though a number of studies have been carried out to explore alternative source for natural dye, more specifically from wastes, more needs to be done in a systemic and scientific manner. Systematic collection of waste from the source and suitable alternatives to convert the waste into concentrates and appropriate recipes for dyeing to produce reproducible shades needs to be explored.

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Chapter

Application of Natural Dyes for Herbal Textiles

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Abstract

Clothing, the second skin of our body, is of immense importance in human life and has been evolved continuously. Textile products having special properties are used for umpteen purposes, and one such product is herbal textiles. Changing trends in clothing, cosmetics, and aesthetics necessitates the development of newer technologies that can benefit end users by enhancing their health and aesthetics and provide a sense of relief and satisfaction. Herbal textiles are prepared by imparting essential oils and herbal extract finishes to textiles. These treatments provide medicinal value and aroma to the garment, which enhance its value. Herbal extracts and oils can be incorporated in textiles by microencapsulation, sensory perception technology, liposomes, dyes, and coated textile technology. The temperatures of the finish, the duration and concentration of plant extract, the blend of herbs, and the equipment used need to be carefully controlled. Herbal textiles are revitalizing and help in keeping the body fresh and healthy. These fabrics also shown to have therapeutic value in ailments like skin allergies, breathing problems, sleeping disorders, and blood pressure. In this chapter, emphasis is given on the significance of herbal clothing and the use of natural herbal extracts to produce them.

Keywords: natural dyeing, herbal finishes, herbal extracts, functional finishes, herbal wear

1. Introduction

Textiles are susceptible to micro-organisms and their products (e.g. toxins) known to cause infection and intoxication. Myriad textile products such as herbal textiles and clothing are available in the market to support healthy lifestyle and hygiene. Most textile products are meant to come in contact with the skin; hence, herbally treated fabrics are better choice as they do not harbor harmful chemicals and thus are ecofriendly. People of yore used to live in harmony with nature and passionately protected their environment; however, nowadays, excessive exploitation of natural resources has led to a phenomenal increase in environmental pollution. This is taking a serious toll on the lives of all living beings on the planet earth. Right kind of fabric is of paramount importance to a healthy lifestyle, and synthetic fabrics' teeming with chemicals and dyes poses severe health threat. One would not like to eat a bowl of pesticide-drenched food. Then, why wear clothing doused in chemicals? Textile dyes obtained from natural sources are usually friendly to human skin and are biodegradable and ecofriendly. Some natural dyes have medicinal properties and impart healing qualities to the fabric. They are ecofriendly, save energy, provide rural employment, and preserve traditional craftsmanship [1].

Clothing is our second skin and plays an important role in human life. Textiles finished or dyed from extracts of various medicinal herbs are called herbal textiles. Textiles and clothing infused with medicinal herbs are becoming popular, especially in urban India. The use of such products helps in providing protection against myriad biological toxins and pathogens. Customers demand for new ranges of technology, owing to upward trend in enhancing beauty, that provide better esthetics along with improved health and a sense of relief and satisfaction during use. This trend has produced Cosmetotextiles.

Among technical textiles, medical textiles are a promising product, which plays an important role in maintaining the human health. Medical textiles consist of textiles used in operative and postoperative tasks on and around the patient and medical practitioners. They are broadly classified as nonimplantable materials, implantable materials, extracorporeal devices, hygiene products, protective, and healthcare textiles. Healthcare, disposable, and nondisposable hygiene products mainly used in hospitals to reduce the chances of contamination by biological toxins and infectious organisms.

India has good potential to produce natural dyes commercially as it is considered as 1 of the 17 mega diversity countries globally [2]. Traditional knowledge facilitates the use of plants and their products for healing the wounds and burn injuries and treating the skin infections. Herbal extracts are being used successfully for imparting antimicrobial finish to textiles [3].

Herbal products derived from plants are gaining popularity worldwide for use in textiles on account of their easy availability, green approach, low toxicity, biocompatibility, and ecofriendly nature [2, 3]. Owing to market demand for textile products having added comfort and functional properties, herbal products may be explored for their potential benefits to produce such products. This mandates scientists to devise methods and techniques to use plants containing bioactive agent in producing value added and more attractive functional textile substrates. Insect repellent, deodorizing/aroma, UV protection, antimicrobial, and flame retardant are some of the new properties, which have been imparted recently to textiles. In recent years, the growing demand for herbal products has led to the idea of developing healthcare textile products using herbal extracts.

Currently, herbal colorants are the most attractive option because of high biodegradability, low toxicity, green chemistry, and potential application in the textile dyeing and finishing industry. Natural colorants from plant sources are considered as novel agents in imparting multifunctional properties such as antimicrobial, insect repellent, deodorizing, and UV protection to textiles. Many natural colorants possess some inherent functions in addition to their coloring attribute. These inherent properties are transferred to the textile substrate dyed with an herbal colorant. Alternatively, dyeing textiles with these herbal colorants can combine dyeing with functionality finishes, an ecofriendly process using less water and energy than currently used separate wet treatments. Recently, increased interest in the use of natural dyes in textile dyeing has been observed due to enforcement of the stringent environmental regulations by many countries because of toxic effects of synthetic dyes. This review gives an overview of the herbal methods available at present for dyeing textile materials with aim to provide other useful finishes [4].

2. The herbal textile concept

Textiles dyed exclusively with herbal extracts without using any chemicals are called herbal textiles. The herbs utilized in dyeing are different from vegetable dyes as they also have medicinal values. These herbs are applied to the fabric using natural ingredients in order to preserve medicinal properties. Further, bleaching of cloth is done by exposing it to sunlight without the use of any chemical bleach. The concept of herbal textiles has been derived from Ayurveda, the ancient Indian method of vedic healthcare. Ayurvastra is a branch of Ayurveda. In Sanskrit, *ayur* means health, and *veda* means wisdom, while *vastra* means clothing when it combines the meaning life cloth [5].

It is made by embedding essential oils and herbs into textiles. A wide range of herbs such as turmeric, tulsi, neem, rose, Indigo, lemon grass, castor, and ber can be used for this process providing medicinal value and aroma to the fabric. Ayurvastra (marketed as cosmetotextiles) has been used for curing several diseases such as diabetes, arthritis, skin infections, hypertension, and hay fever [5].

Herbal clothing is an ancient technique of dyeing textiles in medicinal herbs. This alternative method of treatment originated more than 5000 years ago in India. Its core principles are rooted in the Vedas, which are an ancient book of Hindus. The Vedas date back to around 3000 BC. The science of life or Ayurveda dates back to around 1000 BC. The origins of Ayurveda are also found in the Atharvaveda. It contains incantations and hymns that cure various diseases through mantra. Mythology says that Ayurveda was given to Dhanvantri by Lord Brahma. Charaka Samhita and Sushruta Samhita are the oldest known Ayurvedic texts [6]. About 100 years ago, people from different parts of India were using various forms of natural dyeing in which they repeatedly dipped clothes in herb-based preparations following each wash [7]. These herbs are used traditionally in Ayurvedic treatment and many medical applications. When this fabric is worn, the medicinal property of the fabric is then transferred to the skin. The process of herbal dyeing was developed through extensive research during the age-old dyeing methods practiced since the days of the Indus civilization. Herbs were used to create different colors and to provide various health benefits. The natural/herbal fabrics are 100% organic, completely free of synthetic chemicals, and toxic irritants and are biodegradable [8].

Herbal clothing is prepared from organic cotton fabric impregnated with special herbs and oils for health benefits. Since ancient times, herbal clothing (Ayurvastra) is believed to cure diabetes, skin infections, psoriasis, hypertension, asthma, arthritis, rheumatism, high blood pressure, eczema, and cancer. Antimicrobial property or natural dyes such as indigo, pomegranate, myrobalan, and Indian madder of against bacteria such as *Escherichia coli* and *Klebsiella pneumoniae* have been reported [9]. Herbs such as harad, rakta chandana, shweta chandana, and kumkuma are used in Ayurveda for protecting the skin from harmful rays of sun. The herbal preparations act on melanocytes present in the skin, modulate their function, and block the effect of UV rays on the skin. References in different texts of Ayurveda are replete with examples of wearing pure cotton clothes impregnated with herbs such as chandana, haridra usheera, manjista, and nimbi aragwadha to safeguard human body from inclement weather and to keep it healthy. They used herbal products such as soap nut and lime for washing cloths before soaps and detergents were discovered. Herbal textiles and dyes have great vista of applications in diverse walks of human life. These herbal dyes have been used for centuries to produce colors for fabrics, yarns, leather, food, and so on. Natural dyes exhibit better biodegradation and have a better compatibility with the environment. The application of natural dyes has the potential to earn carbon credit by consumption of fossil fuel-based synthetic dye [10].

3. Mode of action of herbal textiles on the body/herbal couture technology

Skin is the largest sense organ in the human body (**Figure 1**). It can act as a barrier as well as a medium for entry of certain substances in the body. Several

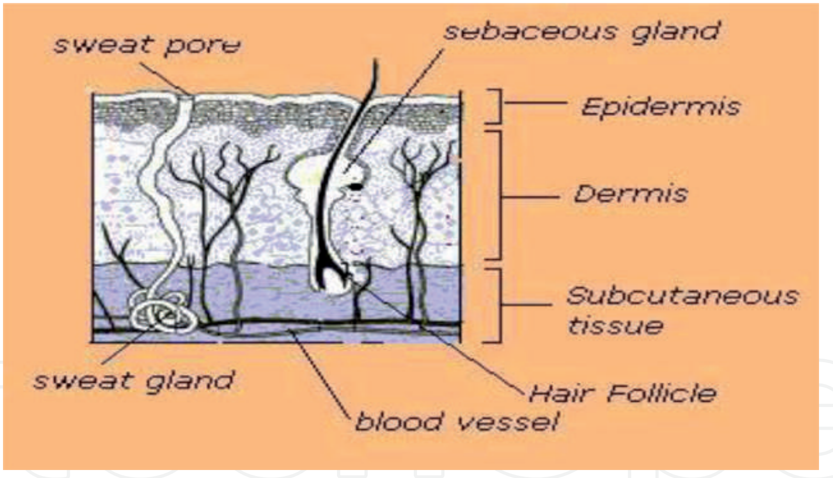

Figure 1.
Structure of the human skin [6].

toxins and chemicals present in the environment get accumulated in the conventional clothing and may enter into the body through the skin. Hence, enhancing skin's ability to resist entry of harmful chemicals and toxins into the body will improve the health. The radiated heat activates herbal molecules of the cloth and is reflected back to the body along with the herbal molecules. Through the blood, the herbal molecules enter deep into the body, show its effects at different levels of the body, and help in curing various diseases [5]. The skin can act not only as a protective barrier but also as a medium for outside substances to enter into the body. In the same way, the skin has the ability to absorb herbs found in our natural dyes. These herbs release their medicinal qualities in the body and strengthen the skin's ability to block and resist the harmful substances. The close contact of herbal clothing next to the skin enhances the body metabolism, which leads to effective elimination of body toxins. The herb-infused and herb-dyed organic fabrics act as healing agents after the herbs being absorbed through the skin [6].

3.1 Penetration of herbal molecules across skin

- Skin temperature helps the herbal molecules to be absorbed into the body through sweat pores to manifest the desired medicinal effect.

- The cloth should be in direct contact with the skin to manifest its curative effects.

3.2 Herbs and healing effects of herbal textile

Herbs are garden plants that are grown and harvested for culinary, aromatic, medicinal, and fibrous uses. Plant herbs are placed in the garden for their unmistakable fragrances, attractive textures, appealing colors, and variety of home uses. Due to current ecoconsciousness, there has been a revival of inherent in ecofriendly natural dyes throughout the world. The application of herbal products has given a new direction toward the treatments of various diseases through textile products. The herbs also do not pollute the environment through contamination of water resources in areas close to processing units. All kinds of shades of red, yellow, brown, orange, and green can be obtained with the help of the herbs.

Functional finishes using natural dyes have been in the vogue that promotes an ecofriendly lifestyle. Natural dyes are environmental friendly, nontoxic, noncarcinogenic, nonallergic, and renewable resources. Biosphere is being gifted with more than 500 plant species that yield the natural dyes [11]. The roots, flowers, leaves, seeds, and barks of more than 200 medicinal herbs, plants, flowers, roots, and barks are used to make the dyes. In herbal textiles, the color is gained from the medicinal preparation only, and no other colorants are used. As a result, its properties will last as long as the color is there. Since the herbs make beautiful shades, herbal wears are also becoming very popular for its primordial elegance.

Only herbal preparations devoid of any chemical are used in dyeing herbal textiles, whereas chemicals such as copper sulfate and ferrous sulfate are used as catalysts when dyeing is done with vegetable dyes. Thus, herbal dyes are different from vegetable dyes as they also impart some medicinal value. Herbal clothing is believed to help restore the balance within the body's systems and strengthen the immune system. Some of the medi-herbs used in herbal textiles give the magical healing quality to the dyed fabric or yarn (**Table 1**) [12–15]. **Table 1** shows the diseases cured by different herbs.

S. no.	Name of the herb	Medicinal properties	Diseases claimed to be cured
1.	**Neem** (*Azadirachta indica*)	Antibacterial and antifungal	Common skin diseases, skin allergy, controls blood sugar levels
2.	**Curry leaves** (*Murraya koenigii*)	Medicinal value	Beneficial in curing arthritis suppresses pain and reduces inflammation
3.	**Shoe flower** (*Hibiscus rosa-sinensis*)	Controls blood sugars	Diabetes
4.	**Fenugreek/ Methi** (*Trigonella foenum-graecum*)	Medicinal value	Controls cholesterol, hypertension
5.	**Amla** (*Emblica officinalis*)	Antifungal, antibacterial, antiviral	Boosts immunity
6.	**Acacia** (*Catechu*)	Antifungal, antibacterial	Used for treatment of parasitic infestation and itching
7.	**Manjistha** (*Rubia cordifolia*)	Medicinal value	Blood purifier and diuretic, leprosy
8.	**Cuscus/Khus khus grass** (*Chrysopogon zizanioides*)	Medicinal value	It helps in fighting asthma
9.	**Arjuna** (*Terminalia arjuna*)	Medicinal value	Cardiovascular support, hypertension, asthma
10.	Pomegranate (*Punica granatum*)	Antibacterial, antiviral, astringent	Antimicrobial treatment, hypertension
11.	**Harad/myrobalan** (*Terminalia chebula*)	Antiseptic, anti-inflammatory, controls cholesterol	Hypertension, anemia, asthma cures, wounds, ulcers
12.	**Aloe vera** (*Aloe barbadensis*)	Antibacterial, antifungal	Burns, wounds, common skin diseases, psoriasis, anemia
13.	**Gurhal** (*Hibiscus rosa-sinensis*)	Controls blood sugar levels	Diabetes
14.	**Jamun** (*Syzygium cumini*)	Controls blood sugar levels	Diabetes controls blood sugar levels

S. no.	Name of the herb	Medicinal properties	Diseases claimed to be cured
15.	**Mahuva** (*Madhuca longifolia*)	Controls blood sugar levels	Control diabetes
16.	**Champa flower** (*Magnolia champaca*)	Controls blood sugar levels	Diabetes, anemia
17.	**Touch-me-not** (*Mimosa pudica*),	Medicinal value	Controls blood sugar levels, diabetes
18.	**Eucalyptus** (*Eucalyptus globules*)	Antiseptic	Relieve congestion, prevent infection, ease muscle soreness
19.	**Tulsi/basil** (*Ocimum tenuiflorum*)	Antibacterial, antiseptic, antispasmodic, diaphoretic, febrifuge, nervine, controls cholesterol	Respiratory disorders, viral, bacterial infections. Controls blood sugar levels, boosts immunity, hypertension, HIV/AIDS
20.	Chitosan	Antimicrobial	Naturally occurring biopolymer having antimicrobial properties
21.	Balloon vine (*Cardiospermum halicacabum*)	Antiphlogistic, analgesic, blood refrigerant, anti-infectious	Cold, fever, renal edema, urinary tract infections,·furuncle, carbuncle, eczema, sprains, external wounds
22.	**Sandalwood** (*Santalum album*)	Alterative, antibacterial, anti-inflammatory, antiseptic, astringent, carminative, disinfectant, diuretic, expectorant, hemostatic, refrigerant, sedative, stimulant	Headache, acute dermatitis, bronchitis, cystitis, eye diseases, gonorrhea, herpes zoster, infection, palpitations, sunstroke, urethritis cools the skin, curing skin itching, burns, its mild fragrance has a soothing effect that helps in fighting stress
23.	**Onion** (*Allium cepa*)	Antioxidant, antimicrobial	Skin diseases, control diabetes
24.	**Indian Madder** (*Rubia cordifolia*)	Antibacterial, anti-inflammatory, antiseptic, astringent, sedative, stimulant	It removes blood impurities and cures various skin diseases, HIV/AIDS
25.	**Indian Mulberry** (*Morinda pubescens*)	Antibacterial, anti-inflammatory	It is blood purifier and used for wound healing, control diabetes
26.	**Saffron** (*Crocus sativus*)	Medicinal value	Controls heart disease
27.	**Indigo** (*Indigofera tinctoria*)	Antibacterial, antiallergy, antiseptic and aromatic, anti-inflammatory properties	Controls cholesterol, helps reducing the insulin resistance, thus controls diabetes, boosts immunity, anemia, skin diseases
28.	**Turmeric** (*Curcuma longa*)	Antihepatotoxic, antioxidant, antimicrobial antiseptic, antiallergic	Fighting skin diseases and helps fight skin disease, anemia, psoriasis, liver disorders
29.	**Henna** (*Lawsonia inermis*)	Blood purifier, anti-irritant, deodorant, antiseptic	Skin irritations such as heat rashes and skin allergies and to cool the body. Because of this cooling property, henna used as a prophylactic against skin diseases like burns, bruises, and skin inflammations, including sores from leprosy

S. no.	Name of the herb	Medicinal properties	Diseases claimed to be cured
30.	**Brazil wood** (*Caesalpinia echinata*)	Blood purifier, anti-irritant	Skin diseases such as burns, bruises, and skin inflammations
31.	**Castor** (*Ricinus communis*)	Blood purifier	Skin diseases
32.	**Baheda** (*Terminalia bellirica*)	Controls cholesterol	Hypertension, anemia
33.	**Guar** (*Cyamopsis tetragonoloba*)	Controls cholesterol	Hypertension
34.	**Safflower** (*Carthamus tinctorius*)	Controls cholesterol	Hypertension, HIV/AIDS
35.	**Reetha** (*Sapindus trifoliatus*)	Medicinal value	Psoriasis, skin disease
36.	**Amaltas** (*Cassia fistula*)	Therapeutic effect	Psoriasis
37.	**Cluster fig** (*Ficus racemosa*)	Therapeutic effect	Psoriasis
38.	**Winter cherry** (*Withania somnifera*)	Therapeutic effect	Psoriasis, rheumatism

Table 1.
Some of the medi-herbs used for herbal textiles.

Different parts of the plants namely root, bark, leaf, fruit, wood, seed, and flower are used to extract dyes. It is estimated that over 2000 pigments are found in various parts of plants; however, only about 150 have been commercially utilized [16]. Herbal cloth production by a particular method is accomplished by utilizing extracts of selected herbs for dyeing cotton/silk/linen, yarn, and fabric. Synthetic/chemical dyes are not used at any stage, and for washing, specific products are used [17].

The process of herbal dyeing was developed through extensive research during the age-old dyeing methods practiced since the days of Indus civilization. Manufacturing herbal textile begins with 100% hand-loomed organic cotton or silk wool, linen, jute, hemp, and so on and their natural blends that are dyed exclusively in herbal infusions for both color and health purposes. No chemical process is involved while dyeing and processing the fabric. The fabrics have to pass through various stages of treatment before they are turned into finished products. Herbal textiles are extra smooth and good for transpiration that helps in recovering from various diseases. Each fabric is infused with specific herbs, which are known to cure diseases. It may help to treat a broad range of diseases and skin infections such as eczema and psoriasis; the fabric also helps with conditions such as heart ailments, rheumatism, arthritis, blood pressure, diabetes, and respiratory conditions, such as asthma and sleeping disorders. It also helps in boosting immunity. The antibacterial and anti-inflammatory properties of herbal medicated clothing extend its use as dressings and bandages. It can also relieve the general body aches, stimulate the weight loss, and strengthen the immune system. It can also be used as energy booster; mood enhancer, for overall wellbeing, calming, and blood purification; and cooling [18]. The most effective time to wear herbal cured clothing is when the body is at rest, such as during sleep, relaxation, or meditation because this is when the body is naturally healing and re-establishing balance. Hence, most of herbal clothing products are sleepwear, bed sheets, towels, meditation clothes, and cotton mats [19].

4. Herbal dyeing process

The herbal textile production begins with pure organic yarn/fabric, and each step is carefully and meticulously controlled without the use of any chemical process in dyeing and processing. The process starts with the gray cloth going through various stages of treatment before it is dyed and becomes ready to wear. Only certified organic cotton, natural cotton, silk wool, linen, jute, hemp, and their natural blends should be used for herbal dyeing. The herbal dyeing process has been developed following the extensive research and is in practice since the Indus civilization. Machines are not used to prepare herbal clothing, and all the processes namely bleaching, dyeing, and other processes are accomplished in big vessels fabricated according to need. Further, no chemicals are added during spinning, weaving, or enhancing the appearance [20].

Most common fabric used is organic cotton, the cotton that is cultivated without chemical pesticides and fertilizers. Silk, wool, coir, linen, hemp banana, nettle, and bamboo are also used to prepare herbal clothing. For handloom, the process starts with the cotton yarn and goes through a process named desizing (to remove loose particles, debris, etc.). The common processes used in manufacturing of herbal clothing are detailed in the following paragraphs.

4.1 Desizing

It involves washing of the processed gray cloth with mineral-rich water and sea salt to remove sizing, gums, and oils used in the weaving.

