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 biodegradable 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 onto 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
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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).
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

| 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 granatotine) 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

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**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.

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**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 aluminium 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 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 Liliaceae 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 quercetin (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.

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**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.

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### 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.
2.4.1 Occurrence

The walnut is an 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 a 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.
2.4.2 Chemical constitution

The colouring component present in walnut shell is juglone (CI 75500), a naphthoquinone (5-hydroxy-1, 4-naphthoquinone) 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,
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].
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 showed 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) 60 min (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 dying 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.
### 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].

| Varying parameters | \(K/S\) at \(\lambda_{\text{max}}\) | CDI | RCR (CDI\(_{\text{max}}\) - CDI\(_{\text{min}}\)) |
|--------------------|-------------------------------|-----|------------------------------------|
| 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 | 676 | 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 pH** | | | |
| 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].

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 \textit{Arachis hypogaea} \textit{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 \cite{70}, phenolic acids, procy-anidins dimmers, oligomers and some tannins. The main colouring components in peanut skin are vanillin, catechin and epicatechin (polyphenolic compounds) \cite{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 \cite{72}. Though they contain appreciable amount of polyphenols. Only a portion of these by-products are used as animal feed or as fertilizers. 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 \cite{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 \cite{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) \cite{20}. Peanut skin discarded as waste from food processing industry contains tannins and thus Pandey et al. \cite{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: \textit{Tamarindus indica} \textit{L.}; Common Name: Tamarind or \textit{imli} (Hindi); \textbf{Part used for dyeing:} Shells and seed-coat of the fruit.
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].

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.

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**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.

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**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 methanolic 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 mediums, 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.

### 2.8 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 acuminate* 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-mordanted 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 stable 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 oxyxarotenoids [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.

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**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%.

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**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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