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Review on Sources and Sustainable Methods of Natural Coloration of Textiles

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Abstract

This paper deals with different natural dyes based on their source and hue along with their chemistry and unique processes their application on different natural textiles like cotton, silk, wool and jute. In addition to environmentally friendly metallic mordants, some sources of natural bio-mordants or mordanting assistants containing poly-phenol tannates, flavones, and chebulinic acid have also been discussed with an emphasis on their natural colour component and their role in enhancing colour yield/dye uptake and improving colour fastness to washing on various natural fibre based textiles. Some case studies on application of these natural dyes have been briefly discussed. Few natural dyes having antimicrobial and UV protection properties for making herbal textiles have also been reported. Such natural dyeing together with finishing will provide a niche market for completely eco-friendly specialised natural fibres based textiles having functional features.

Introduction

The process of textile colouration involves colouration of textile fibres and other related substrates like leather and fur, and it heavily relies on the chemistry of synthetic or natural colouring matter. The two main categories of colouring substances are (a) natural and (b) synthetic. Natural colours can be produced from various plant, animal, or mineral sources [1].

Even while the most natural colourants come from various plant sections, some come from animals and are also found in minerals.

Since ancient times, natural dyes have been used to colour textile fabrics. These dyes can be obtained from different plant parts like roots, stems, barks, leaves, hard wood, berries, fruits, flower petals, and fruit peels. They can also be made from certain insects, shellfish, and fruits and vegetables. Most natural colours are non-substantive to textile fibres, which mean they don't have much colouring power on their own. As a result, fixing natural colours for colourants or printing on any sort of textile fibres, yarns, or textiles requires the use of mordants, mordanting aids, tannins, etc.

Thus, the natural colourants comprise of colours sourced from vegetable and animal matter having different chemical compositions. The Society of Dyers & Colourists defines natural colourant as "The natural dyes and pigments comprise of all the colours obtained from animal and vegetable matter with no or very little chemical treatments." Though mostly non-substantive in nature, the natural dyes are mordantable and attach with textiles through metallic salts or tannins etc. They are mostly mordant dyes, although some belong to the vat, disperse/solvent, direct, acid, basic dye and pigment categories.

Synthetic dyes and pigments are produced using a variety of chemical methods that require a significant amount of energy, pose chemical risks or toxicity during the synthesis process, and produce undesirable/unwanted by-products. These harmful by-products have to be released as effluents or into the atmosphere, which pollutes the environment. The creation of these downstream effluents during synthetic dye production have compelled experimenters to look for environmentally benign alternatives, technologies and manufacturing processes that employ colourants from natural sources for dyeing fabrics, leather, and other materials.

Since consumers have recently been much more aware of the pollution, toxicity, and environmentally unfriendly products and processes, such as dyeing with synthetic dyes, interest in natural colourants has increased and some people have even come to see it as a necessity. In order to create entirely eco-friendly coloured textile materials from natural fibers/fabrics, natural colourants applied with natural mordants like alum, or any tannin rich bio-mordants like *hard*a, gallnut, wallnut, etc., are presently being explored as a subject of research.

Literature review has revealed dyeing with natural dyes is sometimes associated non-reproducible shades (shade variation), non-uniformity of shades, difficulty in matching colours, moderate to poor colour fastness to light and wash, etc. But the bigger problem is the difficultly in its collection, variation in its composition from one source to the other, lack of standardisation for the mordanting and colouring processes, and difficult procedures for application on specific textiles with variations in hue and colour fastness depending on the mordants used. There is also the issue of the high overall cost of dyeing to consider. Further the problem is aggravated due to lack of precise specialist knowledge and expertise and near absence of methodically written records on dyeing procedures of specific natural dyes or their binary combinations on any specific fabrics. Hence there are ample scope and need to study and understand the performance of natural colours on cellulosic and ligno-cellulosic fibres based fabrics like cotton and jute respectively. The same is partially true for other high-value natural fibres like silk and wool, as well as the mixed textiles made from them, as their different functional group content affects the natural colouring in various ways.