4.2 Bleaching

This process is actualized with biodegradable, organic cleaning agents, and surfactants and sun dried. The bleaching of cloth is done naturally by exposing it to sunlight on a natural grass base and by using animal manure. The fabric or yarn is first bleached using cow urine, milk, honey, along with biodegradable, naturally derived, organic cleaning agents, and surfactants such as Saptala (*Acacia sinuata*) and Phenila (*Sapindus mukorossi*) [21]. Many Ayurvedic doctors consider cow's urine highly beneficial in balancing an individual's "doshas" or basic constituents of an individual's physiology and psychology, strengthening the immune systems and as an elixir in giving life [22]. The process ends with exposing the fabric or yarn directly to sunlight.

4.3 Mordanting

To make the colors look bright and retain color fastness, natural mordants such as myroballans, rubhabs leaves, oils, alum, fruit extracts of haritaki, bark of lodhra, minerals, and iron are used. Use of heavy metallic mordants such as copper, chrome, zinc, and tin is avoided as they are not ecofriendly. Mordanting is done before dyeing the fabric or yarn to make the colors look bright and retain fastness.

4.4 Dyeing/medication

The word medication is used instead of dyeing because the medicinal herbs are used to impart required color to fabric or yarn [20, 23]. The yarn or fabric is then medicated in a carefully controlled mixture of herbal medicinal preparations depending on the disease or ailment being treated. Required herbs are applied directly to the fabric with the help of natural ingredients so as to keep the medicinal value of herbs intact [7, 11]. Steps involved are as follows:

- The organic cotton yarn/fabric is dyed carefully with concoction of herbal dyes indicated for the treatment of desired disease(s).
- The herbal dyes are prepared by careful blending of medicinal herbs derived from plants, flowers, roots, and barks.
- The temperature, duration, number of the soaks, blend of herbs, and equipment are carefully controlled.
- Desired herbs with the help of natural ingredients are applied directly on fabric in order to preserve the medicinal value of herbs.
- Shades of red, yellow, brown, orange, and green can be obtained with careful use of herbs.
- Finally, the medicated cloth is cooled and washed several times to remove the nonabsorbed particles and always dried in shade.

4.5 Finishing

The finishing process used after herbal dyeing is also organic and is given by sprinkling pure water on the cloth and then stretching under pressure, using hand rolls, *Aloe vera*, castor oil, and so on [7, 11].

4.6 Recycling residue

After dyeing, residue can be recycled. Solid and liquid wastes are separated by the filtration and used for farming purposes—as biomanure and for watering the fields and also to generate the biogas [11].

5. Technologies used for herbal finishing

Herbal textile is made by embedding essential oils and herbal extract finishes into textiles. Such textiles have their applications in providing medicinal value and aroma to the garment. Microencapsulation, sensory perception technology, liposomes, dyes, and coated textile technology are used for incorporating the herbal drugs and oils in textiles. The temperatures of the finish, the duration and concentration of plant extract, the blend of herbs, and even the equipment used are carefully controlled [24].

Spray drying, air suspension coating, solvent evaporation, sol-gel microencapsulation, and in-situ polymerization are some commonly used techniques used for textile finishing by microencapsualtion. For encapsulation of flavor, fragrance, and oils, spray drying is used in which core particles dispersed in a polymer solution and sprayed into a hot chamber leading to solidification of shell particles on core material and polynuclear or matrix-type microcapsules are obtained. Air suspension coating is done by suspending solid particles of core material in a hot or cold upward moving steam of air. Coating material may be derived from cellulose derivatives, dextrins, emulsifiers, lipids, protein derivatives, and starch derivatives, which is atomized through nozzles into the chamber and deposits as a thin layer on the surface of suspended particles. Solvent evaporation is a process in which the drug is dissolved, dispersed, or emulsified in an external aqueous or oil phase; it has been used to impart antifungal finish to textiles. Sol-gel microencapsulation is used to produce flame retardant, water/oil repellent, UV protecting, antiwrinkle, self-cleaning, flavor and odor containing, and antibacterial textiles. Sol-gel synthesis involves the hydrolysis of silicone monomer and the condensation of silica into a porous structure with a three-dimensional networked structure, which can be tailored for a wide range of useful properties.

Microencapsulation, widely used in textile finishing, is a process in which small capsules having desired properties are prepared using small particles/droplets surrounded by a coating material. The material inside the microcapsule is known as core material, whereas outer coating is called a shell. Microcapsules may have diameter ranging a few micrometers to a few millimeters. Many special and functional properties can be imparted to the fabrics by microencapsulating the core material. This core material can be any substance having a special function to perform for the fabric. Encapsulation has allowed moisturizers, therapeutic oils, and insecticides to be incorporated into fabrics. Microencapsulation of antimicrobial agents is also gaining popularity in sportswear and medical textiles [25].

For preparation of herbal textile by microencapsulation, the "core material" along with the wall material should give a durable finish. This wall material can be certain substances like gum acacia, and the core material is the desired herbal extract. The microencapsulated herbal extracts have shown antimicrobial activity and wash durability up to 20 washes [26]. Microencapsulation is a very popular technique for incorporating fragrance into fabrics. These capsules applied are unbreakable under the normal conditions, and the application of pressure on fabric releases fragrance, which causes healing in aromatherapy [27]. Microencapsulated herbal extracts such as *Aloe barbadensis* Mill, Bitter Gourd, Ginger, and *Cuminum cyminum* Linn have successful application in which the herbal extracts are used as core material and gum acacia as wall material [28]. According to the study, microencapsulated extracts of *Andrographis paniculata*, when applied on the fabric to impart mosquito repellency finish, gave good mosquito repellent activity up to 30 washes [29].

5.1 Microencapsulation in cosmetic textiles

New textile technologies have enabled the application of cosmetic ingredients on fabric to provide its functional benefit to the end-use product, and therefore, cosmetic textiles are moving from laboratory to showrooms. Fragrance finish is one such finish that falls under this category. A fragrance is made from a pleasant smelling aroma compound. Aromachology is a science that studies the effects of fragrances on the human body and mind. It researches how scents can be used to induce relaxation and make life more pleasant. Fragrances can be used in various forms such as essential oils, perfumes, colognes, household products, and potpourri. Some areas in textiles where fragrance finish can find application include Cosmetotextiles.

The major use of microencapsulation in cosmetic textiles is in the application of vitamins, essential oils, skin moisturizing agents, skin cooling, and antiaging agents [30]. Yamato et al. studied microcapsules having active substances that can improve the physiological conditions of human skin. The microcapsule gradually released its content when the textile structure was subjected to light pressure created due to the movement of human body [31].

Aroma finish is a process by which textile material is treated with the pleasant odor producing essential oils and aromatic compounds so that the wearer gets beneficial effects. Various essential oils such as lavender, rosemary, and jasmine are used.

The fragrance compounds and the essential oils are volatile substances, and prolonging life span of their odor is most difficult task in preparation of the textiles for aromatherapy. Microencapsulation is an effective technique to achieve this task. Microcapsules are tiny spherical enclosures containing a liquid or gas and assume the shape of the enclosed particle if containing a solid [32]. With microencapsulation method, fragrance lasts for more number of washes since microencapsulation makes the finished particles more securely attached to the internal structure of the fiber and thus releasing them slowly [33].

5.2 Microencapsules for antimicrobial finishes

Bacteria often cause decay of fabrics leading to loss of various useful properties of fabrics. This problem can be prevented by the use of antimicrobial finishes that can be applied with the help of microencapsulation. This finish is especially beneficial for textiles for medical and technical uses [34].

Herbal extracts prepared from Chamomile, sage, and green tea were applied to cotton fabric using pad-dry-cure method. Treated fabrics exhibited antimicrobial activity against Gram-negative, Gram-positive bacteria and yeasts. This antimicrobial activity was retained even after 10 cyclic washes [35].

Textile materials having antimicrobial activity are used in hygienic, health, personal care, and surgical products. Some products include baby and adult diapers and sanitary pads. Most of the diapers are made by synthetic materials, which are nonbiodegradable and poor antimicrobial properties, and may cause rashes and dermatitis. Natural antimicrobial coatings on diapers obtained by using curcumin, *Aloe vera*, tulsi, and neem have same antimicrobial effect when compared with synthetic antimicrobial coatings [36]. Curcumin or diferuloylmethane (1,7-bis(4-hydroxy-3-methoxyphenyl)-1,6-heptadiene-3,5-dione) is the main phytochemical present in turmeric. This polyphenolic compound has several biological activities including antimicrobial activity. Curcumin is suspected to affect the bacterial cytoskeleton, thus producing the antimicrobial effect [37]. The *Aloe vera* leaves secrete a colorless gel-like substance, which contains about 98–99% water and 1–2% active compounds such as aloesin, aloin, aloe emodin, aloe mannan, flavonoids, saponin, sterols, amino acids, and vitamins. Anthraquinones and their derivatives such as barbaloin-IO-aloe emodin-9 anthrone, isobarbaloin, and chromones, which inhibit the bacterial protein synthesis, are potent antimicrobial agents [38]. Antimicrobial property of tulsi (holy basil) may be attributed to essential oils found in leaves. These essential oils contain germacrene-A, clemene, caryophyllene, eugenol, and caryophylline oxide. Additionally, tulsi leaves also have other biologically active phytochemicals such as rosmarinic acid, oleanolic acid, and ursolic acid. These essential oils and phytochemicals have antibacterial properties owing to their damaging effect of bacterial cell membrane and thus causing leakage of cellular potassium [39]. Neem leaf, bark, and seeds have antimicrobial properties, and it is high, moderate, and low, respectively. Neem contains several bioactive constituents like azadirachtin and other compounds such as nimbolinin, nimbin, nimbidin, nimbidol, sodium nimbinate, gedunin, salannin, quercetin, nimbanene, 6-desacetylnimbinene, nimbandiol, nimbolide, ascorbic acid, n-hexacosanol and amino acid, 7-desacetyl-7-benzoylazadiradione, 7-desacetyl-7-benzoylgedunin, 17-hydroxyazadiradione gedunin, and nimbiol. Quercetin and β-sitosterol, polyphenolic flavonoids purified from leaves of neem have antifungal and antibacterial activities. Neem (*Azadirachta indica*) in plant parts shows antimicrobial property through bacterial cell wall breakdown [40].

6. Herbal finishes by natural dyeing

Wet processing of textile substrates from its preparatory stage to dyeing and then finishing is important for enhancing its esthetic value, removal of impurities, color shade, color pattern, and requisite functionality. Some of the traditional processes used in herbal finishing involve an excessive use of water, energy, and chemicals. Recently, due to global concerns on environmental pollution, sustainability mandates the development of sustainable dyeing and finishing processes using plant waste and nonfood plant extracts [41]. Based on environmental friendly, plant-based products having biocompatibility, biodegradability, and nontoxicity along with insect repellent, deodorizing, flame retardant, UV protection, and antimicrobial activity are in great demand all around the world for producing more appealing and highly functional value-added textiles [42–45]. A wide variety of finishing chemicals from plants are now available in the market that matches the expectations of consumers [46]. Various plants reported as source of natural dyes are teak, mahogany, ketapang, tamarind, mangosteen, mango, suji, pandan, indigofera, guava, banana, and onion [47]. Plant parts including roots, leaves, twigs, stems, heartwood, bark, wood shavings, flowers, fruits, rinds, hulls, husks, and the like used to produce herbal dye. Additionally, most of the herbal dyes have inherent medicinal properties [48].

6.1 Herbal textiles having antimicrobial finish

Natural fibers such as cotton and wool are susceptible to microbial growth and even dust mites because they retain oxygen, water, and nutrients. Hence, antimicrobial finishes should be applied to textiles to destroy or suppress the growth of microorganisms and also to protect the textiles from strength and color loss, unpleasant odor, and quality deterioration [49]. Micro-organisms may deteriorate the clothes in a closet, curtains, carpets, bed, bath and kitchen linens, pillows, and mattresses. Several microorganisms also thrive on the skin, while dust mites live on skin cells sheded on sheets, towels, and clothing. A hospital houses an immense amount of textiles with high volumes of traffic. Because of the constant flow of people, especially those with infectious diseases, specific finish hospital uses are required. Patients, caretakers, and hospital staff are at risk of acquiring infection as inherent properties of the textile fibers susceptible to the growth of micro-organisms.

In past, natural dyes were applied to textiles for simultaneous coloration and antimicrobial finishing successfully. Finish imparted by catechu on wool was found to be effective against *Escherichia coli*, *Staphylococcus aureus*, *Candida albicans*, and *Candida tropicalis*. Observed antimicrobial characteristics and negligible cytotoxicity of catechu indicated the dye as a promising antimicrobial agent for developing bioactive textile materials and herbal clothing [50, 51]. Several natural, nonmetallic, and antimicrobial finishes exist, and one of such antimicrobial finish is chitosan, a deacetylated form of chitin, which is a main component in crustacean shells and reported to be effective against both Gram-positive and Gram-negative bacteria [52]. Many antimicrobial textiles are produced with the combinations of bioactive substances to enhance the antimicrobial efficacy of the finishes and counteract the negative aspects of the treatments. By combining finishes, the occurrence of drug-resistant strains arising from the finish is reduced.

Pure cotton woven/knitted fabrics treated with the selected medicinal 16 medicinal herb extracts such as neem, turmeric, holy basil, and sandal wood have been evaluated for curing seven different diseases such as allergic dermatitis, psoriasis, asthma, liver disorders, headache, joint pain, and sinus trouble/cold. Seven different types of curative garments have been developed. The antibacterial assessments of the medicinal herb extracts treated fabrics and clinical trials have confirmed the correlation between the curative performance and its antibacterial activity. The curative property of the garments in all the cases was found to be significant and lasted for 10–15 washes [53].

Although known since long for dyeing and medicinal value, the protective properties of herbal dyes have been noticed only recently. Several plants used for dye production are classified as medicinal, and some have recently been shown to possess significant antimicrobial activity. Several natural dyes have demonstrated to possess antimicrobial activity like curcumin from turmeric; naphthoquinones such as lawsone from *Lawsonia inermis*, juglone from walnut, lapachol from taigu, and catechin from *Acacia catechu*; and several anthraquinones from *Rubia tinctorum*, *Rubia cordifolia*, *Rheum emodi*, *Punica granatum,* and *Quercus infectoria* [54, 55]. These natural antimicrobial agents may significantly reduce the risk of infections especially when they are used in close contact. Natural bioactive compounds present in natural dyes/pigments have also been reported as significant antimicrobial agents for finishing in green dyeing procedures.

6.2 Herbal textiles with UV protection

Ultraviolet (UV) rays, electromagnetic spectrum between visible light and X-rays, influence the physiology of living organisms. Exposure may cause tanning to skin cancers. Sunscreen lotions and clothing provide protection from dangers of UV rays. Change in construction parameters of fabrics with appropriate UV absorbers and adequate finishing methods may be used to prepare the UV protective fabrics.

Textiles, as a protective shield against solar radiation, have been since ancient times. Textile structures provide the desired characteristics such as pliability, good mechanical strength, softness, esthetics, and other engineered properties, which are required for preparing good sunscreening apparels. Textiles themselves are not able to provide effective protection against UV rays; hence, UV blocking agents should be applied over the fabric to make them UV protective. Quantum of protection required for different skin types is determined by UV radiation intensity and distribution subject to geographical location, time of day, and season. Several UV protection agents have been developed to enhance the UV protection offered by different textiles. Both organic and inorganic UV blocking agents are available. The organic blockers are also termed as UV absorbers because they absorb UV rays, whereas inorganic blockers scatter them [47].

Three yellow dyes obtained from *Rheum emodi*, *Gardenia yellow,* and *curcumin* have been successfully used for simultaneous dyeing and functionalization of silk to render its UV protective [56]. Wool fabric dyed with dye obtained from the eucalyptus leaves using pad-batch and pad-dry techniques showed ultraviolet protection factor (UPF) values ranging between very good and excellent [57]. UV protection properties of chlorogenic acid, the main ingredient of water extract from honeysuckle, on wool have been studied. The wool treated with honeysuckle extract showed good UV absorbance; thus, extract of honeysuckle may be developed as a natural UV-absorbing agent [58]. Natural plant colorants such as madder (*Rubia tinctorum*) and indigo (*Indigofera tinctoria*) and of insect origin cochineal (*Dactylopius coccus*) were applied on cotton fabrics, and indigo was found to have higher UPF values [59]. UV rays induce DNA damage, oxidative stress, and inflammatory processes in skin. *Rheum emodi* contains anthraquinones (rhein, emodin, aloe-emodin, chrysophanol, physcion, and rhein), stilbenoids (resveratrol and piceatannol), and flavonoids. UV protective effect of *Rheum emodi* is supposed to be due to flavonoids, which absorb the UV radiation [60]. Main coloring ingredient of the dye extracted from *Gardenia yellow* is crocin, which is a water soluble carotene, which absorbs the UV rays [61]. Curcumin alleviates the effect of UV rays by reducing the transmittance of UV light across fabric [62]. Madder roots contain anthraquinone derivatives mainly purpurin and munjistin and also small amounts of rubiadin, nordamncanthal, pseudopurpurin, and xanthopurpurin. Anthraquinones are considered to be strong UV absorbers [22].

6.3 Deodorizing herbal textiles

Bacterial growth and/or waste released from human body are the main causes for odor in garments. Recent advances in textile manufacturing have improved the performance of textiles with respect to odor with antimicrobial and UV protection properties. To meet the consumer's growing demand for hygienic clothing, extensive research has been done to deodorizing property to textiles with the application of natural colorants. The deodorizing performance of fabrics dyed with herbal colorants has been compared, and pomegranate was found to be best among gardenia, *Cassia tora* L., coffee sludge, and pomegranate rind [23]. Fabric dyed with gallnut recorded a better deodorizing function against ammonia, trimethyl amine, and acetaldehyde than control fabrics. Dyed fabrics also found to possess an excellent antimicrobial activity against *Staphylococcus aureus* and *Klebsiella pneumoniae* [20]. Young-Hee Lee and co-workers dyed cotton, silk, and wool fabrics with pomegranate (*Punica granatum*) and showed excellent deodorizing property in the dyed fabrics [21]. Cotton fabrics dyed with C.I. Direct Blue 200, a copper complex direct dye, and pre- and post-mordanted with Cu(II) sulfate for deodorization of ethyl mercaptan showed that deodorization effects increased quadratically with the copper ion uptake [63]. These studies indicate that natural and synthetic dyes can be utilized for deodorizing functionalization of textiles.

6.4 Moth resistant and insect repellent textiles

Carpets, blankets, and shawls are prepared from wool, and other similar fibers have properties such as warmth, softness, and flame retardancy protein content of the fiber, which are susceptible to attack by moth as its larvae thrive on the protein present in the wool. Cloth moth (*Tineola bisselliella*) and carpet beetle (*Anthrenus verbasci*) are common moths, which attack the wool items. Dichlorodiphenyltrichloroethane (DDT), permethrin, permethrin/hexahydro pyrimidine derivative, and cyhalothrin are some of the insecticides used to provide antimoth finish. Nano titanium oxide particles have also been utilized as an antifeeding compound on wool fabric to kill larvae of the carpet beetle, *Anthrenus verbasci*, which feeds on wool fibers [64]. Chemicals used for antimoth finishing are detrimental to the environment; hence, natural colorants may be good alternatives. Shakyawar et al. used saffron flower waste, onion skin, henna, myrobolan, silver oak leaf, madder, walnut, dholkanali, and yellow root natural dyes for antimoth finishing and recorded the best results for silver oak leaves, walnut husk, and pomegranate rind [65]. Natural dyes from cochineal, madder, walnut (quinines), chestnut, fustic, indigo, and logwood (flavonoids) were also applied on wool, and their antimoth properties against black carpet beetles were tested, all except indigo, which increased the insect resistance of the wool fabric. Metallic mordants were found to have no significant effect on insect resistance with all natural dyes used. The anthraquinone dyes including cochineal, madder, and walnut were found to be quite effective in protecting wool fabric against black carpet beetles [66].

7. Production and consumption of herbal textiles

Synthetic/chemical dyes are increasingly being banned for use in the textile industry, which have led to severe blow to the handloom industry. At the same time, textiles dyed with natural herbal dyes having medicinal attributes are commanding a huge market due to their inherent advantages. Nowadays, the use of herbal dyes in the textile industry is preferred owing to the advantage of dyes extracted from the medicinal plants, origin from the renewable resources, limited chemical reactions involved in their preparation, biodegradable properties, disease-curing properties, and ecofriendly in nature [18, 67].

Herbal textiles are of two kinds: one category is for curing diseases and the other is for wellbeing. The first category of cloths, which are used for curing diseases, includes sleep wear, bed sheets, towels, meditation clothes, and coir mats. Herbal textiles are mostly used in making such garments that stay close to human skin, so that all the medicinal components could be absorbed through the skin. Herbal textiles can fetch more competitive than conventional fabrics owing to low production cost. For example, a common chemical dye costs around $75 per kg, and a herbal dye costs approximately $5 per kg. Most of the herbs used in dyeing are cultivated in South Asian countries, namely India, Pakistan, and Bangladesh, India being the largest producer. The Handloom Weavers Development Society (HLWDS) of Kerala, India has exported herbal and organic textile worth Rs. 50 Lakh to the United States, Europe, and Japan. The herbal textile is gaining such popularity that the Japanese Government has accorded HLWDS a $40,218 grant. Large consignments of herbal textiles have been shipped to international markets of the United States, Canada, France, Denmark, Italy, Poland, Maldives, Mauritius, Japan, and Sri Lanka [17, 68–70].

Most effective time to wear the herbal-infused clothing is while resting, sleeping, or meditating, when the body is naturally healing and re-establishing balance, so many of the products are created with these factors in mind. The technique for producing the herbal textiles is also used for floor coverings and coir mats. For coir mats, the fibers are first soaked in herbal dyes and then woven into coir mats. Apart from medicated handloom clothes such as sarees, T-shirts, shirts, trousers, kurtas, dhods, chudidhars, nightwear, fashion wear, sportswear, and inner wears, caps for healing headaches, goosery caps for head balance, cooling caps, hair shining caps, hair strengthening caps, bandages, and mask are also prepared [68, 71].

Plant dyes are increasingly being incorporated by designers into their designs contributing to revival of Ayurvastra or herbal clothing. Ayurganic is a line of loungewear inspired from the concept of Ayurvastra. Designer duo Lecoanet and Hemant have revived the tradition of Ayurvastra in Ayurganic, their line of medicinal clothing. The multi-award winning designers who began their journey as couturiers in Paris are now refining the concept of Ayurvastra. Anjelika dreams organic produces fair trade, organic handmade clothing that follows this tradition. Gibie towels specialize in Ayurvastra towels, Ayurvastram in Ayurvastra textiles and yoga mats, and niraamaya in Ayurvastra yoga mats. The handloom weavers' development society in India is producing a wide range of herbal fabrics including sarees, bed sheets, and dress materials using medicinal dyes and exporting them to the United States, Germany, the United Kingdom, Singapore, and Taiwan [6, 11].

"Herbalfab" ecofriendly fabric brand has developed a unique technique to dye with flowers, fruits, roots, and so on. Turmeric, myrabolams, onion, kesu and dhavadi flowers, and natural indigo are used for preparing the natural dyes. This prevents the water pollution by replacing the petrochemical dyes with herbal dyes and imparts the medicinal value to cloth. The colors obtained are unique, which can kick start a new fashion trend. Herbalfab also encompasses a range of other ecofriendly fabrics like khadi, which is handspun and handwoven, nonviolent silk, organic denim, and so on [72].

The Handloom Weavers' Development Society, India produces a variety of home textiles using herbal fabrics in India. They also export herbal clothing such as sarees, bed sheets, and dress materials to other countries. With increasing demand for herbal clothing, companies such as Ayurvastra online have started offering the online products. Many textile industries are getting inclined toward Cosmetotextiles. The brands making herbal clothes are Aura, Cognis, Pantaloons, Quiospheres, and so on. These brands have a range of clothing namely bandee, vest, camisole, men and women's tee, coirs, and so on [73].

8. Benefits and drawbacks of herbal textiles

8.1 Health benefits

- It acts as a revitalizing tonic and helps in keeping the body fresh and healthy. The uses of proper herbs in the textiles have proven to cure diseases such as arthritis and fever. We breathe through our body more than we do through nose. It can improve the skin's natural ability to block and resist the harmful chemicals and toxins from entering the body, which will be beneficial to health.

- Herbal cloths have the ability to protect us from various skin diseases and provide relief from infectious diseases and mental ailments.

- No synthetic/chemical dye is used at any stage of herbal cloth production. For washing and cleaning purpose, the nut or nut powder of *Sapindus laurifolia* is recommended [74].

- Medicinal herbs treated fabrics also have a lot of therapeutic value; thus, the fabric has been found very helpful for people suffering from ailments such as skin allergies, breathing problems, sleeping disorders, and blood pressure.

- The health benefits of herbal clothing and its usage depend on the theory of touch. The body loses toxins when it comes in contact with herbal clothing, and this improves the metabolism. Herbal clothing is also known to help fight against many common diseases such as diabetes, hypertension, skin allergies, asthma, and heart ailments.

- The human body naturally heals itself during sleep or meditation. Thus, when the body is at rest, herbal clothing will work most effectively. The skin is known as the largest organ of the body. Not only it acts as a fence and protects the body, but it can also be the channel for outside germs and toxins to enter into the body. Herbal clothing guards against the harmful toxins trying to enter the body through the skin [75, 76].

- Herbal textiles are mainly used in making sleepwear, undergarments, bed coverings, towels, meditation clothing, and so on, which remain close to the skin absorbing all the benefits it gives out. Herbal textiles are also used in home textile products such as mattresses, coir mats, door mats, bath towels, bed spreads, and carpets.

- Some of herbal constituents are antiallergens and hence are safe for skin contact and are mostly nonhazardous to human beings.

- Natural dyes are usually moth proof and can replace the synthetic dyes in kids' garments.

- We breathe through our skin more than we do through nose, and chemically dyed textiles having carcinogenic amines and chemicals may be allergic and dangerous to human skin. Organic clothing can help reduce the exposure to allergens and other irritants and give a comfortable feeling [77–79].

8.2 Environment benefits

- Preparation of herbal cloths is a labor-intensive industry and hence will provide the job opportunities for manpower engaged in cultivation, extraction, and application on textile, food, leather, and so on.

- Use of natural dyes may earn the carbon credit as it reduces the consumption of petroleum-based synthetic dyes.

- It promotes the agriculture and balances the ecological cycle.

- Waste can be used as manure.

- No heavy metals such as chrome and copper are used in natural/herbal Ayurvedic dyeing.

- Manual farming and organic practices have a lower carbon footprint as the entire process consumes less fuel and energy and emits fewer greenhouse gases than chemical textiles.

- Herbal dyes are extracted from the plants cultivated without the use of synthetic pesticides and fertilizers, and also no chemical defoliants are used.

- Processing is in green environment; hence, workers' health is not compromised, and also water and electric use and toxic runoff are reduced.

- Stringent testing ensures that product is free from contaminants such as nickel, lead, formaldehyde, amines, and pesticides.

- It improves the soil fertility and soil structure by increasing the soil activity, thus reducing the risk of erosion.

- It is the massive saving of precious water.

- It promotes the development of earthworms and other arthropods leading to improvement in the growth conditions of the crop. Furthermore, organic crops thrive on root symbioses and are better equipped to exploit the soil; hence, fields can accommodate a more number of plants, animals, and microorganisms [80–82].

Natural fibers and natural dyes used in the preparation of herbal clothing allow its natural breakdown without damaging the environment compared to the synthetic material, which takes hundreds of years to breakdown and leaves the pollutant material in soil [66, 67, 83].

8.3 Other benefits

- Herbal textiles are ecofriendly, and also residues they produce can be further used for making the other green products.
- Solid and liquid wastes from herbal dyeing are amenable to recycling producing the organic manure.
- Some of the ecofriendly industries producing the herbal textiles produce organically recycled paper bags by utilizing the residual organic and herbal dyed fabrics that can reduce the pollution due to plastic bags [84].
- It also promotes the cultivation of herbs required for the production of herbal textile products.
- Herbal dyes add value to the cotton fabrics in ecofriendly manner.
- Herbal fabrics are lightweight and so used to construct the perfect breezy clothes. The biggest advantage of cotton herbal cloth is that it is cool in summer and warm in winter [85, 86].
- The color shades produced by herbal dyes are unique, hence commanding high demand in market.

8.4 Drawbacks

- Herbal fabrics and dyes are not good substitutes for synthetic dyes offering broader range and variations.
- Retaining color and its herbal benefits demands utmost care in washing; herbal cloths should be washed separately, preferably hand wash or gentle machine wash with bleach-free detergents and should be shed dried. This causes inconvenience to the user.
- The dyeing process is cumbersome and time taking, and each stage should be carefully controlled.
- Medicinal herbs impart colors; hence, color choice for consumer is very limited as only few herbs can be used for dyeing a fabric that meant to be used in a particular disease condition.
- Herbal cloths show the curative effects, but progress is slow.
- Though there are no complaints on allergic reaction by the consumers, some people think that the herbal clothing shows side effects on children [87–89].

9. Conclusion

Textile industry has realized its negative contribution to the environmental pollution through the harmful chemicals used in manufacturing processes and is continuously trying to find solution for this problem. Herbal dyeing is one important step toward achieving the organic lifestyle, thus reducing the environmental pollution. Herbal textile products are devoid of pesticides, fertilizers, antibiotics, growth hormones, genetically modified organisms, additives, irradiation, or sewage sludge, hence avoiding many ailments that are common with conventional clothing.

Modern consumer's demand for novel ecomaterials is expected to increase in future. Recently, efforts have been made toward the development of commercially viable natural colorants by making advances in aspects such as identification of new sources, formulations, extraction, purification, and stability techniques. In spite of enthusiastic studies discussing the socioeconomic viability of natural dye production and applications at commercial scale for sustainable utilization of bioresources, there is a need for investigations related to hygiene and ecosafety, which have great future for the discovery of relatively better and more stable natural pigments that may have wider industrial applications.

Herbal textiles have tremendous scope in world textile market and may become a major textile product in future. The blend of herbs and textiles to achieve the health in an ecofriendly manner is the great way of adopting the healthy life. Herbal clothing is receiving the increased awareness all over the world. Herbally treated textiles are one of the great solutions to revive and increase the share of Indian handloom industry in world textile market. Furthermore, herbal clothing is nontoxic, noncarcinogenic, biodegradable, and ecofriendly; these characteristics make them an obvious choice for modern informed costumer looking for a healthy product. Additionally, the production of biocolorants to meet the rising demand shall force entrepreneurs to take up this venture for greater profits leading to more employment generation.

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Chapter

A Practical Guideline of Few Standardized Ready Made Shades of Natural Dyed Textiles

Anowar Hossain

Abstract

Marigold flower *Tagetes erecta* L., Arjuna Bark *Terminalia Arjuna*, Eucalyptus leaves *Eucalyptus Radiata*, Peach/Jam Leaves *Acacia acuminata*, Pecker leaves *Cinnamomum tamala*, Guava leaves *Psidium guajava*, Basil leaves *Ocimum basilicum*, Jackfruit wood *Artocarpus heterophyllus*, Catachu fruit *Senegalia Catechu*, Bohera fruit *Terminalia bellirica*, Betel nut fruit *Areca catechu*, Haritaki fruit *Terminalia chebula*, Mahogany fruit peel, Mahogany seed peel, and Mahogany seed *Swietenia macrophylla* are the common natural sources in Bangladesh, an Asian country which were experimented in terms of mordanting free natural coloration on cotton fabric under conceptual confirmation of referred journal where author has been picked the idea from the generation of available shade in his research laboratory and tested from different laboratories and it may be establish as mordant free natural dyeing for specific colorant on the basis of color fastness and shading behaviors. Fifteen standardized Ready Made Shade (RMS) has been presented with CIE color parameters, color fastness, wash fastness, and light fastness grading. A reproducing guideline for every Ready Made Shade (RMS) has been mentioned in this chapter.

Keywords: readymade shade (RMS), natural dyeing, eco-friendly dyes, natural extraction, mordant free dyeing

1. Introduction

A concept of readymade shade (RMS) has been developed by using numerous number of natural dyes which were collected from agri-production unit and different village sources in Bangladesh prepared and powdered in color processing unit, extracted with water solvent process, dyed and experimented the feasibility of coloration in textile chemistry laboratory to generate different hues of natural dyes after effective implementation on natural fiber based textiles which outcome of color may be beneficial for primary selection of color tone by the textile technologist of natural coloration cum customers for the decision-making of specific color tone both for product development and fashion concerns who are genuinely searching an eco-friendly dyes under the consideration of repeated hue without which the real output of natural coloration, sustainability of dyes and natural dyeing process as well as its actual production of different hue is being a challenged now as the textile technologists are habituated the essence of readymade shading behavior of synthetic dyes whereas toxicity is the main endangered for the human being.

2. Background

Environment pollution is the great challenge of color scientists in dyeing and finishing plant, so eco-friendly coloration is the key target of recent researchers and manufacturers. Natural coloration is being accepted by the environment scientist and related committee [1–3]. Researchers in the area of natural coloration have an immense intension to minimize pollution [4]. Focus on science and engineering on natural dyes based research, extractions, purifications, and implementations are rapidly climbing [5–9]. In this chapter, mordant free natural coloration and its feasibility was experimented to minimize environment pollution load which is not limited to synthetic dyeing but also natural dyeing in terms of mordant free coloration concepts for specific standardized shade. Author also experimented to establish an approach of green mordanting [10, 11] in natural coloration [12–16] although mordant [17] has an effect for the augmentation of fiber surface color and surface chemistry [18] and its appropriate affinity in terms of fastness properties [1, 19–24]. Light fastness [21–24] of natural dyed fabric is a critical issue for natural colorant if dyes sources and collection processes are not maintained accurately. There are many sources of natural colorant, using by the researcher as per availability of origin to origin in the world. Researchers are already invented and proposed like Jatropha flower [25], red sandal wood [26], wood of *Artocarpus heterophyllus* [27], Acalypha [28], areca nut [29], *Butea monosperma* [30], neem leaves [31, 32], Gomphrena globosa [33], Onosmaechiodes [34], *Nerium oleander* flower [35], Kesula flowers [36], Parijataka (Nyctanthes Arbor) flowers pigment [37], Hibiscus ovalifoliusand *Sesbania aegyptiaca* [38], Bark of Macaranga Peltata [39], Cutch, Ratanjot and Madder [40], Mesta Calyx [41], Kapila, Onion, Tesu [42], myrobalan, gallnut, pomegranate [43], Marigold flower [44, 45], *Areca Catechu* [46], Eucalyptus leaves [47], Eucalyptus bark [48, 49], Jackfruit wood [50], Peach [51], Arjuna [52, 53], Catechu [54] and others. Developments of natural indigo shade in comparison with synthetic dyes [55] are not only prime objects for coloration but also maximum natural dyes have medicinal value [56, 57], noncarcinogenic, production-friendly, and environment-friendly. Research and development of natural dyeing on cotton, jute, and silk fabric was done by Samanta and his researchers team at DJFT, University of Calcutta, India have an technical output, research impacts and motivation for future researchers, and manufacturing unit for the contributions of dyes selection, extraction, analysis, standardization, and commercialization [58–68]. Color matching [69] and reproducing of shade is another challenging issue for the manufacturing company and colorists when natural dye is compared with synthetic dyes but there are so many colorants of natural sources that can be possible to reproduce almost nearest shade. Tolerating percentage/acceptable range of color differences, ΔE value, and other properties can be standardized in a convincing way with local and foreign customer of any country as the real customer of any textile product is not going to sell the product after shade matching with spectrophotometer without enjoying its esthetic value, so a little bit lightness and darkness matter is not a trading challenge of dyer and manufacturing plant. Comparing to synthetic dyes, natural dyed fabric has a great market demand as the people who are concern about the intimidation of environment. Thus natural dyes have diverse applications and multi fiber based production options that are already available in literature for cotton [68], jute [66], wool [70], silk [34] based dyeing, processing in both laboratory stage and bulk production (**Figures 1**–**15**).

RMS of Marigold (Figure 1): Waste Marigold flowers were collected from flower garden, washed and 5 days dried with summer sun light, and made it crispy and fine powdered with grinding machine. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

3. Standardized Ready Made Shade (RMS) of natural dyed textiles

3.1 Natural coloration of cotton fabric with Marigold flower, types of dyes: natural, source of dyes: Marigold tree. scientific name: *Tagetes erecta*

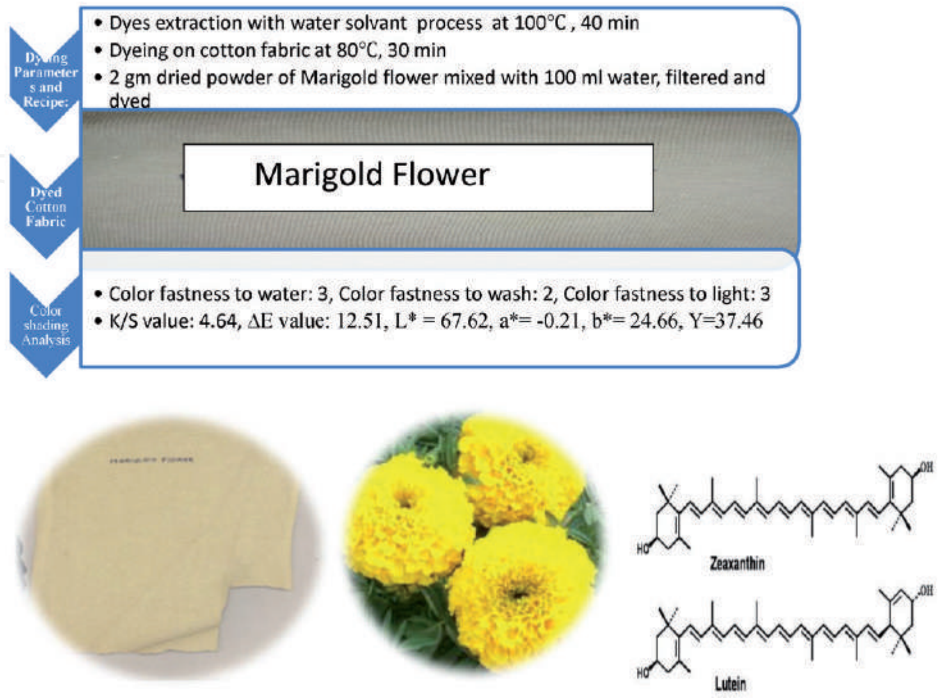


Figure 1.
RMS of Marigold.

3.2 Natural coloration of cotton fabric with Arjuna Bark, types of dyes: natural, source of dyes: Arjuna tree, Scientific name: *Terminalia arjuna*

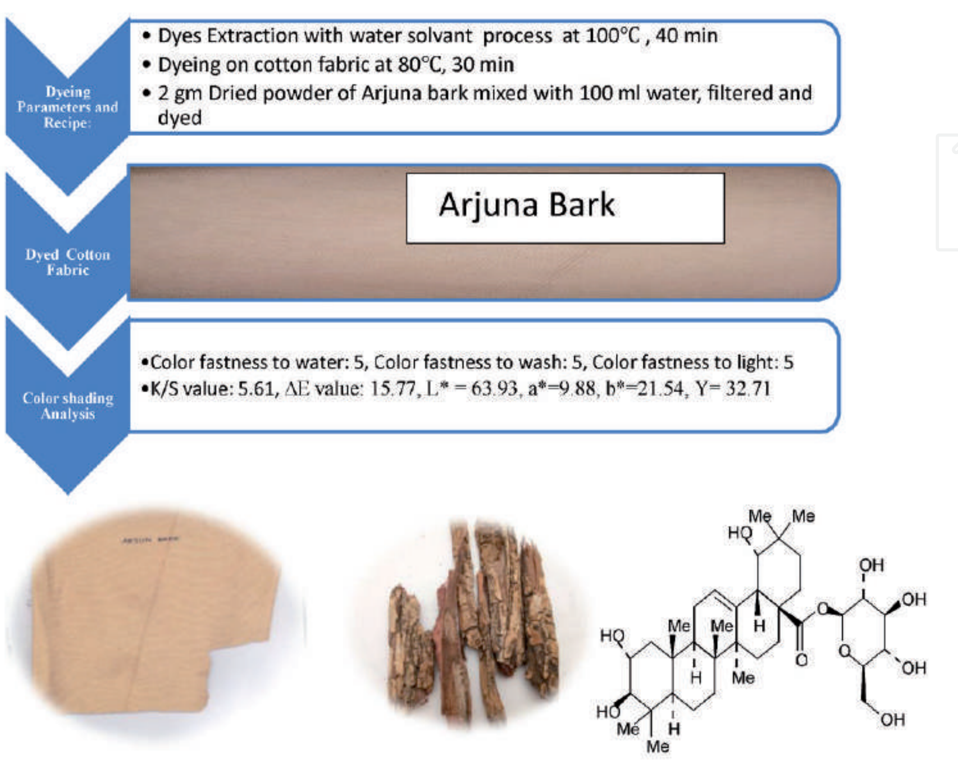


Figure 2.
RMS of Arjuna Bark.

3.3 Natural coloration of cotton fabric with Eucalyptus leaves, types of dyes: natural, source of dyes: scientific name: *Eucalyptus radiata*

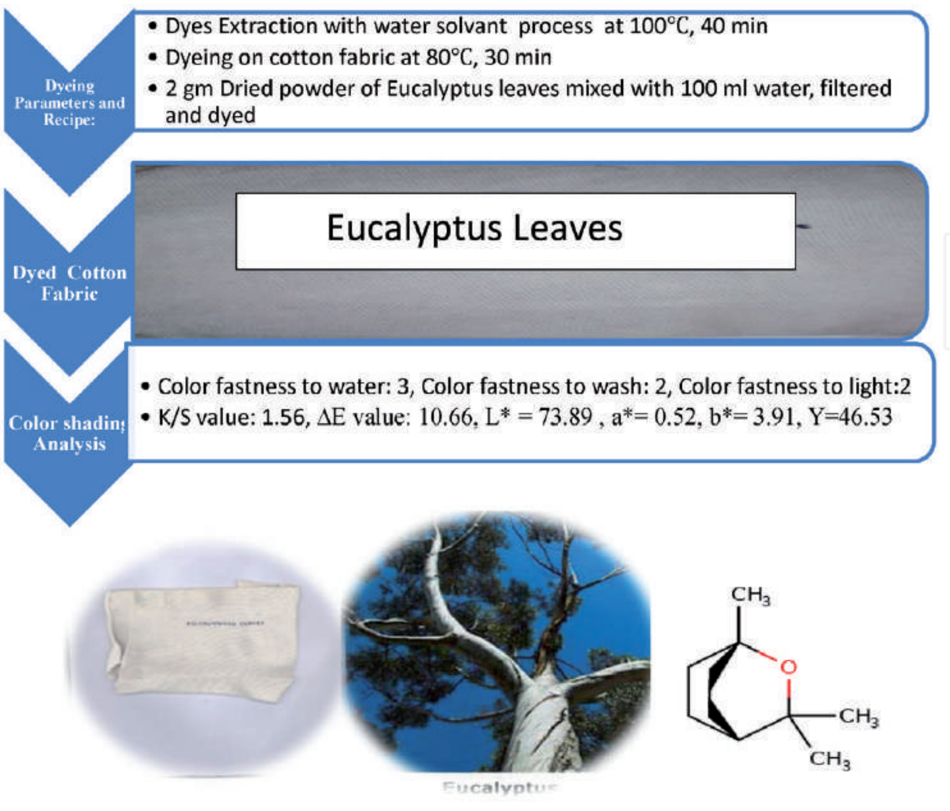


Figure 3.
RMS of Eucalyptus leaves.

Coloring components Zeaxanthin and Lutein structural group of marigold flower may be responsible for good color combination has OH group may be showed good color fastness properties with cellulosic fiber. Lutein ($C_{40}H_{56}O_2$) and Zeaxanthin ($C_{40}H_{56}O_2$) molecules may prevent UV damage for its strong antioxidant properties.