Use of natural colourants in cave painting and dyeing of on cotton, jute, wool and silk fabrics has been known since ancient times [2]. Growing consumer awareness on advantages of using eco-friendly textiles and clothing has lead to a change in the consumer preferences which is now in favour of sustainable and eco friendly textiles. Prior to the recent breakthrough of chemistry and scientific analysis of these natural colourants, along with its fixation with appropriate mordants/pre-treatments and post treatments, the foundation of natural dyeing was the heritage textiles and handicrafts coloured using traditional methods of natural dye application. There are ample references now available in this regard in literature [2,3].

Due to its environmental friendliness, natural colouring has emerged as a niche product branding in everyday clothing, which has generated added consumer interest towards use of such natural fiber-based textiles coloured with natural colourants. Not all natural colourants are necessarily eco-friendly and hence natural colourants must always be verified for eco-friendliness before use or application.

Classification of natural colourants

Colourants from nature can be classified as per (a) sources of origin (plant, animal and mineral), (b) chemical constitution (indigoid, anthraquinones, naphthoquinones, flavones, carotenoids, dihydropyrans and anthocyanidins and tannin rich colourants) and (iii) colour application system as in the case of synthetic dyes (direct, acid, basic, vat, disperse and mordant) [3-5].

Natural Colourants as per their Hues and Sources

The majority of natural colourants are sourced from different parts of a plant like roots, heart wood, stems, leaves, flowers, fruits, rinds, shells, cocoons, etc. that are cut, dried, ground to powder, then boiled in aqueous media or alternatively in any specific solvent or acid/alkali media to produce a filtrated extract that can be used as a natural colour source [3-5]. Thus, natural colourants may be categorised according to the predominant wavelength of colour reflected with a touch of the presence of another hue or wavelength of colour to achieve tonal diversity or variation in shades/colours that appear on textiles.

Therefore, natural colours are divided into categories according to their predominant hue/colour, such as blue coloured dyes, yellow coloured dyes, red coloured dyes, brown coloured dyes, etc. Important natural colourants, their source, chemistry, and application technique are all discussed here, to enable readers understand the chemistry of colour components present in them and their application process for dyeing different textiles.

Colour Class -1: Blue Coloured Dyes (Natural Indigo)

Indigo is the only important natural blue dye obtained from the leaves of the *Indigofera tinctoria* plant. This colour is also popularly referred to be the "king of natural dyes" and ased since antiquity for producing blue colour. In the present time, it is the most popular blue colour used for denim textiles. Indigotin is the primary colouring component of indigo. Several cultivators in India and Sri Lanka produce natural indigo from indigofera species in the form of cake or powder.

Natural indigo containing indigotin with presence of minor indirubin gives different UV-Vis spectral peaks compared to synthetic indigo [6]. The UV-Vis peaks (in the UV region) of synthetic indigo appear with a sharp peak at 228 nm (1.18 OD), while that of natural indigo appears at 293 nm (1.425 OD). Similarly, in visible region, the UV-Vis spectral peaks of synthetic indigo appears at 608 nm (0.20 OD) and that of natural indigo appears at 610 nm (0.201 OD). In contrast to pure synthetic indigo, which has this UV-Vis peak at 608 nm, natural indigo exhibits a shift of the visible peak to 610 nm due to the presence of additional minor impurities or small amounts of indigorubin.

Another natural indigo class vat dye made in Europe is woad. Pala Indigo (*Wrightia tinctoria*) and Knot knoen weed (*Polygonum tinctorium*) are two more plants that are

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used to traditionally produce indigo. Natural indigo usage started to drop around 1987, when BASF started making synthetic indigo.

Colour Class -2: Red Coloured Dyes

Red natural dyes can be found in a variety of vegetal sources. Below is a list of several well-known sources.

Indian Madder (Manjistha/Rubia): Madder, also known as *manjishtha*/*manjeet* is obtained from the woody branches, stems or roots of *Rubia cordifolia* plant. It is sometimes referred to as the "queen of natural colourants. Alizarin is the primary pigment found in the *Rubia tinctorum* species of European madder. Indian madder is a combination of the compounds munjisthin and purpurin. After BASF began producing synthetic alizarin, the usage of natural madder colour began to decline.