RMS of Arjuna Bark (Figure 2): Dried Arjuna bark were plucked from Arjuna tree, washed and 5 days dried with summer sun light, and made it crispy and fine powdered with grinding machine. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

Ferrous ion of Arjuna Bark may be influencing the L*, a*, b*, Y value for providing outcome of deeper color and OH group is responsible for good fastness properties. Arjuna bark has phytosterol, lactones, flavonoids, phenolic compounds, and tannins, glycosides where tannin may be responsible for coloring agent.

RMS of Eucalyptus Leaves (Figure 3): Semi-dried leaves were plucked from Eucalyptus tree, washed and 5 days dried with summer sun light, and made it crispy and fine powdered with grinding machine. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

OH group of cellulose can easily make a bonding with dyes group of Eucalyptus. Eucalyptol is a colorless compound which is responsible for antibacterial properties but may be remaining pigment or other phytochemical compound is making color on the fabric surface.

3.4 Natural coloration of cotton fabric with Pecker leaves, types of dyes: natural, source of dyes: Pecker tree, scientific name: *Cinnamomum tamala*

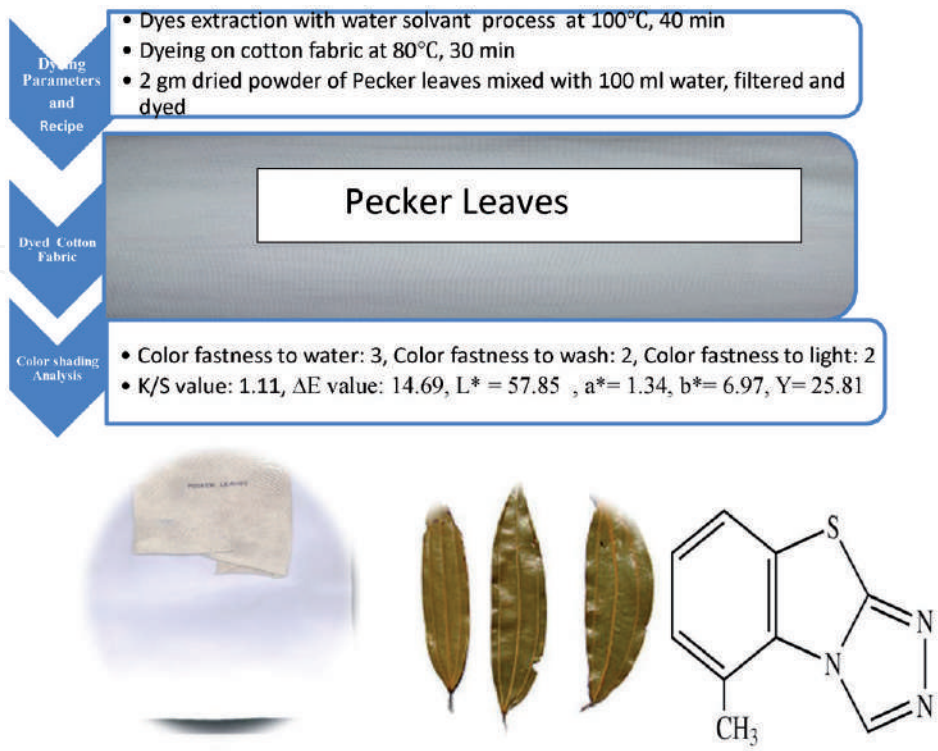


Figure 4.
RMS of Pecker leaves.

RMS of Pecker Leaves (Figure 4): Semi-dried leaves were plucked from pecker tree, washed and 5 days dried with summer sun light, and made it crispy and fine powdered with grinding machine. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

OH group of cellulose can easily make a bonding with dyes group of Pecker leaves. Eugenol is the main constituent of antimicrobial properties and other components like pinene, camphene, and limousine may be responsible for color formation.

RMS of Guava Leaves (Figure 5): Semi-dried leaves were plucked from tree, washed and 5 days dried with summer sun light, and made it crispy and fine powdered with grinding machine. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

OH group of dyes is responsible for good color fastness properties. Mixed components of flavonoid and tannin are highly responsible for bluish color of cotton fabric. Acidic pH and phenolic compound of guava leaves may generate flammable characteristics of guava leaves colored fabric.

RMS of Basil Leaves (Figure 6): Fresh and green Basil leaves were plucked from tree, washed and 5 days dried with summer sun light, and made it crispy and fine powdered with grinding machine. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

α terpineol remaining in dyes of Basil leaves may be responsible for dyeing of cotton fiber and OH group is making strong affinity with cellulose. Phytochemical constituents and medicinal properties of Tulsi leaves may create pathogen protective finish of cotton fabric like antimicrobial, COVID 19, and other viruses.

3.5 Natural coloration of cotton fabric with Guava leaves, types of dyes: natural, source of dyes: Guava tree, scientific name: *Psidium guajava*

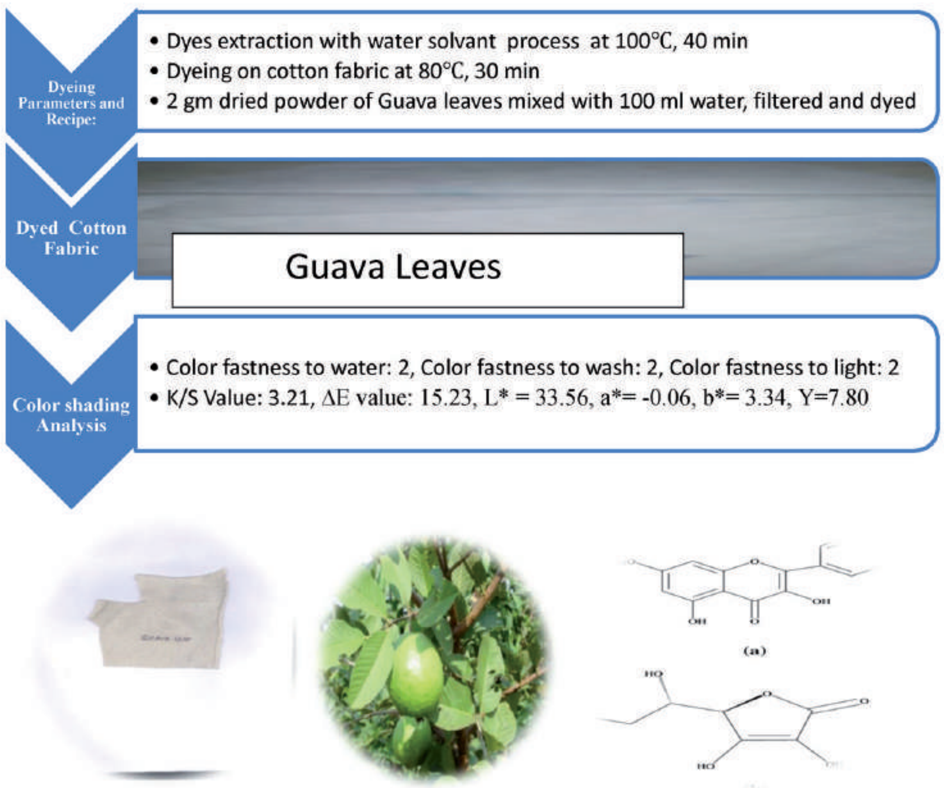


Figure 5.
RMS of Guava leaves.

3.6 Natural coloration of cotton fabric with Basil leaves, types of dyes: natural, source of dyes: Basil tree, scientific name: *Ocimum basificum*

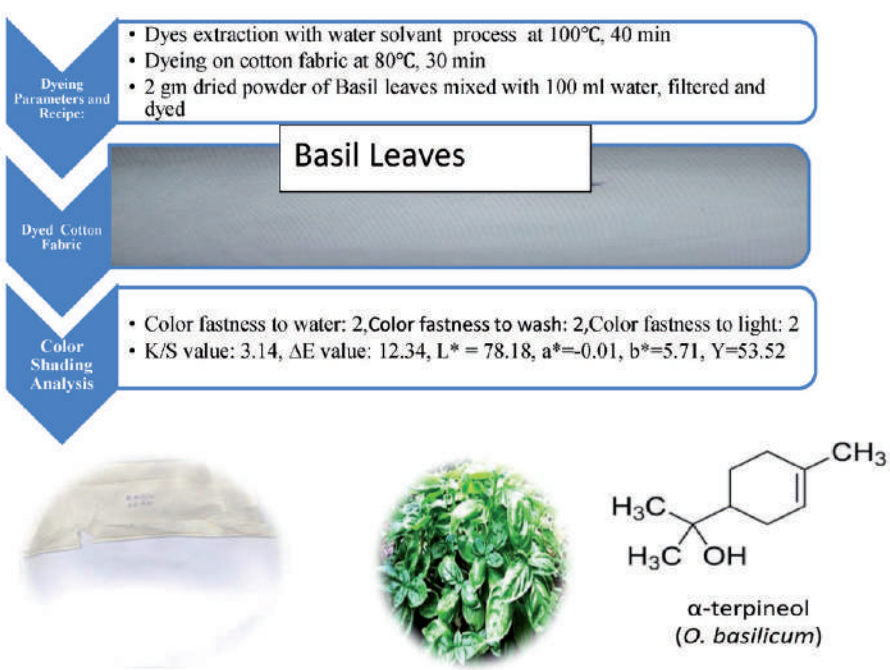


Figure 6.
RMS of Basil leaves.

3.7 Natural coloration of cotton fabric with Jackfruit wood, types of dyes: natural, source of dyes: Jackfruit tree, scientific name: *Artocarpus heterophyllus*

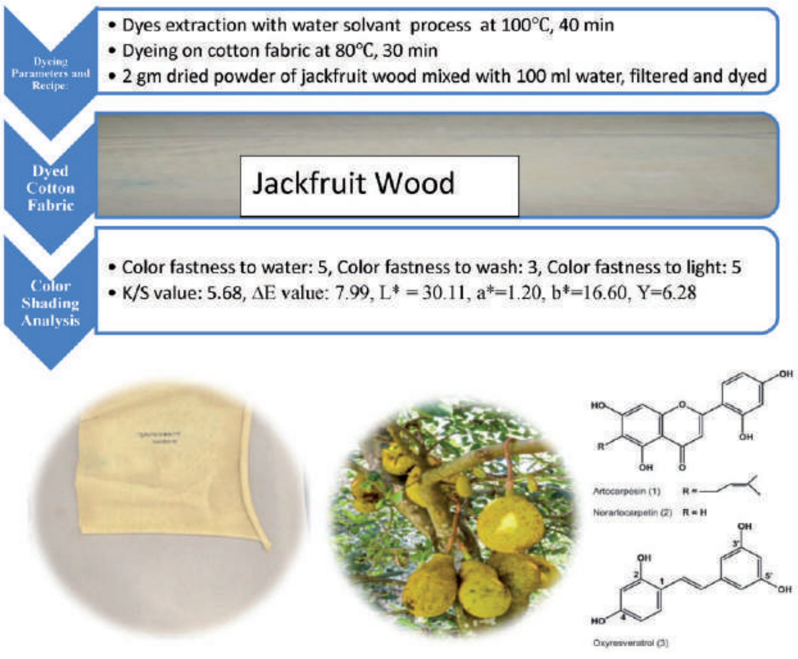


Figure 7.
RMS of Jackfruit wood.

3.8 Natural coloration of cotton fabric with Catechu fruit peel, types of dyes: natural, source of dyes: Catechu tree, scientific name: *Senegalia catechu*

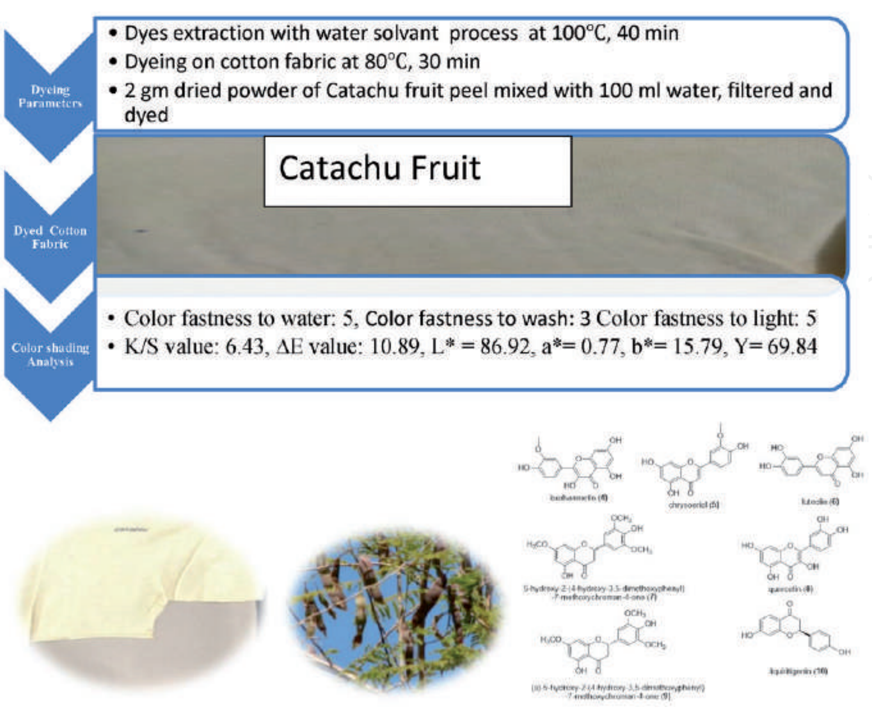


Figure 8.
RMS of Catachu fruit.

3.9 Natural coloration of cotton fabric with Bohera fruit peel, types of dyes: natural, source of dyes: Bohera tree, scientific name: *Terminalia bellirica*

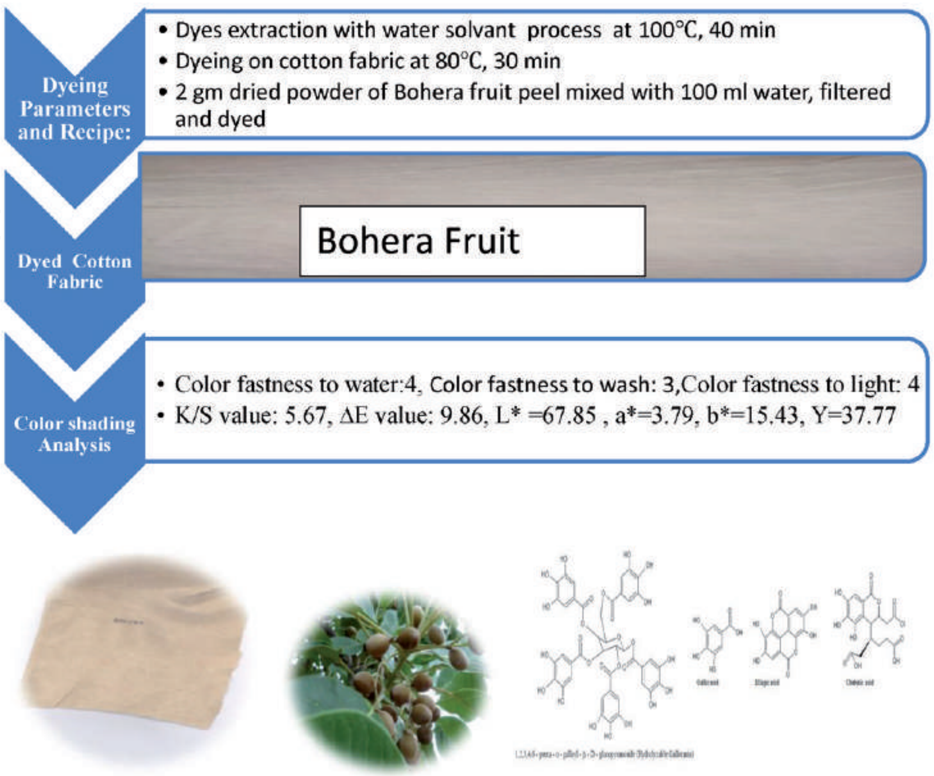


Figure 9.
RMS of Bohera fruit.

RMS of Jackfruit Wood (Figure 7): Powder of Jackfruit wood were collected from saw mill, filtered and 5 days dried with summer sun light. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

Artocarpesin and Norartocarpetin group of golden yellow color jackfruit tree wood has phenolic compounds, apigenin, curcumin may be responsible for making a yellow color having an optimum value of L*, a*, b*, Y as well as OH group is improving color fastness properties.

RMS of Catachu Fruit (Figure 8): Semidried Catachu fruit were plucked from tree, washed, fruit peel were cut into small piece, 5 days dried with summer sun light, and made it crispy and fine powdered with grinding machine. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

Catachu is making off white color with soft hand feel and may be remaining natural pigment in the chemical components is responsible for dyeing without mordanting. Coloring component in the catechu is catechin having molecular formula $C_{15}H_{14}O_6$.

RMS of Bohera Fruit (Figure 9): Bohera fruit powder was purchased from herbal medicine shop, Tongi, Dhaka, Bangladesh. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

3.10 Natural coloration of cotton fabric with Mahogany seed, types of dyes: natural, source of dyes: Mahogany tree, scientific name: *Swietenia macrophylla*

Figure 10.
RMS of Mahogany fruit, seed.

3.11 Natural coloration of cotton fabric with Mahogany fruit (outer peel), types of dyes: natural, source of dyes: Mahogany tree, scientific name: *Swietenia macrophylla*

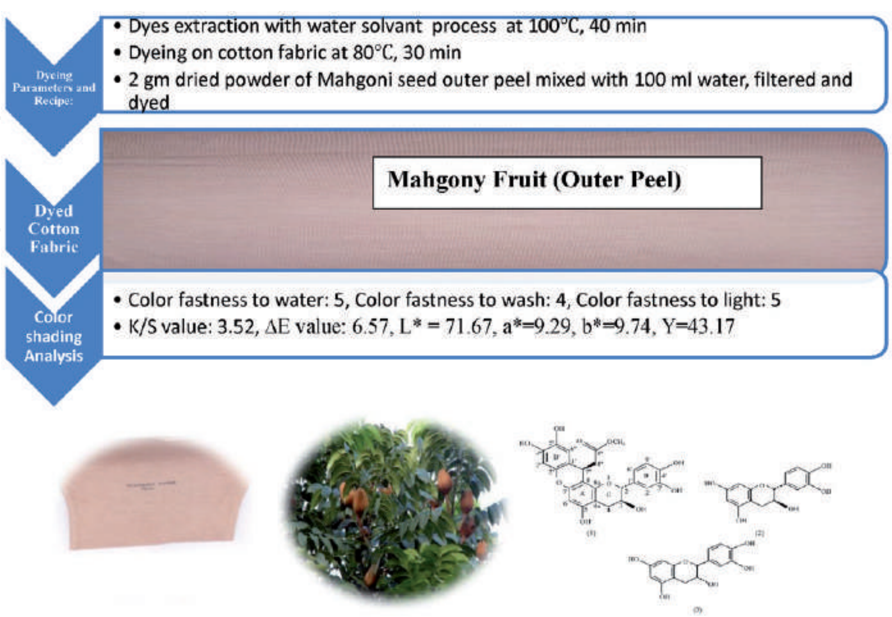


Figure 11.
RMS of Mahogany fruit, outer peel.

3.12 Natural coloration of cotton fabric with Mahogany seed (outer peel), types of dyes: natural, source of dyes: Mahogany tree, scientific name: *Swietenia macrophylla*

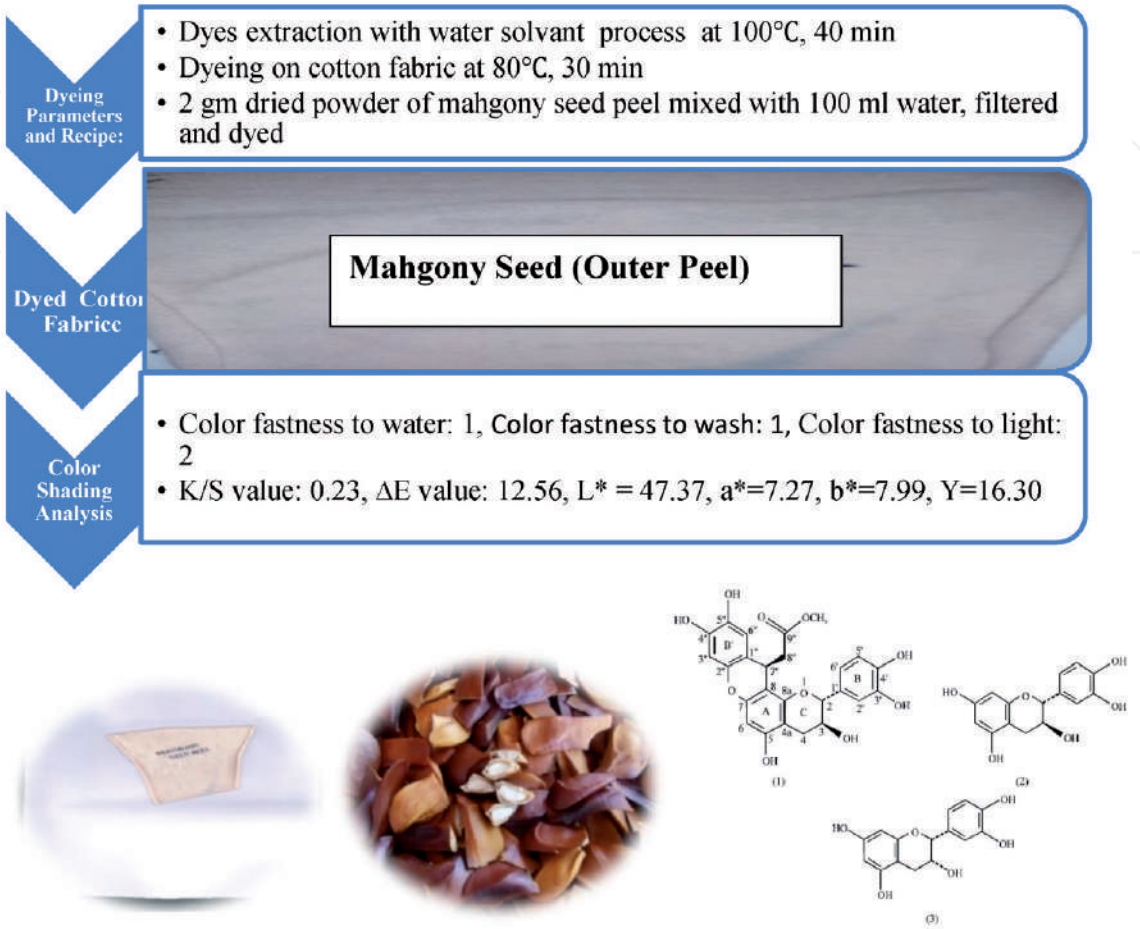


Figure 12.
RMS of Mahogany fruit, outer peel of seed.