Pinkish red tone from madder is often produced by using alum. A mixture of alum and iron together produces a powerful red colour. A variety of red shades may be created by using alum as a principal metallic salt in conjunction with various tannin-based bio-mordants. Madder-dyed cotton that has been treated with cationic substances like chitosan has a high colour fastness rating for washing. It is possible to distinguish between natural/genuine madder (containing manjisthin) and synthesized alizarin by comparing the UV-Vis spectral analysis of both in the visible region (400-700 nm). The characteristic peaks of the UV-Vis spectra of synthetic alizarin are at 426 and 491 nm, while the corresponding peaks for natural madder (rubia), are at 398 and 426 nm and contain manjisthin.

Brazil Wood/ Sappan Wood: Sappan wood, sometimes referred to as 'patang', is obtained from The small tree *Caesalpina sappan* can be found in the Philippines, Malaysia, and India. Brazil wood (*Caesalpinia Echinata*), which gets its name from the Portuguese word 'braza', meaning 'glowing like fire' also has the same hue in its wood. Aqueous extract of the dye can be used to colour textiles in a red hue with or without the addition of alum mordant and deeper hue is obtained when dyeing is carried out in an alkaline dye bath. After treatment with cationic agents can improve wash fastness of these dyes. This colour mixes well with turmeric to produce orange tones, and with catechu it creates a rich maroon hue.

Morinda: The roots and branches of *Morinda citrifolia*, growing in Sri Lanka and India is used to get red dye. A three to four years old tree can yield maximum colouring matter; while a matured tree typically has little colour. Colour is extracted from the uprooted wood and the free acids in the wood material must be removed before being used for dyeing. Vibrant red tones, such as grand eloquent red and chocolate colour, may be generated through the application of different mordants.

Safflower: Safflower is an annually growing herb and was first cultivated in Afghanistan. Its seeds, which are abundant in polyunsaturated fatty acids, are mostly grown for oil painting. Safflower florets have historically been used for colour extraction that was prized for its vivid cherry-red hue. It has two colour components: 0.3 to 0.6% of crimson-red water-insoluble carthamin, and abundance (26–36%) of a water-soluble yellowish component that wasn't employed as a dye or colour. Before using for dyeing, the yellowish non-colorant matter from the carthamin must be completely eliminated since even in little amounts, it can influence the pure pinkish or scarlet red hue. Colour from dried florets is extracted by continuously washing it with acidulated water to remove all water-soluble yellow coloured matter. The residue containing the crimson red colour is removed with a sodium carbonate solution and washed with diluted acids, dried or mouldered into galettes for use in dyeing. The yellowish portion of safflower florets is now being used to dye pre-mordanted cotton in a light shade. Safflower has been used to give silk and cotton garments a cherry-red colour by direct dyeing. The wash and light fastness of dyeing developed from safflower is poor.

Colour Class -3: Yellow Coloured Dyes

Yellow colour can be obtained from several natural plant-based sources. Below is a list of some of the most prominent and well-known sources.

Turmeric: A well-known natural colouring agent, turmeric is also used as a food colour. Fresh or dried turmeric rhizomes are used to extract the colour. Curcumin belonging to the diaryl-methane class is the chief colouring matter present in turmeric. The substantive dye can be directly applied on dye cotton, hair and silk. The shades produced have moderate wash fastness but poor light fastness. Use of natural source based mordants and tannin (from *harda*/ myrobolan) can improve wash fastness. Better colour fastness to wash and light can be obtained by post-dyeing (topping) turmeric dyed fabric with indigo.

Saffron: Saffron, an ancient yellowish spice from the Iridaceae family obtained from the dried stigmas from the *Crocus sativus* plant. It is grown in Iran, India, and the Mediterranean and used for culinary cuisine, as a food colourant, and for medicinal uses. The colour is extracted in water from the stigmas of saffron flowers by boiling. The dye imparts a bright yellowish colour and can be applied directly on cotton, hair and silk. Alum mordant with this colourant produces an orange-yellow hue known as saffron yellow.