OH group and its tannin constituent may be created a natural shading environment on the surface of cotton fiber and its medicinal properties may create the dyed fabric antimicrobial. Flavonoid and falvins constituent of Bohera fruit may have adaptive capabilities of viruses and microorganisms.

RMS of Mahgony Fruit, Seed (Figure 10): Semi-riped Mahogany fruits were plucked from tree, washed, seeds were separated with sharp knife, cut into small pieces and 5 days dried with summer sun light, and made it crispy and fine powdered with grinding machine. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

OH group may be responsible for good dye-absorption and ferrous ion may be responsible for dyeing. Phytochemical constituents and other medicinal properties may have a good source protective clothing like insect repellent, antimicrobial, COVID19, and other viruses.

3.13 Natural coloration of cotton natural coloration of cotton fabric with Haritaki fruit peel, types of dyes: natural, source of dyes: Haritaki tree, scientific name: *Terminalia chebula*

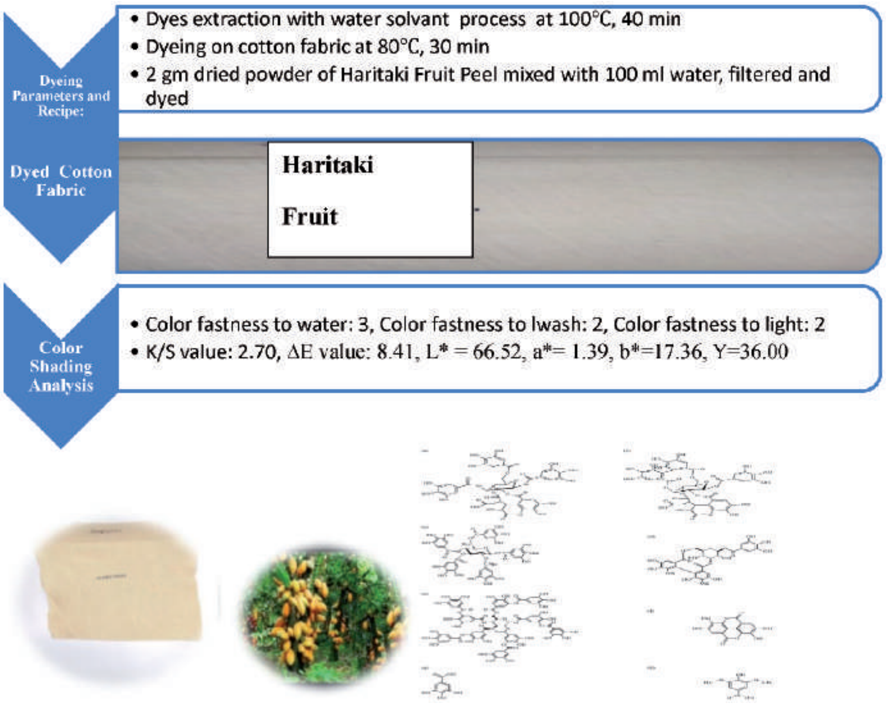


Figure 13.
RMS of Haritaki fruit.

3.14 Natural coloration of cotton fabric with Betel nut, types of dyes: natural, source of dyes: Betel nut tree, scientific name: *Areca catechu*

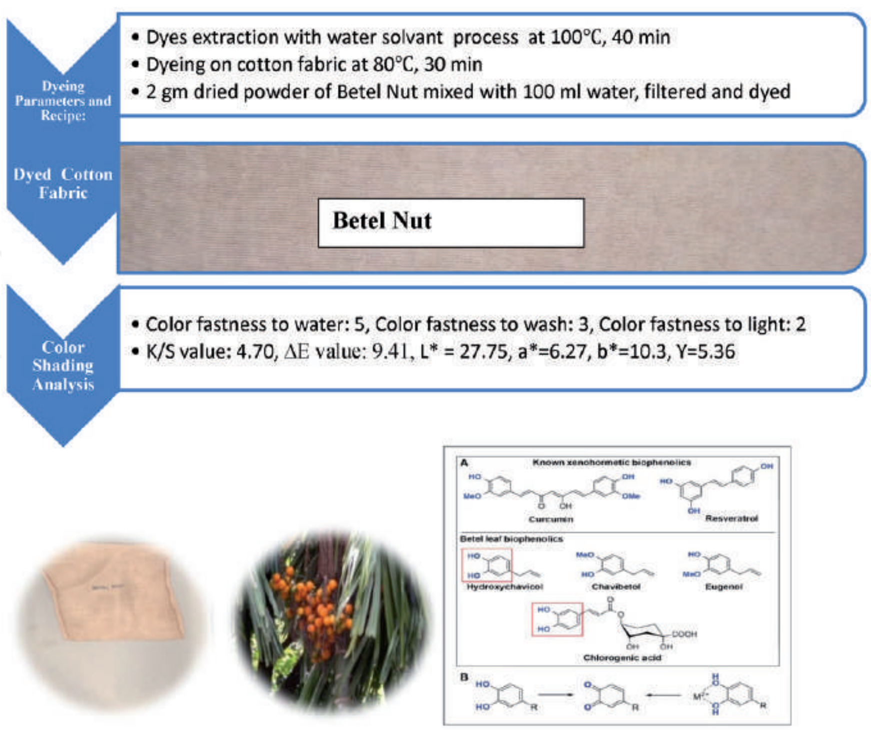


Figure 14.
RMS of Betel nut.

3.15 Natural coloration of cotton fabric with Peach leaves, types of dyes: natural, source of dyes: Peach leaves, scientific name: *Acacia acuminata*

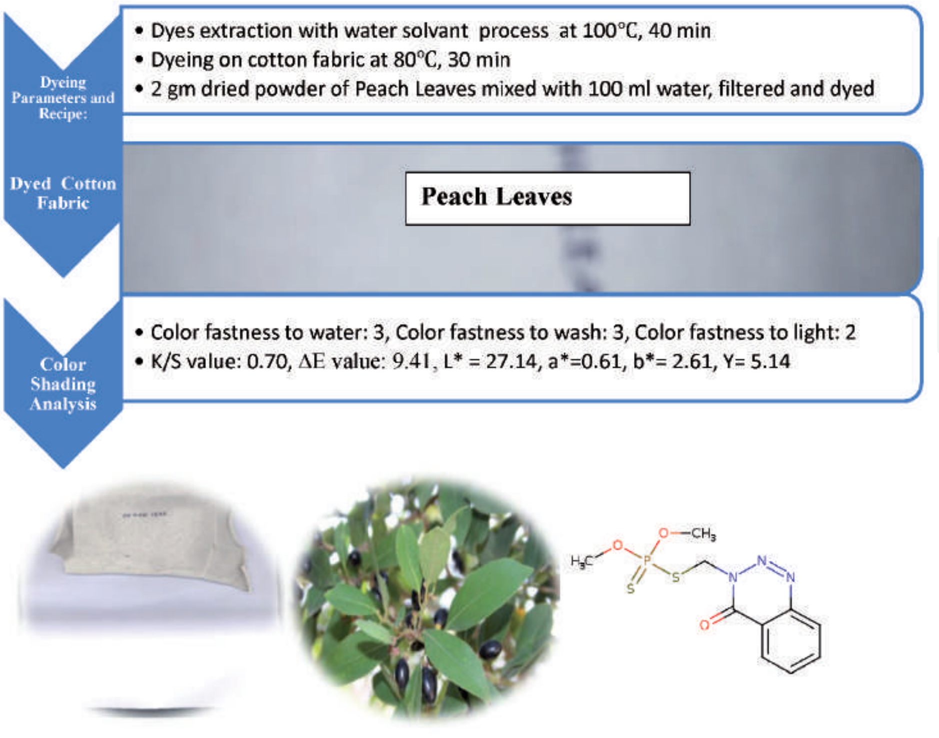


Figure 15.
RMS of Peach leaves.

RMS of Mahgony Fruit, Outer peel (Figure 11): Semi-riped Mahogany fruit were plucked from tree, washed, fruit peels were separated with sharp knife, cut into small pieces and 5 days dried with summer sun light, and made it crispy and fine powdered with grinding machine. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

OH group and ferrous ion may be responsible for good dye-fiber bonding phytochemical constituents and other medicinal properties may have a good source protective clothing like insect repellent, antimicrobial, COVID19, and other viruses.

RMS of Mahgony Fruit, Outer peel of Seed (Figure 12): Semi-riped Mahogany fruit were plucked from tree, washed, seed peels were separated with sharp knife, cut into small pieces and 5 days dried with summer sun light, and made it crispy and fine powdered with grinding machine. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

OH group and ferrous ion may be responsible for good dye-fiber bonding. Phytochemical constituents and other medicinal properties may have a good source protective clothing like insect repellent, antimicrobial, COVID19, and other viruses.

RMS of Haritaki Fruit (Figure 13): Haritaki fruit powder was purchased from herbal medicine shop Tongi, Dhaka, Bangladesh. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

OH group and ferrous ion may be responsible for good dye-fiber bonding. The plant is constituted of Glucoside, Tannins, Gallic acid, Ethyl gallate, and Chebulinic acid where tannin may be responsible for coloring the cotton fiber.

RMS of Betel Nut (Figure 14): Ripe Betel nut were purchased from local village shop, separated the peels, and grinded to make it powder form. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

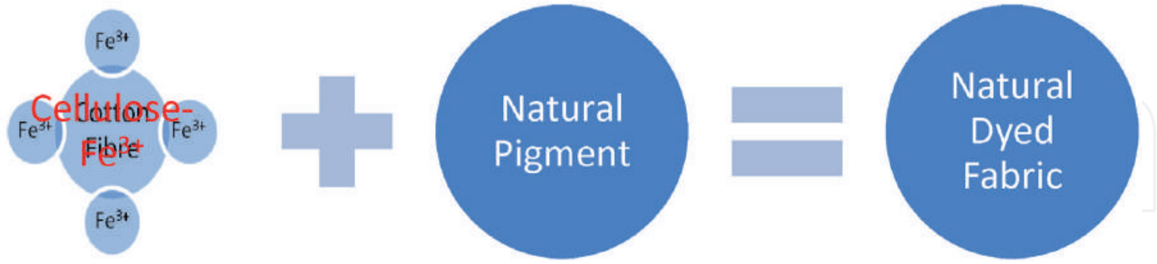
OH group can create a bond between dye and fiber and remaining curcumin may be responsible for coloration and dyed fabric may be slightly flammable due to having hydroxychavicol in the chemical constituent of betel nut.

RMS of Peach Leaves (Figure 15): Semi-dried Peach leaves were plucked from Peach tree garden. Two gram powder was mixed with 100 ml water, heated at boiling temperature for 40 min, and filtered properly. The dyes solution was used for cotton fabric dyeing in open bath medium for 30 min at 80°C.

Minimum color strength is found but fastness is lightly higher, increasing dye percentage may improve the color on the fiber surface. Remaining ferrous, copper, and zinc may be responsible for coloring the cotton fiber. As per chemical constituent, antimicrobial and anti-inflammatory can be found.

4. Method of natural fixing for mordant free dyeing

Maximum natural dyes sources from natural tree leaves, roots or fruits have ferrous ion, tannin, curcumin, catechin, OH group, and other known and unknown phytochemical constituent. Ferrous ion, tannin, curcumin, and catechin are making various color formations, and OH group is creating affinity with cellulosic fiber as well as the fastness properties may be increased if the dyes have natural pigment which may influence the capability of remaining dyes on the fiber surface after washing. Drying/curing with higher temperature may impact the color making duller or brighter. So specific dye-fiber system of natural fixing for mordant free dyeing is also possible but exact curing/drying temperature should be fixed for getting expected outcome of shading. So expected mechanism of mordant free natural coloration may be proposed as below although dyes and fiber substances may create changing of it.



5. Testing process

5.1 Color fastness to wash method

ISO 105-C06:2010, color fastness to water method: ISO 105-E01 and color fastness to light was tested by AATCC TM16 inQ-SUN XE-2 Xenon Test chamber.

5.2 Machine used for color parameters

Color parameters (L^*, a^*, b^*, Y) were measured by Hunter Lab Spectrophotometer, machine name: ColorFlex EZ, Model 45/0 LAV, Geometry

Figure 16.
Color Flex EZ, Spectrophotometer.

45°/0°, viewing area: large, visible spectrum (400–700 nm), testing condition: $D_{65/10°}$, room temperature of testing lab: 18°C (**Figure 16**).

6. Representation of dyed fabric and picture of dyes sources

All dyed shades were scanned with HUAWEI Smart Phone, Camera-13 MP, distance between fabric sample and camera position: 12–16 inch. So there is a possibility of having difference with actual shade. Picture of actual dye-fiber system was also scanned with HP Scanjet 4890 Photo Scanner. All sources of dyes pictures have been mentioned on the basis of grown trees in Asian Countries, specially in Bangladesh and scientific name was used accordingly. All the pictures of chemical structure mentioned here indicate the group of tree, not exactly indicating the chemical structure of specific parts of trees which one is extracted and dyed on cotton fabric.

6.1 Overcoming contradiction of natural dyeing

A huge number of researchers are working on natural dyes, still have a challenging for uneven dyeing, appropriate mordanting and dyes availabilities, shade matching where I can put a logic to protect our environment and human life as well.

6.2 Uneven dyeing

Automation in extraction and dyeing process.

6.3 Appropriate mordanting and dyes availability

Selection of specific dyes for commercial production and/or wastage can be used as dyes sources.

6.4 Shade matching

Customers are not always asking for shade matching when they are shopping although some natural dyes have attractive shading and repeated shading also possible.

7. Conclusion

Mordanting free coloration was practiced establishing the feasibility of environment friendly natural coloration which is the ongoing research of author and a part of his research has been included here. Shade variation of natural dyed fabric may influence the uncontrollable factors for the collection of natural dyes sources on the basis of season to season, region to region and a same source but different parts of same dye sample source. So it is very tough to make a specific declaration of shading behaviors by the textile technologist but specific expertise in natural dyes extraction and natural dyeing may minimize the problem whereas expertise in dyeing with synthetic dyes and dyeing with natural dyes are not same for bulk production.

Following directions for the real shade development mentioned above:

1. Fabric should be well scoured and bleached.

2. Dyes materials should be clean, dried and powdered properly, improper drying and inappropriate powder formation may be responsible for unexpected shading and uneven dyeing.

3. Extraction time, dyes percentage may be maximized or minimized if the actual color is not observed.

4. Improper filtration of dyes may create uneven dyeing.

5. Dyes should not be powdered in wet or sticky condition which may effect the changing of light fastness and color shading.

6. Insect affected dyes source may create the changing of shade.

7. Synthetic mordanting and chemical medium dyes extraction may improve the shade but the concern of chemical and cost.

8. For open bath dyeing, liquor ratio should be higher, and stirred continuously to reduce color mark on the dyed fabric.

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Chapter

Betanin: A Red-Violet Pigment - Chemistry and Applications

Deepak Devadiga and T.N. Ahipa

Abstract

Nowadays, the demand for eco-friendly/nontoxic natural colorants is growing as an essential alternative to potentially harmful synthetic dyes. Betanin is the chief red pigment of beetroot, and it is the only betalain approved for use in food and pharmaceutical products as a natural red colorant. This chapter is mainly dealing with the betanin pigment, and also the chapter is subdivided into six sections covering the chemistry of betanin, extraction of color using various novel techniques (like microwave- and ultrasonic-assisted extraction) from raw plant material, biosynthesis of betanin followed by chemical synthesis of betanidin, and also the effect of pH, temperature, and light on the stability of betanin followed by its applications.

Keywords: beetroots, betanin, red pigment, extraction, chemical synthesis, stability

1. Introduction

Vegetable beetroot (*Beta vulgaris* L.) has the notable scientific interest, because it is a rich source of nitrate (NO_3^-), a compound with advantageous cardiovascular health effects through the endogen production of nitric oxide (NO) [1, 2]. There are two classes of pigment in plants, i.e., betalains and anthocyanins. Beetroots are the chief sources of betalains which is a water-soluble nitrogen pigment with heterocyclic ring, which can be further subdivided into two classes depending on chemical structure: betaxanthins comprising indicaxanthin; vulgaxanthin I and II, accountable for orange-yellow coloring; and betacyanins, such as betanin, isobetanin, neobetanin, and prebetanin, accountable for red-violet coloring [3, 4]. The most abundant betacyanin is betanin (betanidin 5-O-β-D-glucoside) and is the only pigment which is an approved natural colorant for the use in food products prescribed by the Food and Drug Administration (FDA) in the United States [5, 6]. **Figure 1** represents the chemical structure of betanin.

According to experimental studies, raw beetroot generally contains water (87.1%), carbohydrate (7.6%), protein (1.7%), fat (0.1%), and betanin (0.03–0.06%) [7]. In addition to natural food colorant property, betalain also exhibits antimicrobial, antiviral, and antioxidant activities [8]. Moreover, beetroot dye has nutrient value along with nontoxic nature; therefore, it even finds application in dyeing industry where the health aspect is a foremost criterion. Also, this natural dye extracted from beetroot is eco-friendly in nature and does not cause any environmental problems in contrast to the commercially available synthetic dyes [7].

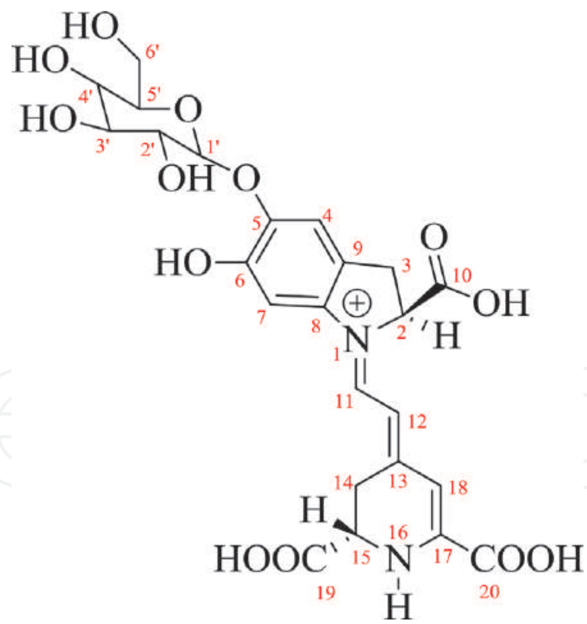

Figure 1.
Chemical structure of betanin.

2. Chemistry of betanin

The first report on the crystallization of betanin was communicated by two independent groups Schmidt and Schonleben [9] and Wyler and Dreiding [10]. These two groups employed an electrophoretic strategy for betanin purification. Wyler and Dreiding [11] recognized three products which were formed by the alkaline degradation of betanidin (**Figure 2**); these were 4-methylpyridine-2,6-dicarboxylic acid, formic acid, and S-cyclodopa(5,6-dihydroxy-2,3-dihydroindole 2-carboxylic acid). When these three products are placed in correct relationship with one another, they form the betanidin's carbon skeleton and also the configuration of the second carbon [12].

2.1 Position of the β-D-glucosyl group

To identify the position of β-D-glucosyl group, betanin was reacted with diazomethane in the presence of air yielded tetramethyl derivative of neobetanidin which was then converted to 6-methoxy-neobetanin-trimethylester by hydrolysis and acetylation [13, 14]. In the alkali condition, neobetanin cleaved to yield 5-hydroxy-6-methoxyindole 2-carboxylic acid. Its methyl ester was prepared from

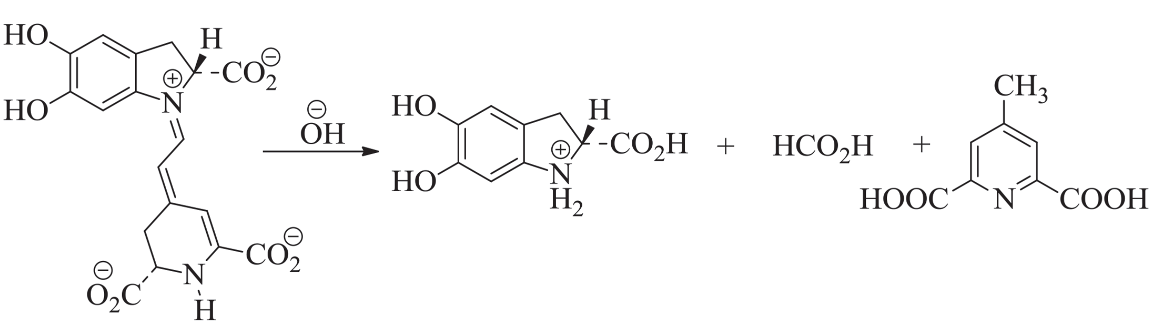

Figure 2.
Alkaline degradation of betanidin.