Annatto: A small tree *Bixa orellana* belongs to the Bixaceae family. Also known as annatto, the tree is well-known for its yellowish-orange or cream-coloured obtained from its seeds. It is extensively used in the colouring of butter and cheese. Its pulp contains a lot of tannin and is used in the dyeing cotton, hair and silk in extremely pale orange-yellow tones. Colour from the seeds is extracted under boiling alkaline conditions.

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Barberry: The roots, branches, and stems of the *Berberis aristata* plant are a rich source of colour which can be used to directly colour silk and hair. But the dye can be applied to cotton only with the proper mordanting. The primary colour component in barberry is berberine, an alkaloid. The pigment creates a brilliant yellowish hue having good fastness to washing and average fastness to light.

Colour Class-4: Yellowish-Orange Coloured Dyes

Marigold: Marigold (*Tagetus spp*.) is a plant that produces vivid yellowish or orange flowers. It is available in a variety of colours, such as yellow, golden-yellow and orange. Two of its glycosides termed lutein, together with quercetagétol and flavonol, are the primary components of marigold colourants. It may be used to colour hair and silk in deep yellowish hues and imparts good wash fastness. Improvement in fastness can be achieved through mordants. To achieve brilliant colours, cotton fabrics must first be pre-treated with natural bio-mordants that contain tannic acid/ tannin followed by metallic mordants.

Flame of the Forest (Tesu/Palash): *Butea monosperma*, also known locally in India as *tesu*/ *palash*, gives bright reddish-orange to yellowish-orange coloured flowers. The *tesu* flower petals can be used to extract the colour for dyeing all textiles made of natural fibres. The main colour component in *tesu* is butein. With use of suitable mordants, bright yellowish-orange to yellowish-brown colours can be produced.

Kamala: The dried fruit capsules of kamala (*Mallotus phillipensis*) provide a reddish-orange tint that may be used to dye silk and hair in bright orange-yellow and golden-yellow hues. Colours produced on cotton dyed with kamala have moderate wash fastness.

Onion Peel: The outer layer of an onion (*Allium cepa),* often known as the onion skin or onion peel, is typically discarded after being used to cook meals. However, this leftover onion skin may be used to dye fabrics a natural yellow hue. Flavonoids, which make up the colour component of onion skin, impart vibrant hues on silk and hair. Cotton may be coloured with this dye using appropriate mordants. Onion skin produce shades with moderate wash and light fastness on cotton.

Weld: Another very significant yellow-coloured plant, known as Weld (*Reseda luteola*), is mostly found in Europe. Flavonoid id the colouring matter found in weld and it gives a good yellow colour with good wash fastness on natural fiber based textiles.

Dolu /Himayalayan Rhubarb: After properly mordanting, the roots and rhizomes of Himalayan rhubarb (Rheum emodi), popularly known as "dolu," provide a yellow colour that can be used to dye hair, silk, and cotton. This colour has remarkable wash and light fastness.

Colour Class -5: Brown Dyes Coloured Dyes

Pomegranate Rind (Anar Peel): Fruit rinds of pomegranate (*Punica granatum*) also known as *anar*, are rich in tannins and have a light brown colour component. They are used for both tannin-based bio-mordanting and colouring and are generated as a waste. Its extract yields a brownish-yellow colour and can be used to colour hair, silk, and cotton with good overall colour fastness. Ooccasionally it is used to enhance the light fastness of fabrics dyed with turmeric.

Myrobolan (Harda/Haritaki): Dried fruits of myrobolan (*Terminalia chebula*), commonly known as *harda* or *haritaki*, having high content of tannins also contain a light natural colour that is utilised to produce brilliant yellowish-brown tones on textiles. It is used extensively as '*triphala*' in ayurvedic medication. Chebulinic acid, ellagic acid and other tannins are present in myrobolan and are utilised for better anchoring of the dye to the fibre. Myrobolan is mostly used as a bio-mordant to anchor different natural colours on textiles. Fabrics dyed with myrobolan have exhibited medicinal grade antimicrobial and antifungal properties.

Colour Class-6: Grey and Black Coloured Dyes

Gallnut: Oak galls, also known as gallnuts or *maju phal*, contain a significant amount of elagi-tannin and are used for both bio-mordanting and dyeing with light grey brown colour. With iron ($FeSO_4$) mordant brown to greyish black colour can be obtained. Catechu, or cutch, which is made from *Acacia catechu* heart wood, is used to directly dye cotton, silk and hair in a brown hue. It has a lot of tannins and may be used with iron mordant to get a black colour. Good colour fastness was achieved by the use of iron mordant and logwood extract from the heart wood of the *Haema-toxylon campechianum* tree found in Mexico and the West Indies.

Some promising sources of natural colours are described below with their common and botanical names, part of plant used and colour obtained for selected natural colorants are given in **Table 1.**

Colours from Animals

The majority of the natural colours from animal origin are produced by insects are mainly red in colour. The oldest natural dye from animal origin is Tyrian purple, obtained from seawater mollusc, Murex which is presto-deep violet and thus an exception. For getting only 1 gram of colour, thousands of molluscs required ad this demonstrated its undeniable value. As a result, it was used by the royal family only.

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Table 1: Common and botanical names, part of plant used and colour obtained for selected natural colorants.

Common Name	Botanical Name	Part of the Plant Used	Color Obtained
Siam Weeds	*Eupatorium odoratum Ageratum conyzoi-des*	Whole plant	Yellow
Goat Weed	*Atrocarpus heterophyllus*	Whole plant	Yellow
Jack Fruit Wood	*Delonix regia*	Bark	Yellow
Gulmohar	*Tectona grandis*	Flower	Olive green
Teak (Tectona)	*Acacia nilotica*	Leaves	Yellow
Babul	*Nymphaea alba*	Leaves & Bark	Yellow/brown
Water Lilly	*Dahlia variabilis*	Rhizomes	Blue
Dahlia	*Emblica officinalis*	Flowers	Orange
Amla	*Ziziphus mauritiana*	Bark	Grey
Berberry (Indian Jujube)	*Moringa pterygosperma*	Leaf	Pink
Drum-stick	*Kigelia pinnata*	Leaf	Yellow
Sausage tree	*Spathodeacompanulata*	Petals, Heartwood & Bark	Yellow, pink
African Tulip	*Tamarindus indica*	Flower & Leaves	Yellow, orange
Tamarind	*Rumex maritmus*	Seeds	Yellow, brown
Eucalyptus	*Eucalyptus camaldulensis*	Leave & Bark	Brown
Pomegranate Rind	*Punica Gratinum*	Peel of Fruit	Maroon
Red Sandalwood	*Ptero-carpus santalins*	Wood	Red

Cochineal, which is still used to colour fabrics, is a significant animal dye made from insects of the genus *Dactylopius coccus*. The colour comes from the bodies of cactus-dwelling female insects (Opuntia species). One of its main colourants is carminic acid. Cochineal produces cheerful red colour on protein fibres with good fastness to washing and light. Carmine, a brilliant red colour made using aluminium calcium chelates, is also used as food colouring. Kermes is another natural colour derived from animals i.e *Kermes licis*. Although it is inferior to cochineal in terms of fastness, its hue has been known to protein fibres since ancient times.

Another well-known animal-origin colour that has been utilised since ancient times for the blood red colour of protein fibres is lac. It is obtained from the lac's hardened rod-like hiding (sticklac). *Kerria lacca* is observed to grow on the twigs and branches of several tree species found in India and the South East Asia. It is created as a derivative from sticklac during the production of shellac resin.

Colours from Minerals

Several mineral colours like red ocher, cinnabar, yellowish ocher, malachite, raw sienna, azurite, ultramarine blue, talc, and water colour black are found in nature and may be utilised for colouring. Ocher red was used by monks in ancient times to colour their blankets. It is now commonly used in paintings, showcase crafts, pottery along with natural binders such as guar gum, gum arabic, shellac, and others.

Colours from Microbes and Fungi

Some bacteria produce secondary metabolites that are coloured. *Bacillus*, *Pseudomonas*, *Brevi-bacterium*, *Flavobacterium*, *Achromo-bacter*, *Rhodo-coccusspp* are some bacteria that produce colour. Additionally, it has been reported that some bacteria can create indigo when exposed to petroleum compounds. Microbes as a source of colour offer a benefit since they can easily grow under controlled conditions on inexpensive substrates. Nylon can be dyed with prodigiosin colour derived from *Serratia marcescens*. Some oriental traditional foods are coloured using the fungus, *Monascus purpureus*. Lichens and mushrooms have been employed as sources of colouring in Europe and certain other regions of the world.