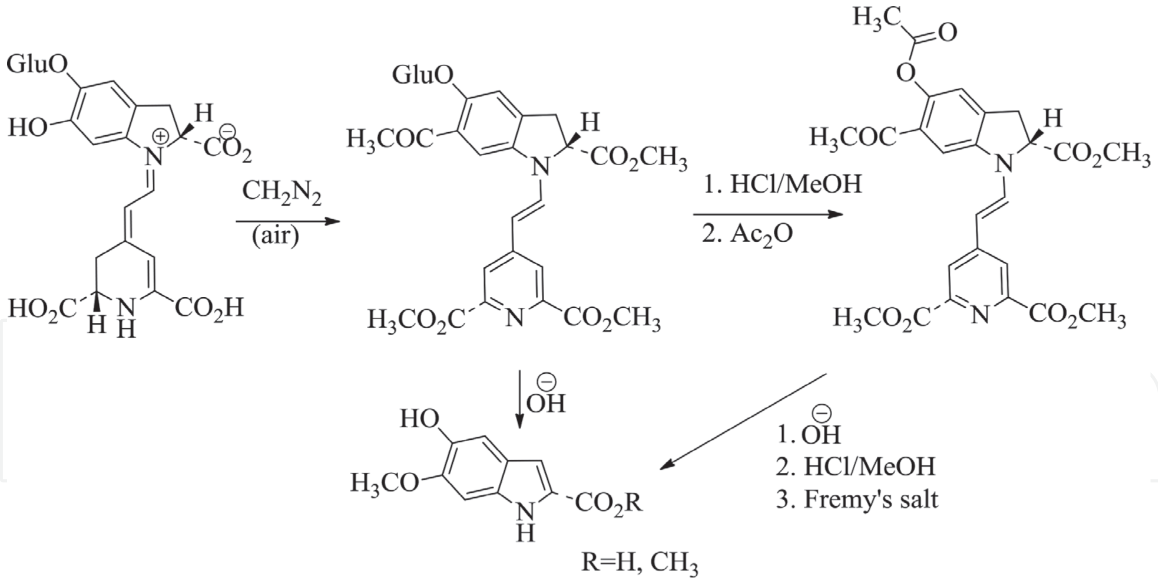

Figure 3.
Reactions that revealed the glucosyl residue position in betanin.

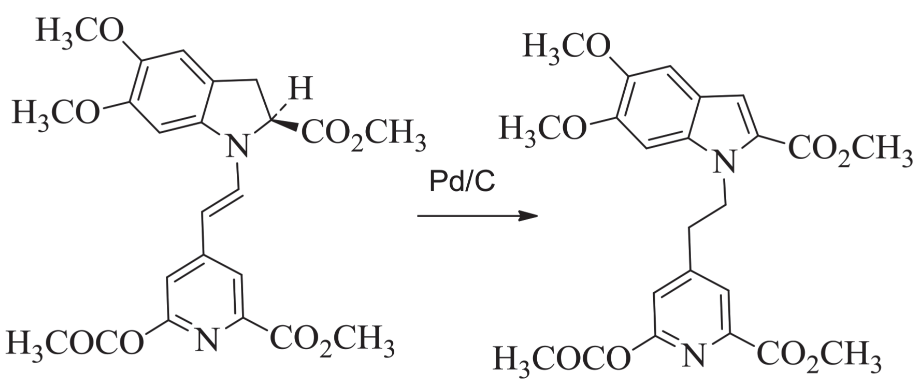

Figure 4.
Reaction which confirmed the presence of vinylene connecting group.

acetylated molecule by degrading in basic condition, and further it was esterified and oxidized. Further, analysis of all these reactions together revealed the glucosyl residue position (**Figure 3**). In addition, NMR of betanin in trifluoroacetic acid and hydrolytic studies of β-glucosidase [15] revealed that betanin was an O-β-D-glucopyranoside.

The yellow-colored compound, i.e., 5,6-di-O-methylneobetanidin trimethyl ester, converted into colorless compound, i.e., 5,6-di-O-methyl-2,3-dehydro-11,12-dihydro-betanidin trimethyl ester, by the palladium-catalyzed disproportionation reaction which confirmed the existence of vinylene connecting group (**Figure 4**) in the derivatives of neobetanidin [12].

Further, betanin can also be interconverted into betanidin. To prepare betanidin, betanin is initially reacted with the excess of L-proline in the presence of dilute ammonia which results in the formation of indicaxanthin which can later be converted into betanidin by reacting it with excess S-cyclodopa [12].

3. Extraction techniques

The drawbacks of conventional approaches, such as time-consuming methods, safety risks with some hazardous solvent systems, contaminated product, and comparatively less yields, have increased the demand for the novel processing methods

Variants	Solvents
V_1	Distilled water
V_2	1% citric acid
V_3	0.5% citric acid
V_4	0.2% citric acid
V_5	0.1% ascorbic acid
V_6	50% ethanol
V_7	20% ethanol
V_8	0.5% citric acid + 0.1% ascorbic acid
V_9	0.2% citric acid + 0.1% ascorbic acid
V_{10}	20% ethanol + 1% citric acid
V_{11}	20% ethanol + 0.5% citric acid

Table 1.
Betanin extraction from beetroot using different solvent systems.

[16, 17]. To improve the betalain extraction, some of the pretreatment methods have been proposed, such as pulsed electric fields [18], gamma irradiation [8], and low-direct current electric fields [19]. But, such methods are quite expensive when compared to the solvent extraction techniques. Further, certain nonthermal techniques, like ultrasound (sonication) processing, and microwave-assisted extractions are also significantly productive in order to enhance the extraction yields of bioactive molecules with minimum degradation [17].

Neagu and Barbu [20] studied the betanin extraction from beetroot using different solvent systems by solid-liquid extraction technique (liquid/solid ratio is 5:1). **Table 1** presents the different extraction solvents used in this study. Results revealed that the highest betanin content of about 20 mg/g of beetroot was obtained with the use of weak acid solution (i.e., V_8, using 0.5% citric acid +0.1% ascorbic acid). Also, they extracted the considerable amount of betanins by using ascorbic acid added solutions. Thus, it is clear that the acidic medium influences positively during the low-temperature extraction process.

In case of ultrasound (sonication) processing, ultrasonic-assisted extraction approach requires the use of ultrasound (with 20–2000 kHz frequencies range) which generally increases the cell wall permeability and generates the cavitations. Because of cavitations, cell membrane breaks down, and internal materials (color and oil) come out [21].

On the other hand, microwave-assisted extractions are also contributed significantly to speed up the sample digestion and extraction of bioactive molecules from matrices. Here, the microwave energy has been utilized and employed in this extraction process. Moreover, this microwave energy induces molecular motion by the rotations of dipoles and migration of ions without varying the structure of the molecules provided the temperature of the system is not too high [21].

4. Biosynthesis of betanin

The biosynthetic pathways for the betanin molecule are depicted in **Figure 5** [22]. Three enzymes such as 4,5-DOPA (dihydroxyphenylalanine)-extradiol-dioxygenase, tyrosinase, and betanidin-glucosyltransferase were involved in the biosynthesis of betalains in the cytoplasm [23]. From arogenic acid, the amino acid L-tyrosine was

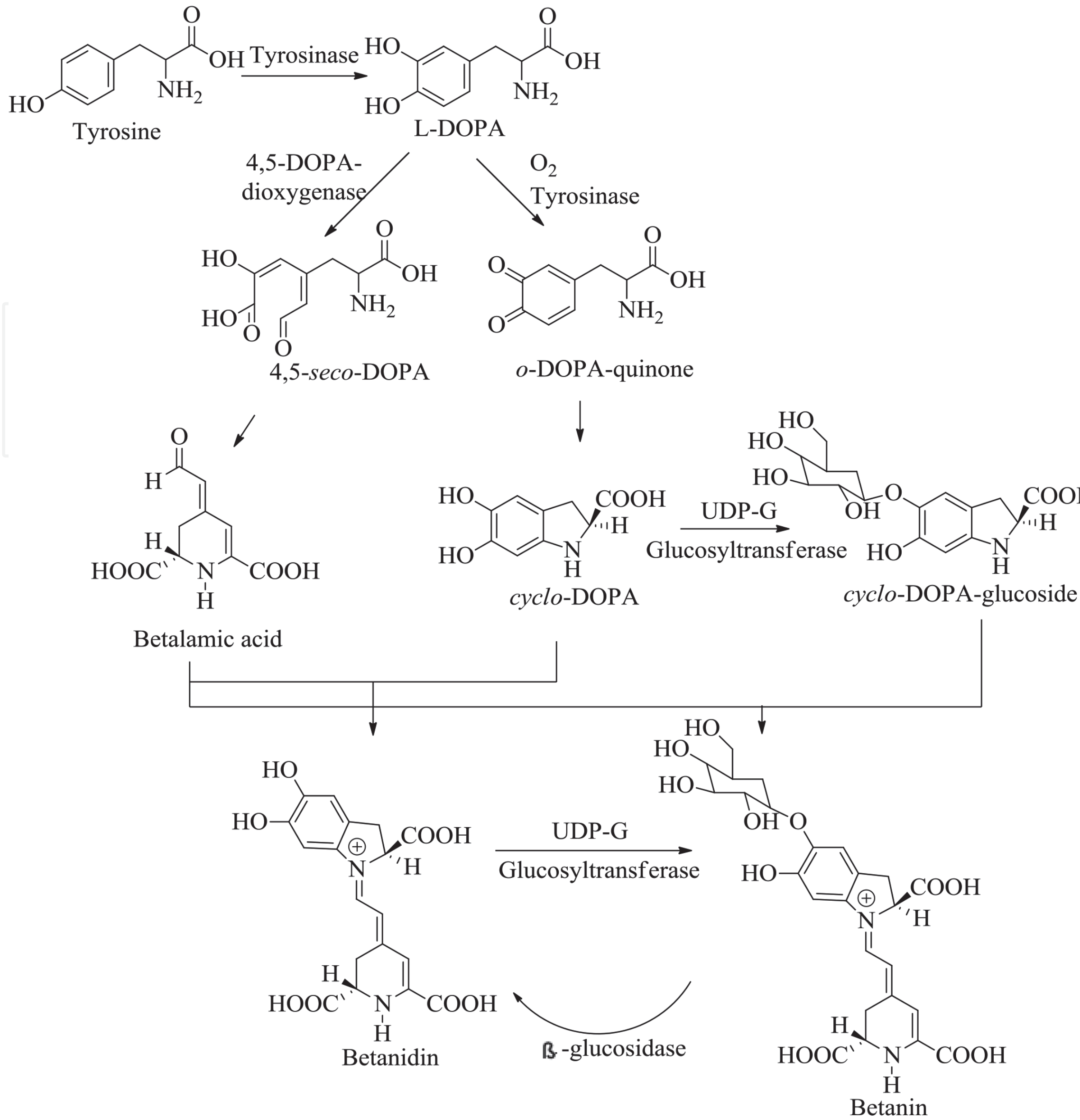

Figure 5.
Biosynthesis of betanin.

formed enzymatically over the shikimate pathway [24], which is the starting material for the biosynthesis of L-DOPA [25]. During this conversion, tyrosinase enzyme helped to convert tyrosine to DOPA through hydroxylation. Further, between carbons 4 and 5 of the L-DOPA cyclic ring was opened by 4,5-DOPA-extradiol dioxygenase enzyme to give 4,5-*seco*-DOPA [26–28] which is then converted into betalamic acid by intramolecular condensation between aldehyde and amine groups [25]. Further, L-DOPA is transformed into *o*-DOPAquinone in the presence of molecular oxygen [29]. Furthermore, it was spontaneously cyclized due to the nucleophilic attack of amino group on the ring system to yield *cyclo*-DOPA [23]. However, *cyclo*-DOPA can also be obtained from the cyclization of L-DOPA in the presence of cytochrome P450 (CYP76AD1) [30].

Finally, betanidin was formed by the formation of imine bond between *cyclo*-DOPA and betalamic acid which is then converted to betanin using betanidin-5-O-glucosyltransferase enzyme by connecting glucose unit of uridine diphosphate-glucose (UDP-G) to the hydroxyl group in position 5 [31]. But this reaction can be reversed in the presence of β-glucosidase [32]. Additionally, it is also concluded that enzyme *cyclo*-DOPA-5-O-glucosyltransferase catalyzes the transport of glucose molecule on *cyclo*-DOPA, by which the *cyclo*-DOPA-glucoside condense with betalamic acid to yield the betanin [33].

5. Chemical synthesis of betanidin

The chemical synthesis of betanidin is illustrated in **Figure 6** [34]. For the synthesis of betanidin, 4-hydroxypyridine-2,6-dicarboxylic acid is used as the starting material which upon hydrogenation and followed by esterification yields all products in *cis* form. Further, Pfitzner-Moffatt oxidation reaction converted the secondary hydroxyl group to a ketone. In the next step, it is converted to semicarbazide using Horner-Wittig reagent. Then, the obtained semicarbazide is further hydrolyzed to give unsaturated ketone, which is converted to betalamic acid by Pfitzner-Moffatt oxidation. Reacting betalamic acid with L-cyclo-DOPA methyl ester yielded betanidin trimethyl ester which is then converted to betanidin through acid hydrolysis by using concentrated hydrochloric acid.

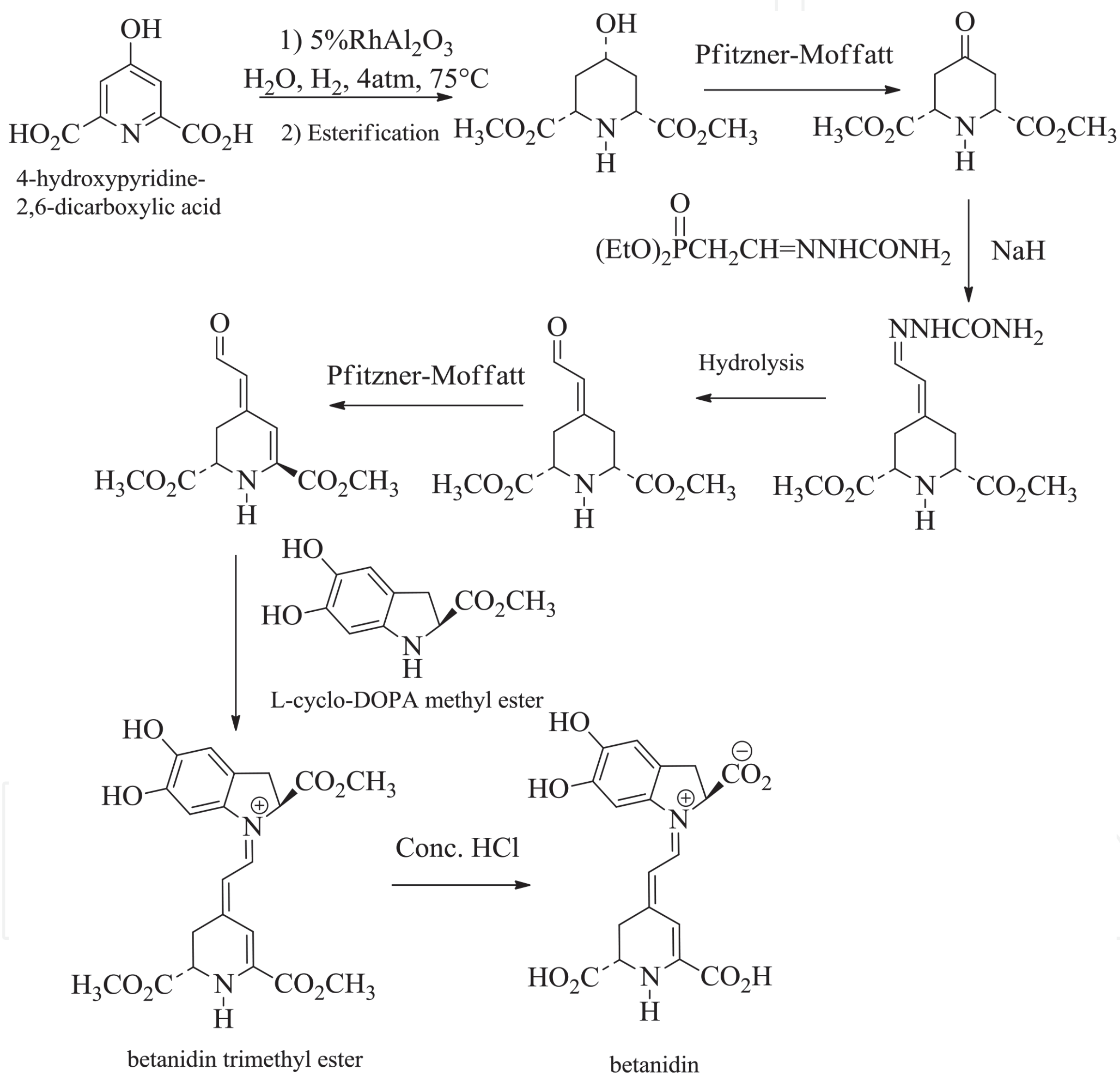

Figure 6.
Chemical synthesis of betanidin.

6. Characterization of betanin

6.1 UV-vis absorption spectra of beetroot extracts

The UV-vis spectra of beetroot extracts in different solvents (ethanol, methanol, and water) are depicted in **Figure 7** [35]. Strong absorption band was observed at around 530 nm in the visible range for the red beet juice which was attributed to the betanin pigment. As the solvent changed from ethanol (532 nm) to water (542 nm) and to methanol (544 nm), absorption maximum shifted towards longer wavelengths. These results showed that methanolic and aqueous extracts mainly contain

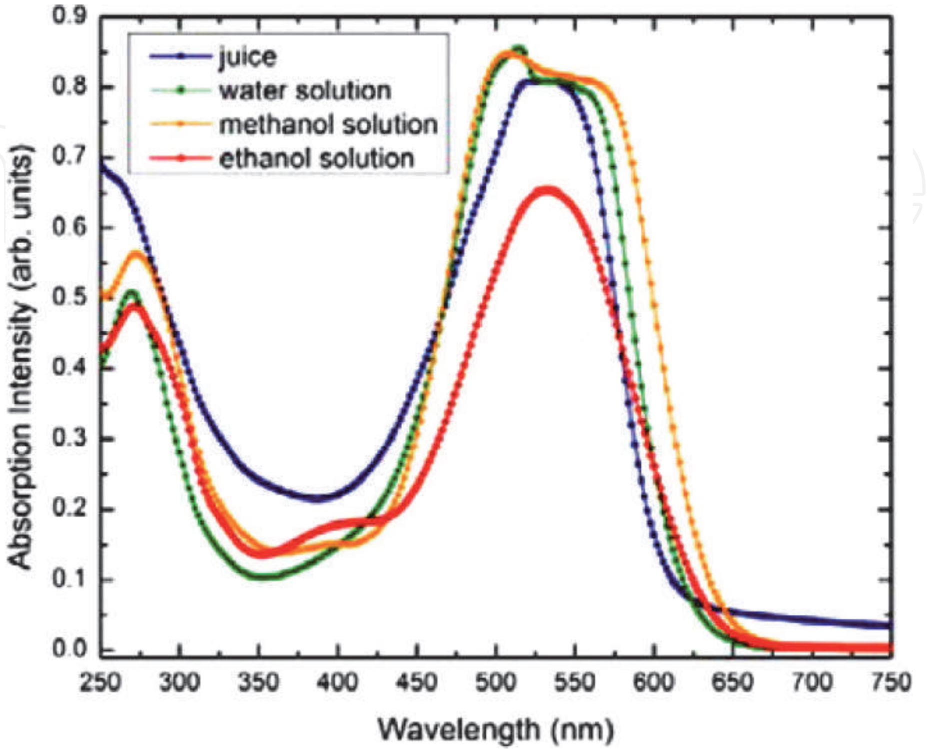

Figure 7.
The UV-vis spectra of beetroot extracts in different solvents (ethanol, methanol, and water).

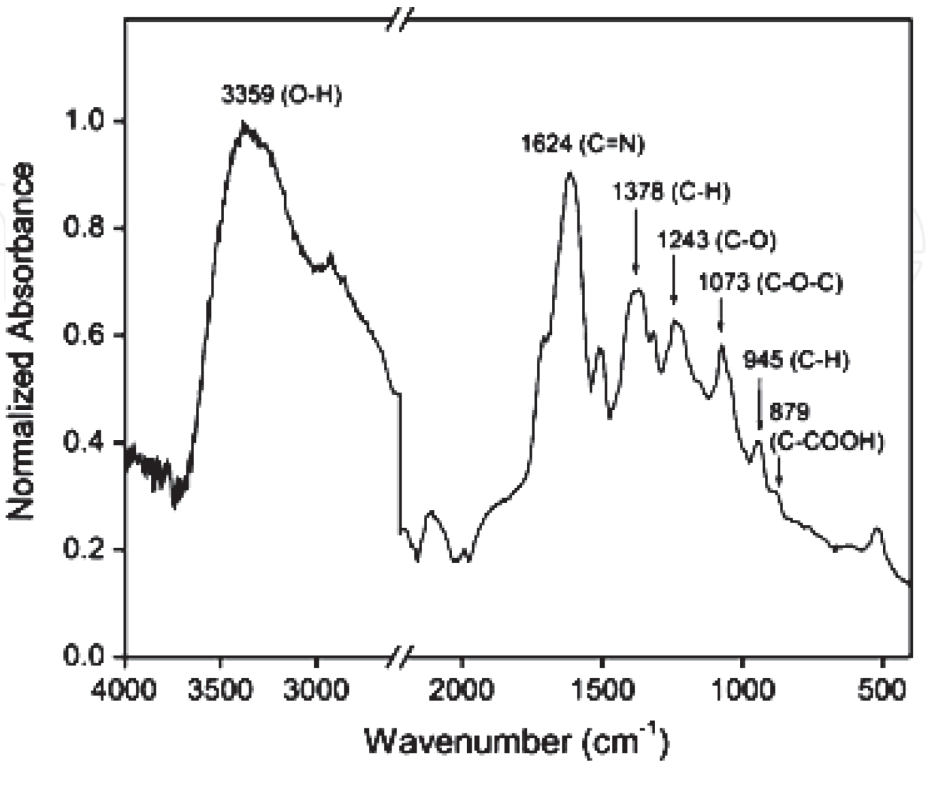

Figure 8.
FT-IR spectrum of betanin (scale range, 400–4000 cm^{-1}).

betanidin, whereas ethanolic extract mainly contains betanin. The intensity of absorption maxima for the aqueous and methanolic extracts is approximately equal, higher than the ethanolic extract.

Further, partially overlapped two absorption bands were observed in case of aqueous and methanolic extracts. For aqueous extract the second band has an absorption maximum at 515 nm and for aqueous extract at 509 nm. These bands are only observed in high concentrated extract, and it vanishes as the solution diluted. Hence, it can be attributed to the formation of supramolecular structure in the concentrated solutions.