Mushrooms and lichens and produce magnificent violet and purple colours on textiles. Tyrian purple and Royal purple was the colour obtained from mollusc in the middle ages. Additionally, they have been used to dye hair brown as well as in shades of yellowish-brown.

Since the 1970s, colourants made from mushrooms have gained popularity. Some Cortinarius species are referred to as mushroom colourants because of the intense colouring matter contained in their growing bodies. Some anthraquinone hues, like as foremodin and dermocybin, are present in *Cortinarius sanguineus* (blood-red web-cap) in glucoside form. Some anthraquinone colours in glucoside form for emodin and dermocybin are present in Cortinarius sanguineus (blood-red coloured web-cap).

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Natural Colours as per their Chemical Constitution

Since natural colours have complex chemical structures comprising not just of single compounds but combinations/ mixtures of several compounds with significant colour components and other assisting non-colour components like tannins, etc, it is challenging to identify them under a single chemical name [3-5]. Their chemical names are occasionally difficult to understand because to their complexity and the presence of both colour- and non-colour-containing components. Natural colourants have common names that are derived from their plant sources; but they are more popularly known by their regional languages. There is a colour indicator that identifies the chemical nature of natural colours by acting as a reference for the chemical nature and the special features/properties of those colourants. Natural dyestuffs are classified as follows on the basis of major chemical structure:

I. Indigoid colours

II. Anthraquinone colours

III. Naphthoquinone colours

IV. Benzoquinone colours

V. Flavonoid colours

VI. Carotenoid colours

VII. Tannin-based colours

Indigoid Colours

Indigotin (C.I. Natural Blue 1, C.I. 75780) and its derivatives form the primary and most significant colour component in the Indigoid natural dyes, whose presence almost completely satisfies presence of all primary colours including blue. Indigotin is present in large quantities in the leaves of several indigofera species, including *Indigotin tinctoria*, *Indigotin errecta*, and *Indigotin sumatrana*, among others. Its structure is similar to that of synthetic indigo (C.I. Vat Blue 1, C.I. 73000), but it also contains a small amount of red colour indirubin, which gives cotton textiles dyed with natural indigo a rich, sanguine reddish-blue tone. Assam indigo (*Strobilanthes flaccidifolius*), woad (*Isatis tinctoria*) and pala indigo (*Wrightia tinctoria*) were also employed as colourants in Europe for blue colour until it was supplanted by superior natural indigo from India. Since indigo is not soluble in water, it must be reduced to become soluble in water as in the case of any vat dye. Indigotin in this water-soluble form is used to colour cotton and other cellulosic and ligno-cellulosic materials. Following dyeing, ambient air oxidises the leuco form to reestablish its original indigotin structure having a blue hue. It has excellent wash fastness and light fastness. Another indigoid hue with exceptional colour fastness is tyrian purple, which is sourced from molluscs belonging to the Purpura and Murex genera and found in the Mediterranean. Chemically it is the indigo 6, 6’ dibromo derivative.

Anthraquinone Colours

This group includes a lot of natural pigments, especially those that give reddish colours. Alizarin made from madder species (European) is the most intriguing natural hue in this group (*Rubia tinctorum*). Other colouring agents include morinda, Indian madder (manjishth/manjeet), lac and cochineal among others. Florets of safflower (*Carthamus tinctorius*) contain a natural red dye called carthamin, which has a structure based on benzoquinone. It gives a cherry red and pink tone to cotton and silk, although with poor fastness.

Naphthoquinone Colours

Henna, walnut shells, and other natural colours are examples of those in this category. Lawsone, a 2-hydroxy naphthoquinone, is one of the colouring matters in henna, while juglone, a 5-hydroxy naphthoquinone is found in the walnut shells. This class of dyes also generate an orange, red, or reddish brown tone, which is one of the characteristics of anthraquinone-based dyestuffs.

Flavonoid Colours

The majority of the naturally occurring yellowish colours have a structure made up of hydroxyl or methoxylated flavones. Colourants with this flavone-based chemical constitution are employed to produce a variety of hues ranging from light-yellow to creame colours. In Europe, *Reseda luteola* (weld) or Dyer’s rocket are often used for brilliant and fast colours on silk and hair. Flavone, luteolin, often known as CI Natural Yellow 2, is one of the colouring components present in weld. Marigold flowers (*Tagetus spp*.) petals also contain querceta-getol (a flavonol), which has luetin as its primary chemical component. Dihydropyrans are almost structurally similar to flavones. Haematin and its leuco derivative haematoxyl the major colouring component found in Logwood (heartwood of *Haematoxylon campechianum*, CI Natural Black 1) also belong to this class of natural dyes. This category also includes colours derived from *Caesalpinia echinata* (Brazilwood) wood and *Caesalpinia sappan* (Sappan wood) wood. Brazilin, the colourant material in both, is oxidised to produce the reddish dye with a multicolour tone known as brazeilein.

Butein is a yellowish-orange colour that comes from Butea monosperma (tesu/palash flower) flowers. Both butein and rottlerin, the primary orange red powdery colouring substance in ripe fruit capsules of *Mallotus phillipensis* (Kamala, CI Natural Orange 2), produce a yellowish colour and have chalcone structures that can be considered as open-chain analogues of flavonoids.

Carotenoid Colours

The primary colourants in this class of natural dyes are the bixin and nor-bixin that are found in annatto seeds. Crocin in is the colourant present in the stigmas of the saffron flowers and is

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often known as saffron smirch smirch. Nictanthin having a similar carotenoid structure is found in the orange pigment contained in the corolla tubes of *Nyctanthes arbor-tristis* flowers.

Tannin Rich Colours

Tannin-rich colorants contain acidic and polyphenolic compounds that are found in certain sources of natural colours. For instance, gallnuts, babul bark, pomegranate peel, and myrobolan containing ellagic acids, gallo-tannins, or chebulinic acids besides poly-phenolic colour components like catechin, epi-catechin, etc. This class of colourants also has a tendency to alter colour in response to a change in mordant and this depends on the colour-mordant-fibre complex formed with or without use of different metallic mordants. Babul (*Acacia nilotica*, CI Natural Brown 3) and cutch (*Acacia catechu*) belong to this chemical family. These tannin-rich natural colours can also be utilised as bio-mordants rather than colouring agents as natural dyes because of their variable percentages of hydrolysable and non-hydrolysable tannins and coloured compounds present in them. However, when used for dyeing with a mixture of such bio-mordants with metallic mordants, applied sequentially one by one before dyeing, they give light yellow to brown colours.

Natural Colours with Other Unclassified Chemical Structures

The well-known spice and culinary colour turmeric contains curcumin, a yellowish pigment that is related to the chemical structure of diaryl methane. The only known naturally occurring cationic (basic dye) colour is berberine, is an alkaloid and is contained in the roots and stems of *Berberine vulgaris* and *Berberis aristata*, and in the bark of the *Phellodendron amurens* (cork tree).

Colour Extraction Process from Natural Dyes

The amount of natural colourants found in natural resource materials is extremely low and making their extraction crucial. To extract the colour components included in their original natural resource material, they need a specialised extraction process with the appropriate pH, temperature, length of extraction, material to liquor ratio and extraction media [1-2,4,8-12]. Then there are the effects of various chemical additives, such as alkali, acid, and alcohol, on the effectiveness of the extraction process, as well as some physical interventions, such as the use of sonicator vibration, magnetic vibration, and radiation of various types, which have varying effects on the extraction of natural dyes. In order to extract colour from varied sources of different natural colourant resources [8-14], under various extraction conditions, it is necessary to determine the optimized conditions [8-14].