6.2 FT-IR data of betanin

Different characteristic absorption bands corresponding to the functional groups of betanins were observed, and its FT-IR spectrum is shown in **Figure 8** [36]. The absorption band around 3359 cm^{-1} was ascribed to the —OH bond stretching vibration [37]; on the other hand, the absorption band around 1624 cm^{-1} was ascribed to the C=N bond stretching vibration [38, 39]. The next absorption band located at 1378 cm^{-1} was ascribed to the C—H bond extension stretching vibration, while the absorption band at 1243 cm^{-1} was ascribed to the C—O bond of the carboxylic acid stretching vibration [38, 39]. Another absorption band centered at 1073 cm^{-1} was ascribed to the C—O—C linked symmetric stretching vibration [40], the absorption band at 945 cm^{-1} was ascribed to the C—H bond deformation, and lastly the absorption band at 879 cm^{-1} was ascribed to the C—COOH bond stretching vibrations [41].

6.3 ^{1}H, ^{13}C, and LC-^{1}H NMR data of betanin

The ^{1}H and ^{13}C NMR data of betanin was obtained by dissolving it in D_2O, and its LC-^{1}H NMR data was also obtained by dissolving it in acetonitrile (MeCN)/D_2O/0.05% trifluoroacetic acid (TFA) using 500 MHz frequency at 25°C. **Figure 9** represents the LC-^{1}H NMR spectrum of betanin and followed by **Table 2** which represents the ^{1}H, ^{13}C, and LC-^{1}H NMR data of betanin [42].

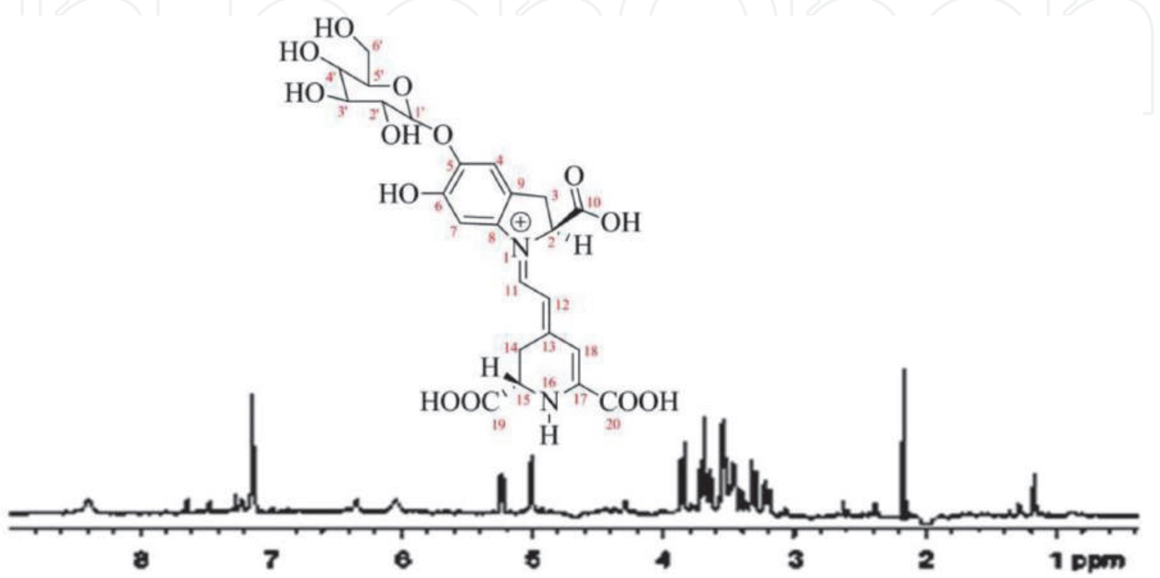

Figure 9.
LC-^{1}H NMR spectrum of betanin.

Atom numbering	^{1}H NMR δ-[ppm], mult, J [Hz]	^{13}C NMR δ-[ppm], mult, J [Hz]	LC-^{1}H NMR δ-[ppm], mult, J [Hz]
2	4.92, *dd*, 3.1, 10.3	65.0	5.23, *dd*, 2.0, 10.2
3a/b	3.53, *dd*, 11.5, 16.9 3.10, *dd*, 4.3, 16.8	32.7	3.66, *dd*, 10.4, 16.7 3.31, *dd*, 2.0, 16.7
4	7.06, *s*	113.9	7.13, *s*
5	—	144.0	—
6	—	146.1	—
7	6.98, *bs*	100.0	7.12, *bs*
8	—	137.4	—
9	—	124.1	—
10	—	175.8	—
11	8.19, *bs* (*d*, 12.6)[b]	144.4	8.38, *bd*, ≈11
12	5.84, *bs* (*d*, 12.6)[b]	106.9	6.04, *bd*, ≈11
13	—	—[a]	—
14a/b	3.20, *bm* 3.12, *bm*	26.5	3.21, *dd*, 7.4, 17.2 3.41–3.59 (overlap)
15	4.40, *bt*, 7.1	53.1	Overlapped by D_2O
17	—	—[a]	—
18	6.22, *bs*	—[a]	6.34, *bs*
19	—	—[a]	—
20	—	—[a]	—
1′	4.98, *d*, 7.4	101.4	5.00, *d*, 6.9
2′	3.55 (overlap)	75.7	3.41–3.59 (overlap)
3′	3.55 (overlap)	73.9	3.41–3.59 (overlap)
4′	3.41 (overlap)	69.3	3.41–3.59 (overlap)
5′	3.52 (overlap)	76.2	3.41–3.59 (overlap)
6′a/b	3.85, *dd*, 1.6, 12.3 3.70, *dd*, 5.3, 12.3	60.6	3.86, *dd*, 1.4, 12.6 3.70, *dd*, 5.5, 12.6

[a]*Chemical shifts were not observable.*
[b]*After acidification (TFA) to pH 2, s = singlet, d = doublet, dd = doublet of doublet, bs = broad singlet, bd = broad doublet, bt = broad triplet, bm = broad multiplet.*

Table 2.
^{1}H, ^{13}C, and LC-^{1}H NMR data of betanin.

6.4 Mass spectrum of betanin

The mass spectrometry of betanin in the positive ionization mode exhibited a molecular ion (*m/z* 551, $[M+H]^+$, 100%). **Figure 10** represents the obtained mass spectrum of betanin molecule [43].

6.5 Thermogravimetric (TG) analysis

The dynamic thermogravimetric (TG) analysis was conducted on fresh betanin and dried betanin (**Figure 11a** and **b**) [44]. Since fresh betanin contains water, immediate mass loss is noted in the temperature range of 40–100°C. Further, the degradation temperature of betanin dye is noted at the temperature of about 204°C.

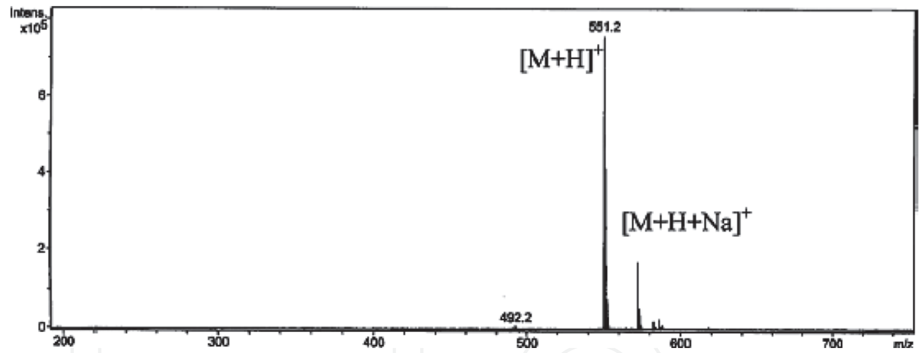

Figure 10.
Mass spectrum of betanin.

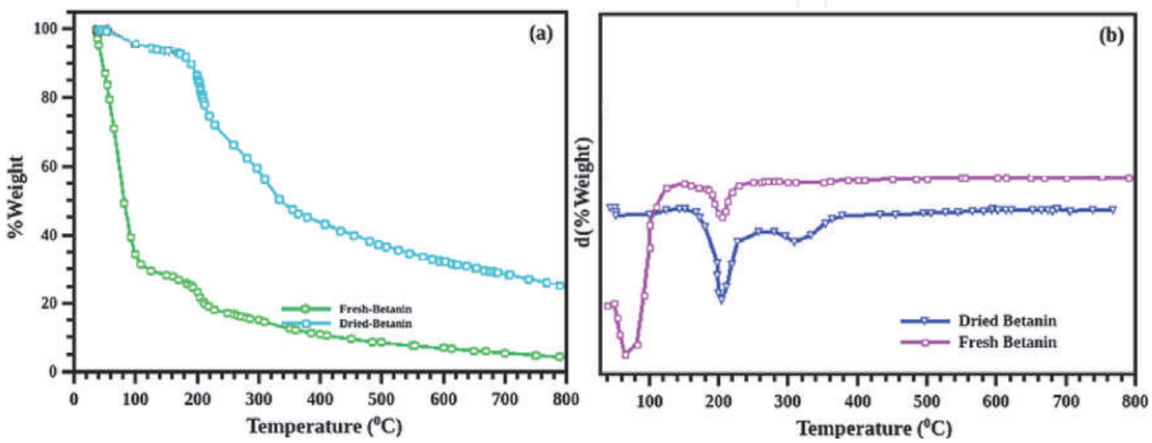

Figure 11.
(a) TG curves and (b) derivative TG curves of fresh and dried betanin.

Furthermore, the dried sample has also exhibited similar behavior except the mass loss at the beginning (in the temperature range of 40–100°C).

7. Factors effecting the stability of betanin

- *pH*: in the buffers of pH 2–9, betanin was stored at 4°C for 7 days and measured the visible spectra at both starting and end of this time span. No shifts in the absorption maxima were noted in between pH 4 and pH 7. A shift of about 2 nm towards a shorter wavelength with a decreased absorbance intensity was observed in case of the buffer which has pH less than 4. In the 575–650 nm region, the spectrum has slightly increased absorbance, and the solution color changed to red-violet from red. While in case of the solutions which has pH value of above 7, i.e., at pH 9, the absorption maximum moved to a longer wavelength region (544 nm) by decreasing the intensity. In the 575–650 nm and 400–450 nm wavelength regions, the absorbance increased to a considerable extent, and the solution color changed to violet from red. These results showed that between pH 3 and pH 7, storage had no effect on betanin solutions, and above and below of these pH values causes the considerable losses of betanin. Visible spectra of betanin compound at pH 2, 5, and 9 are illustrated in **Figure 12** [12, 45].

- *Temperature*: on heating the red color of betanin solutions starts to diminish, and finally it turns to brown color. The color loss was followed by the betanin assay, and the rate indicates that it follows first-order kinetics. The graph indicates that at 100°C, the degradation rate at pH 5 is still less than it is at pH 3 and pH 7. Further, the betanin compound is more stable between pH 4 and 5. However, betanin in beet juice is far more stable at pH 5, which reveals a protective effect by the constituents of juice. The rates of degradation for

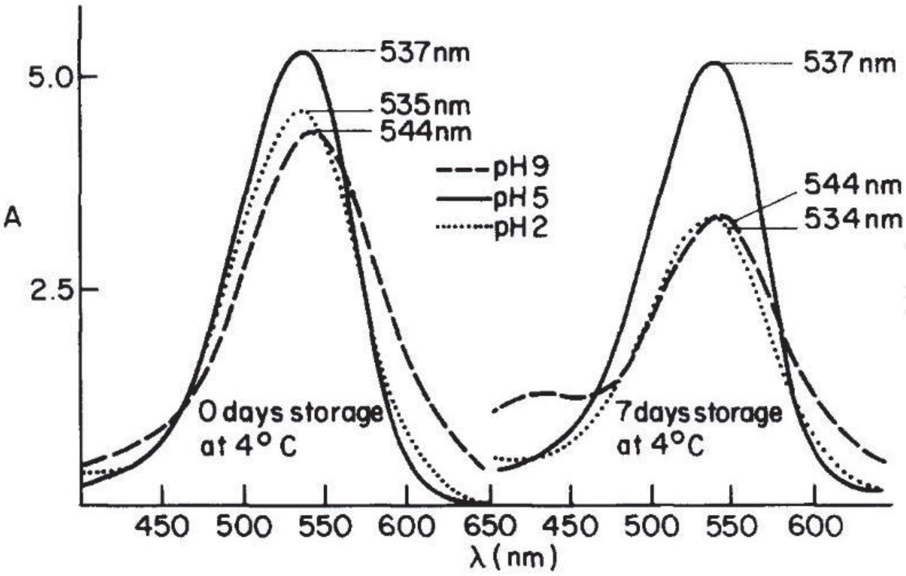

Figure 12.
Visible spectra of betanin compound at pH 2, 5, and 9.

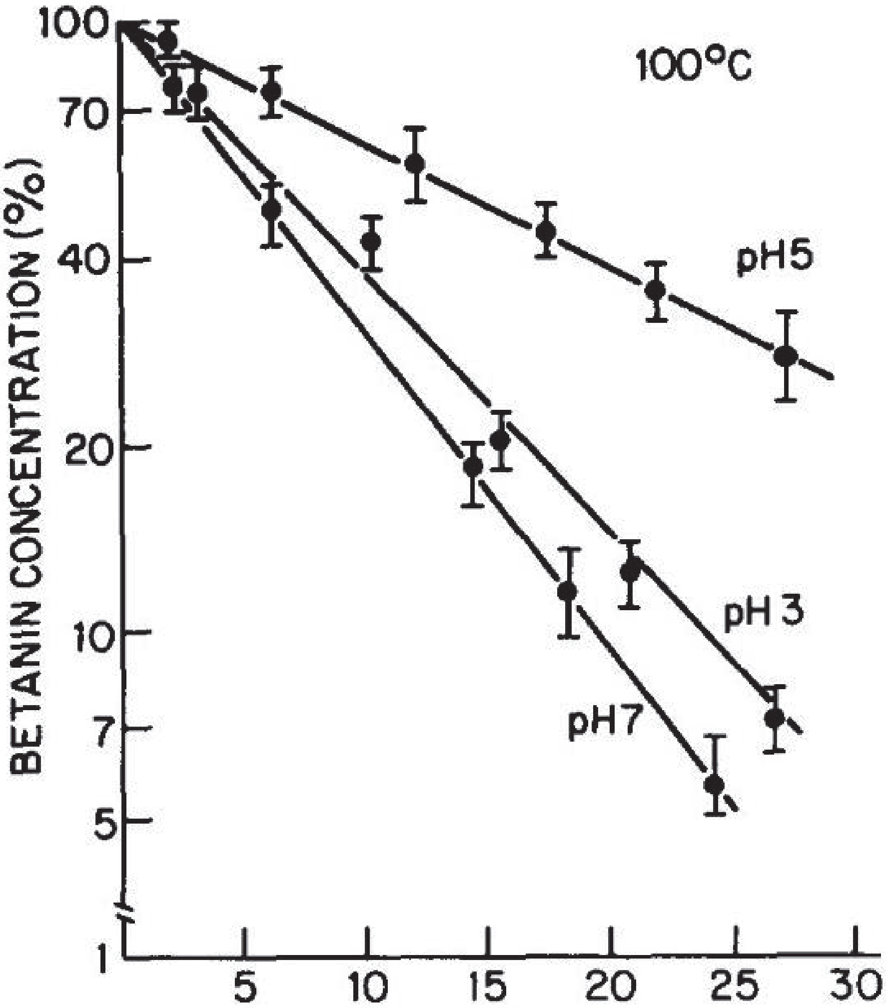

Figure 13.
Rates of degradation for betanin molecule in a system at 100°C at pH 3, 5, and 7.

betanin molecule in a system at 100°C at pH 3, 5, and 7 are depicted in **Figure 13** [12, 45].

- *Light*: at 15°C and pH 7, the presence of light increased the rate of degradation by 15.6% and air (rather than nitrogen) by 14.6%. Both light and air together increased the rate by 28.6% [12, 42].

8. Applicability of betanin

8.1 Dyeing acrylic fabric

Pure betanin dye can compete with synthetic dyes in color depth shade properties and in color fastness properties. Guesmi et al. [43] studied the dyeing of betanin on modified acrylic fabrics and evaluated the effect of dye bath pH, salt concentration, dyeing time, and temperature on dyeing. In a dye bath having sodium chloride (0–15 g/L) and a dye of 30 mg/L concentration with the 40:1 liquor ratio, modified acrylic fabric was dyed using conventional heating.

8.1.1 Effect of pH on dyeing

Over the pH range 1–5, increase in pH increases the adsorption of betanin onto acrylic fabric. Color strength decreases as the pH increased above 5. Generally, amino functional groups of acrylic fibers get protonated as the pH value decreases. Thus, ion-ion forces induced with ionized carboxyl groups in betanin. Betanin may exist in cationized or on monoanion form in a strongly acidic environment which results in the lower depth of dyeing at pH less than 4, and also it is due to the betanin stability loss at low pH [43].

At pH 5 maximal color strength was observed, whereas at pH 4, little decrease in color strength was observed; this is attributed to the increased carboxyl groups in this range and to the high thermal stability of betanin molecule. The number of protonated terminal amino functional groups of fabric decreases at pH > 5, which causes the decreased ionic interaction between the carboxylate anion of the dye and acrylic fibers, thus lowering its dye ability. The structures of betanin molecule as a pH varied are depicted in **Figure 14** [43].

8.1.2 Effect of salt addition

Color strength decreases as the salt concentration increases, hence dyeing without salt addition is the best condition [43].

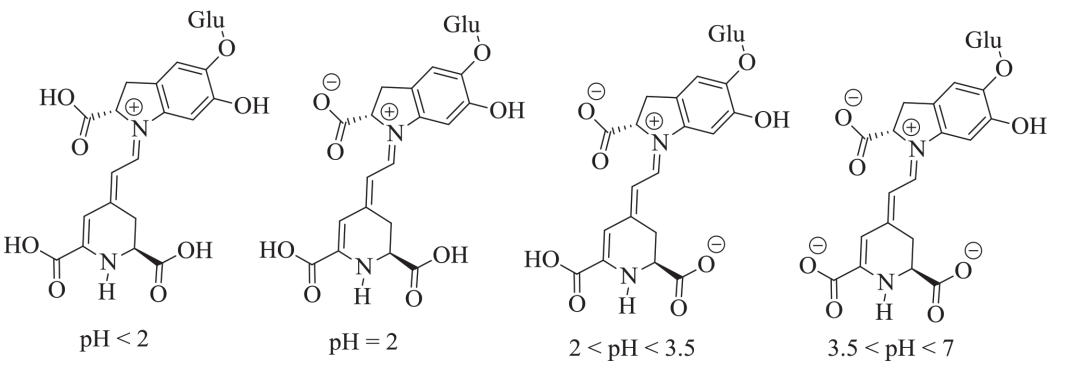

Figure 14.
Structures of betanin molecule as a pH varied [46].

8.1.3 Effect of temperature on dyeing

As the temperature of dyeing increases, the color strength increases up to 50°C, and further by increasing the dyeing temperature, the color strength decreased slowly which is attributed to the decrease in stability of dye at higher temperatures [43].

8.1.4 Effect of time span on dyeing

Color strength increases as the dyeing time increases up to 30 min; from 30 to 45 min, there is no change in color strength, and then it started to decrease as the time increases [43].

8.1.5 Color fastness

The fastness properties of betanin-dyed acrylic fabrics are shown in **Table 3**. The fastness properties of the dyed samples were examined according to ISO standard methods, the specific tests conducted for color fastness to rubbing is as per ISO 105-X12:1987, the color fastness to water is as per ISO 105-E01:1989, the color fastness to washing is as per ISO 105-C02:1989, and the color fastness to light is as per ISO 105-B02:1988 (carbon arc) [43]. It was noted that rubbing, washing, and water fastness of unmordanted acrylic fabrics exhibited significantly good property. But, the light fastness of unmordanted acrylic fabrics was found to be bad. However, light fastness was found to increase from rating 3* to 4* in premordanted fabrics using manganese sulfate and ferrous sulfate, and light fastness was increased from rating 3* to 5* by using cobalt sulfate. Nevertheless, the other mordants did not affect the light fastness of premordanted fabrics. It was found that for the improvements of color strength and light fastness, cobalt sulfate was established as the best mordant [43].

Similarly, Guesmi et al. [46] in the year 2013 studied the dyeing of wool fabric using betanin and chlorophyll-a as biomordant. In a dye bath having sodium chloride (0–5 g/L) and a dye with 40:1 liquor ratio, wool fabric was dyed using conventional heating. Results revealed that the increase in the concentration of biomordant increases the color strength values. They also investigated the effect of variables on the color of dyed fibers and noted that from pH 3.5 to pH 4.5, the color strength considerably increases, the color strength was found to be better without salt than with salt, and the color strength of dyed wool increases as the increase in temperature was up to 40°C and starts to decrease slowly till 50°C. Further increase in temperature, the color strength decreases in pronounced manner. According to the authors, color strength increases with the time span (up to 45 min) of dyeing, and

	Fastness to dry rubbing	**Fastness to wet rubbing**	**Fastness to washing**	**Fastness to water**	**Fastness to light**
None	5*	5*	4*–5*	4*–5*	3*
Manganese sulfate	5*	5*	4*	4*	4*
Ferrous sulfate	4*	4*	3*	3*	3*
Zinc sulfate	5*	5*	4*–5*	4*–5*	4*
Aluminum potassium sulfate	4*	4*	3*	3*	3*
Cobalt sulfate	5*	5*	4*–5*	4*–5*	5*

Table 3.
The fastness properties of betanin-dyed acrylic fabrics represented with the rating scale using star (*).

then it starts to decrease because betanin losses thermal stability, and also it starts to escape from the fiber. Dye exhaustion was examined in both ultrasonic and conventional dyeing approaches. It was exhibited that in a shorter time span of dyeing, sonication increases the dye exhaustion from rating of 30% to rating of 60%. The fastness properties of dyed wool were studied against wet rubbing, light, washing, and dry rubbing. Unmordanted and mordanted samples have good fastness properties too.

8.2 Medicinal application

Antioxidant activity of betanin in biological lipid domain has been exhibited in human macromolecules, like lipoproteins of low density, whole cells, and membranes [2]. Moreover, betanin has attracted researchers because of its anti-inflammatory activities and hepatic safety activities in whole human cells [47]. In cultured endothelia cells, this molecule regulates the redox-mediated signal transduction pathways which is required in responses during inflammation, and betanin also showed antiproliferative effects on tumor cell lines in human [48–50]. In both tumoral and healthy hepatic cell lines in the human body, betanin translocates the antioxidant response element (erythroid 2-related factor 2 (Nrf2)) from the place of cytosol to the place of nuclear domain, which regulates m-RNA and protein levels of antioxidant/detoxifying enzymes, which includes heme oxygenase-1, NAD(P)H quinone dehydrogenase-1, and glutathione S-transferase and, in these cells, bears anticarcinogenic and hepatoprotective effects [51]. Also, it exhibits antidiabetes properties by controlling the activities of liver markers enzymes [52–54].

8.3 Betanin as food colorant

Betanin is the oldest and most abundant red food colorant which has been established in the market, which is noted as E-162 in the European Union and in the United Sates; it is known as 73.40 in the twenty-first chapter of the Code of Federal Regulations (CFR) section of the Food and Drug Earth Administration [2, 5, 6].

Betanins are most commonly used for coloring of ice cream and powdered soft drink beverages. Additionally, betanin is used in some of the sugar confectionery, like sugar coatings, fruit or ice cream fillings, fondants, and sugar strands. At the final part of the processing, it can be added while preparing hot processed candies. Also, it is used in soups as well as bacon and tomato.

9. Summary

In this chapter, the first and second section covered the chemistry of betanin which contains reactions that revealed the glucosyl residue position and presence of vinylene connecting group in betanin. Further, the third section narrated the extraction techniques which mainly included the microwave- and ultrasonic-assisted extraction method. Furthermore, the fourth and fifth sections elucidated the biosynthesis of betanin molecule and chemical synthesis of betanidin molecule, respectively. In addition, different characterization techniques were also explicated in the sixth section which includes UV-Vis, FT-IR, ^{1}H NMR, ^{13}C NMR, LC-^{1}H NMR, mass spectrum, and thermogravimetric analysis of betanin. Also, the factors effecting the stability of betanin were explained in the seventh section which covers the effect of pH, temperature, and light on the stability of betanin. Lastly, the applicability of betanin was taken into account in the eighth section which comprised of dyeing of acrylic fabric, dyeing of wool fabric, and medicinal and food colorant applications of betanin.

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Chapter

Printing of Textiles Using Natural Dyes: A Global Sustainable Approach

M. Kavyashree

Abstract

Globalization has completely changed the fashion industry and its approach toward sustainability. Fast changing trends are majorly focused on synthetic dyes and fabrics. And these products have failed building the bridge between sustainability and environment-friendly designs. Therefore, this chapter is aimed at the approach toward design development and development of sustainable eco-friendly approach for screen printing by using natural dyes on fabrics like silk and cotton. The chapter is based on sustainable design approach which gives much provision for using natural dyes for everyday change in fashion. The chapter also focuses on color fastness properties of the used dyes. These results were evaluated by carrying out color fastness tests for light, wash, rubbing, and perspiration using standard testing methods. Design methodology used in the study also has the potential for skill development programs for entrepreneurs and also contributes to rural development programs by creating sustainable growth.

Keywords: natural dyes, indigo, sappanwood, madder, screen printing, eco-friendliness, sustainable approach, silk and cotton textiles

1. Introduction

The use of natural dyes has been in India for thousands of years and is part of India's cultural identity. In ancient times, dyed textiles were symbols of status and, because of their cost, were reserved only for the wealthiest classes. Dye sources were earlier extracted from plants, animals, and minerals. Madder and indigo were the major dyes used in India since 2500 BC. With the intervention of synthetic dyes, the use of natural dyes for textile dyeing has been reduced tremendously. Fast fashion demands low cost, simple, and reproducible dyeing or printing process. Both the synthetic dyes and natural dyes play a key factor for coloration and ornamentation of textile materials like silk and other fibers. Synthetic dyes are playing important and predominant role for coloring the majority of silk materials, while natural dyes are being specially used for developing uncommon soothing shades on silk yarns or fabrics in small-scale sector or rural sector, which is gradually gaining popularity due to its nontoxicity and eco-friendly character against possible unsafe ecotoxicity criteria of synthetic dyes [1, 2].

Textile printing in India has been practiced over centuries. Screen printing using natural dyes in India has helped in creating bridge between conventional printing techniques and mass production of textiles in an efficient way. In this chapter natural dyes such as madder, sappanwood, and indigo are used under optimized temperature, pH, and duration for extraction. The cotton and silk substrates are prepared for screen printing by scouring the fabrics. Screens are developed based on the patterns obtained by conventional shibori dyeing techniques. During printing process, madder, sappanwood, and indigo dye extract were used to provide color. More often it is assumed that color will change depending on printing methods and the substrate used. But in natural dyes, this expectation is somewhat high [3], and especially in printing processes, the effect of mordant type is of great importance for the shade of the color as printing is totally a different approach compared with dyeing of textiles. As a sustainable approach toward screen printing, mordants and binding agents used in this chapter are natural. 20% alum is used with madder and sappanwood extraction as metamordanting agent. Tamarind kernel powder is used as thickening agent for madder and sappanwood. Cornstarch is used as thickening agent for indigo.

2. Material and methodologies

2.1 Fabrics

Most of the studies available for dyeing with natural dyes relate to woolen textiles, and such studies on silk are still insufficient and that study on cellulose or lignocellulose textiles is scant. Hence, there is ample scope of undertaking an integrated study of the surface appearance, durability, dye intake, and screen printing of silk and cotton with selective natural dyes.

Silk and cotton fabrics have been considered for experimentation in this chapter. As silk fabrics are best available for commercial use, this delicate filament fiber is well known for its sheen, texture, water absorbency, dye affinity, thermal tolerances, and insulation properties [4]; its dyeability is one of the prime properties needed to analyze the screen printing of shibori designs using natural dyes.

2.1.1 Pretreatment for fabrics

Natural fabrics must be treated to ensure good dye absorbency before dyeing or printing to remove any natural or added impurities while manufacturing and handling the textile materials. Silk and cotton fabrics used in this experiment are treated for scouring, desizing, and degumming. Like in any other dyeing and printing processes, it was found that pretreatment process influenced the printability of cotton with natural dyes [5].

2.2 Dyes and mordants

Commercially available natural dyes like madder, sappanwood, and indigo are used for the experiments, which are sourced from KMA Exports, Tindivanam, Tamil Nadu. Dye sources used for this study are easily available, and the effluents are not hazardous for the human health and nontoxic to the ecosystem. Myrobalan and alum were used as metamordants while preparing the printing paste of madder and sappanwood. Alum is used to treat the fabrics for its low environmental toxicity [6].

2.2.1 Thickening agents

The thickening agents are tamarind kernel seeds and cornstarch.

2.2.2 Reduction of indigo

According to discovery of European wood vat reduction recipe from medieval manuscripts [7], it was found that a hitherto unknown bacterium, *Clostridium isatidis*, is responsible for indigo reduction; this bacterium reduces the indigo particles to soluble leucoindigo. As per the traditional indigo fermentation process, vat routinely requires the addition of red dye madder, which contains high concentration of anthraquinones; these quinines significantly increase the rate of indigo reduction by *C. isatidis*. As quoted by J N Liles in his book *The Art and Craft of Natural Dyeing*, in 1794, Bancroft suggested that the use of glucose alone will reduce the indigo and if reduction did not adequately occur, the addition of oxides of tin can be used. Hence, in this experiment instead of sodium dithionite, jaggery will be used along with sodium hydroxide to keep the vat alkaline.

Reduction with jaggery and madder: indigo cake was powdered, and 10 g was taken in a vat; 1 l of water was kept over water bath at temperature of 60°C, to which 20 g of madder and jaggery was added. About 4 g of sodium hydroxide was then added to the solution, and the solution is maintained at pH 12 by adding sodium hydroxide.

2.2.3 Dye extraction with madder and sappanwood

Dye extraction was carried out at acidic and neutral medium for madder and sappanwood at the temperature of 70°C for 60 min, where 15 g of madder and 15 g of sappanwood were used with two separate vessels containing 150 ml of water. When the extracted solution cools down to room temperature, 7.5 g of alum is added as metamordanting agent for both madder and sappanwood dye extract. Additionally, 15 g of myrobalan was added to madder dye extract solution as mordanting agent. Then the extracted solution of madder and sappanwood was steeped for 12 h, which was later stirred and filtered.

2.3 Printing paste preparation

To make printing paste of indigo, 100 ml water was taken in a beaker and 20 g of cornstarch is added and boiled, to which 100 ml of fermented indigo solution was added and left for further fermentation for 12 h.

To make printing paste of madder and sappanwood, 15 g of tamarind kernel seed powder was taken for each solution and mixed with constant stirring using an electric blender.

2.4 Printing technique

The printing process involves the printing pastes, fabrics, and the screen developed using shibori designs. Fabric is laid on a waxed wooden surface to ensure no movement of fabric while printing. The print paste is guided through the screen mesh or the open areas of the design on the fabrics by using a flexible rubber squeegee. A constant pressure is maintained while drawing the paste from one end to another and during each printing. The printed fabrics are then allowed to air-dry for 24 h.

2.5 Washing and fixing

Printed fabrics were washed with plain water to remove the cornstarch and tamarind seed kernel powder. Indigo samples were washed with cold water followed by hot water with constant stirring. Printed samples of madder and sappanwood were treated with dhawadi (*Woodfordia floribunda*) as postmordanting for fixing the color on to the substrates. Fixation was done at 60°C for 40 min followed by cold water rinse.

3. Testing and evaluation methods

3.1 Color parameters and color fastness properties

Dried samples were then analyzed further for color parameters and fastness according to the specified standards. **Figures 1** and **2** shows printed cotton and silk using indigo, **Figures 3** and **4** printed cotton and silk using madder, and **Figures 5** and **6** printed cotton and silk using sappanwood.

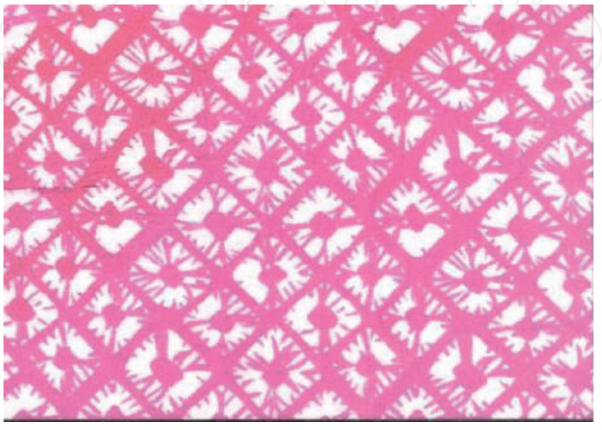

Figure 1.
Sappanwood on cotton fabric.

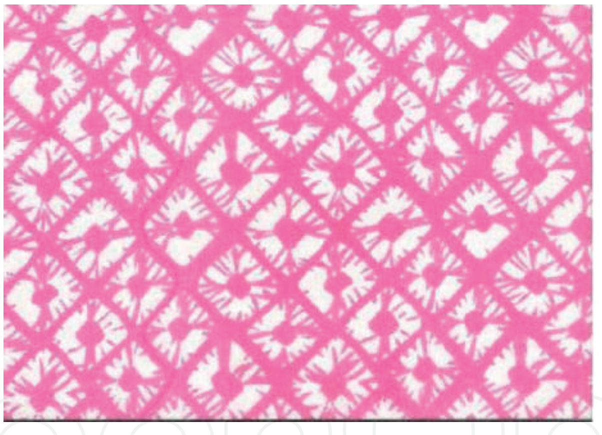

Figure 2.
Sappanwood on silk fabric.

Figure 3.
Madder on cotton fabric.

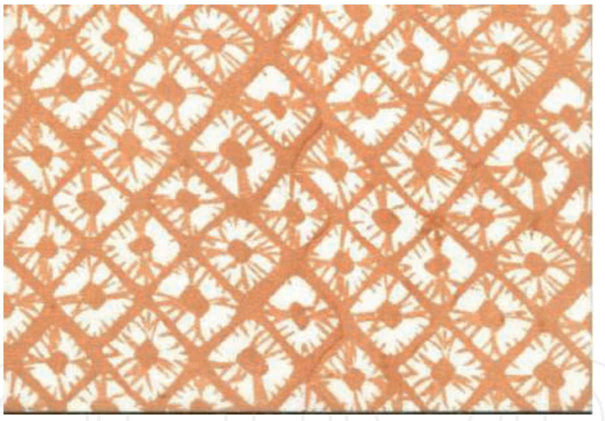

Figure 4.
Madder on silk fabric.

Figure 5.
Indigo on cotton fabric.

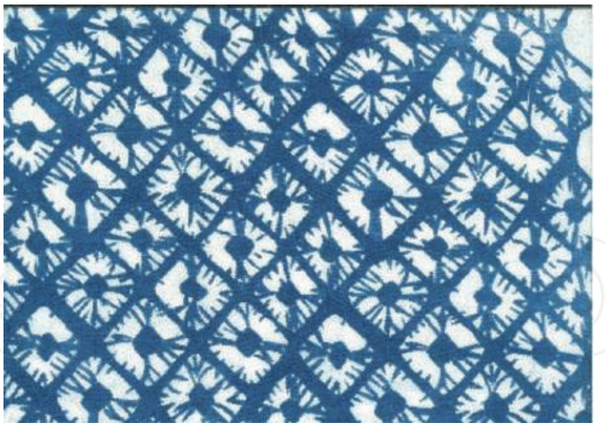

Figure 6.
Indigo on silk fabric.

3.1.1 Evaluation of CIE lab coordinates

Color value of the sample was analyzed based on L*a*b* values using reflectance spectra through spectrophotometer. The L* value is a measure of lightness and darkness of the color to define the color on a two-dimensional chromatic space of green-red axis and blue-yellow axis. Negative a* value indicates greenness in the screen printed samples and negative b* value indicates blueness in the screen printed samples, while positive a* value indicates redness and positive b* value indicates yellowness in the screen printed samples. L*, a*, and b* values were evaluated for five samples, and the average readings are recorded in **Tables 1–3**.

3.1.2 Measurement of color fastness properties

Color fastness to washing of dyed samples was determined by launder-o-meter in accordance with method prescribed in ISO 105-C02. Color fastness to rubbing was determined by crockmeter in accordance with method prescribed in AATCC crockmeter method [8]. Color fastness to light of dyed cotton and silk fabrics was assessed on IS 2454 1985. Light fastness of printed fabric is influenced by chemical and physical state, concentration of dye, nature of the fibers, and mordant type [9]. Color fastness to perspiration of dyed cotton and silk fabrics were determined by perspi-o-meter in accordance with method prescribed in ISO 105-E04:2013 test method. The data obtained are recorded in **Tables 4–6**.

Fabrics	Mordants	CIE color coordinates			Description
		L*	a*	b*	
Cotton	Simultaneous mordanting (alum)	59.44	30.14	−9.60	Lighter, more red, and more blue
	Postmordant (*Woodfordia floribunda*)	60.88	27.30	−11.49	Lighter, more red, and more blue
Silk	Simultaneous mordanting (alum)	56.53	40.13	−17.91	Lighter, more red, and more blue
	Postmordant (*Woodfordia floribunda*)	60.09	37.45	−8.23	Lighter, more red, and more blue

Table 1.
Color characteristics of sappanwood with mordanting.

Fabrics	Mordants	CIE color coordinates			Description
		L*	a*	b*	
Cotton	Simultaneous mordanting (alum)	61.21	13.84	19.14	Lighter, more red, and more yellow
	Postmordant (*Woodfordia floribunda*)	61.32	13.07	19.11	Lighter, more red, and more yellow
Silk	Simultaneous mordanting (alum)	67.69	13.53	11.29	Lighter, more red, and more yellow
	Postmordant (*Woodfordia floribunda*)	61.56	16.40	17.01	Lighter, more red, and more yellow

Table 2.
Color characteristics of madder with mordanting.

Fabrics	Reducing agents	CIE color coordinates			Description
		L*	a*	b*	
Cotton	Jaggery	38.90	−2.94	−22.10	Lighter, more green, and more blue
Silk	Jaggery	51.33	−1.17	−25.74	Lighter, more green, and more blue

Table 3.
Color characteristics of indigo with honey and jaggery.

Fabric	Mordants used	Fastness properties								
		Washing		Light	Perspiration				Rubbing	
		Change in color (cc)	Staining on cotton (cs)		Alkaline		Acidic		Dry	Wet
					cc	cs	cc	cs		
Cotton	Simultaneous mordanting (alum)	1/2	5	1	3/4	4	1/2	4	4/5	4
	Postmordant (*Woodfordia floribunda*)	1/2	5	1/2	3/4	4	1/2	4	4/5	4
Silk	Simultaneous mordanting (alum)	4	5	1/2	3/4	4	2	4/5	5	4/5
	Postmordant (*Woodfordia floribunda*)	3	5	1/2	3	4/5	2	4/5	5	4/5

Table 4.
Color fastness for sappanwood.

Fabric	Mordants used	Fastness properties								
		Washing		Light	Perspiration				Rubbing	
		Change in color (cc)	Staining on cotton (cs)		Alkaline		Acidic		Dry	Wet
					cc	cs	cc	cs		
Cotton	Simultaneous mordanting (alum)	4	5	2/3	4/5	5	4/5	5	5	4
	Postmordant (*Woodfordia floribunda*)	3/4	5	2/3	5	5	4	5	5	4
Silk	Simultaneous mordanting (alum)	4	5	2	4/5	5	5	5	4/5	3/4
	Postmordant (*Woodfordia floribunda*)	4	5	2	4/5	5	5	5	4/5	4

Table 5.
Color fastness for madder.

Fabric	Reducing agents	Fastness properties								
		Washing		Light	Perspiration				Rubbing	
		Change in color (cc)	Staining on cotton (cs)		Alkaline		Acidic		Dry	Wet
					cc	cs	cc	cs		
Cotton	Jaggery	4/5	4/5	3	5	5	5	5	4	3
Silk	Jaggery	4	5	2/3	4/5	5	4/5	5	4/5	3

Table 6.
Color fastness for indigo reduced with jaggery.

4. Results and discussions

It is indicated by CIE color coordinate results. The a* value indicates redness or greenness, and b* value indicates yellowness and blueness. From **Table 1**, it can be seen that a* values are positive which indicates redness and b* values are negative which indicates blueness in printed samples with both metamordanting and post-mordanting agents. From **Table 2**, it can be seen that a* values are positive which indicates redness and b* values are positive which indicates yellowness in printed samples with both metamordanting and postmordanting agents. From **Table 3**, it can be seen that a* values are negative which indicates greenness and b* values are negative which indicates blueness in printed samples with both metamordanting and postmordanting agents.

The light fastness properties of printed cotton and silk fabrics dyed in sappanwood with alum and dhawadi (*Woodfordia floribunda*) as mordants (see **Figures 1** and **2**) are found poor which shows that dyes are unstable to photodegradation. Screen printed samples were evaluated for rubbing fastness. Rubbing fastness grades of dry rubbing are found very good to excellent, but for wet rubbing fastness, it is good. Washing fastness grades for color change for both mordants were found poor for cotton and average for silk, and color staining was observed to be good for all the samples. Washing fastness grades clearly reveal that color change was observed in both the mordants and very slight color staining was seen on cotton and silk fabrics. Screen printed samples showed moderate to good performance during alkaline perspiration test and poor performance during acidic perspiration test (see **Table 4**).

The light fastness properties of printed cotton and silk fabrics dyed in madder with alum and dhawadi (*Woodfordia floribunda*) as mordants (see **Figures 3** and **4**) are moderate which shows that dyes are slightly stable to photodegradation and printing paste interaction is good. Screen printed samples were evaluated for rubbing fastness. Rubbing fastness grades of dry rubbing are found very good to excellent, but for wet rubbing fastness, it is good. Washing fastness grades for color change for both mordants were found good for cotton and very good for silk, and color staining was observed 5 for all the samples. Almost all screen printed samples showed good to excellent performance during alkaline and acidic perspiration tests (see **Table 5**).

Data for color fastness to washing, rubbing, light, and perspiration of cotton and silk screen printed samples (see **Figures 5** and **6**) with natural indigo reduced using natural ingredients has found out during the study by following standard testing methods. The use of natural auxiliaries has resulted in good fastness rating on cotton and silk screen printed samples with indigo. Based on the result, the printed fabrics have good color fastness properties in terms of wash, light, and perspiration. Rubbing was found moderate to poor (see **Table 6**).

5. Conclusions

In the current world of fast fashion, there is an increased concern globally toward the use of hazardous and carcinogenic synthetic dyes like azo and benzidine; these dyes have irrevocable effects on nature and mankind. The growing awareness about sustainability and environment-friendly dyes has created an essential platform for young researchers to revive and experiment with traditional approach of textile dyeing and printing. However, the textile dyed from natural dyes does need value addition to reach the desired market acceptance; printing different motifs using natural dyes can help overcome the requirements of value addition for textiles. Natural dyes have always been preferred for its soothing colors; with the experiment and results obtained in this chapter, it can be seen that the screen printing on cotton and silk fabrics using indigo, madder, and sappanwood has resulted in promising colors and also can be considered as the recommendable alternative to harmful synthetic dyes. This chapter was focused on the application of conventional shibori patterns for screen printing using natural dyes. From the history of fashion, it is known that the comeback of green clothing is in high fashion. In addition, design process of textile design is also served as eco-route in this chapter. There is a huge scope to textile printing using natural dyes; new sources of natural dyes and new trends in fashion applications can create a global niche market for textiles and for natural dyes. This research can be an innovative method for textile design with screen printing. The major significance of this chapter was to add a value on textile by improving the esthetic functions and commercial values of fabric. The application of these techniques can serve as a new and complete method to create innovative fabric design.

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