Aqueous Extraction of Natural Dyes: As a part of preparing for extraction of colouring components from the naturally sources, the colour-containing materials (roots, barks, woody branches, leaves, petals of flowers, seeds, and vegetables and fruits skins or peels, etc.) must be dried under the sun and then to be ground/broken or crushed/pulverized into small pieces followed by overnight soaking in water, alkali, or acid. It must be filtered to remove non-dye elements after being heated or boiled for a specific amount of time and at a specific temperature with intermittent stirring. One of the drawbacks of this hot aqueous extraction process is that some of the colourants or colour components may degrade during boiling. This temperature at which a dye component may break can be determines through a Differential Scanning Calorimetry (DSC) study of individual natural dye as has been previously reported [9]. The dye components must be water soluble for this method to work; otherwise, extraction will fail.

Extraction in Acid/Alkaline Mediums: The majority of natural colourants are glycosides, making them hydrolizable or chemically degradable soluble compounds or simple water soluble. However if insoluble, the colouring components in natural dyes can be extracted under acidic/alkaline conditions [10–14] while maintaining pH and other extraction variables. Removal of colour from *tesu* flowers involves an acidic hydrolysis mechanism. Extraction under alkaline conditions is appropriate for colourants having phenolic –OH groups. This technique can also be sued to extract colourants from seeds of annatto, lac and saffron.

Extraction Using Ultrasonic/Microwave Energy: Vibrating sound wave of ultrasonic sonicator supplies higher energy for vigorous stirring of natural resource materials thereby leaching out the colour component in better way. Additionally, sonicator vibration [12,13], in an aqueous extraction-bath helps in increased swelling of the materials, which aids in the extraction of the colour. This process for extracting natural colourants using a sonicator has a number of benefits over a basic hot/boiled aqueous bath method, including advantages of use of less volume of water, reduced time and lower temperatures. Microwave radiation ionizes better the dye component to leach out with additional bulk heating inside water molecules by friction of water molecules and polar dye molecules by the action of changing frequency and oscillation of polar molecules subjected to microwave heating at particular mega-hertz the microwave is operated by change of +ve and -ve poles in two electrode points of microwave.

Extraction Using Enzyme: Natural colouring materials can also be extracted using an enzyme as a bio catalyst. The natural colour components are easily loosened from the resource materials through enzymatic hydrolysis increasing colour yield at lower temperatures and shorter processing times [12-14]. Thus the efficiency of colour extraction from natural colouring materials is increased by application of an enzyme. Natural indigo dye solution can be successfully extracted using an enzyme-assisted hydrolytic mechanism or system. Indimulsin enzymes are used to break the glycoside bond between indigotin and indican to produce glucose and indoxyl. A suitable mixed enzymes like cellulase, amylase and pectinase is shown to be considerably more effective in extraction of colorants from natural source materials like dried and crushed roots, stems, leaves, flowers, seeds etc. and extraction of colour from annatto has been effectively carried out in a mixed enzyme bath.

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Extraction Using Organic Solvents/Alcohol: Extraction of natural colours with the aid of alcohol or organic solvents such petroleum, acetone, ether and chloroform, ethanol gives better colour yield, requires less volume of water, less temperature, and less time [10-12]. In comparison to a simple hot/boiling aqueous bath of extraction, it is invariably a more versatile type of extraction where along with water soluble components, alcohol soluble components are also easily comes out in the extracted liquid.

Mordants and Mordanting

Cellulosic or ligno-cellulosic materials do not have attraction or affinity for natural colours; hence they need another bridging compound known as mordants to attract and fix natural colourant molecules [10-14]. Mordants are those substances that have affinity for textile materials as well as natural colours and can act as a bridge between the textile fibres macromolecules and natural dye molecules [14]. Therefore, even though natural colourants don't have an affinity for any particular textile fibre, they can still be applied by using mordants or assistant compounds like metallic salts or many other specific compounds like *harda*, babul bark, pomegranate peel, etc. In the case of very few natural colours having some sort of affinity for the specific textile fibres like wool, silk as protein fibres, the use of mordants improves the fastness by forming a complex between the natural colourant, the mordants that is insoluble in water, and the active group of textile fibres (–OH group in cellulose). In contrast to pure cellulosic (cotton) fibres, these vegetable dyes/natural dyes occasionally may react with celluloses and hemicelluloses or lignin in jute and linen, but the resulting colour produced is duller and lighter as opposed to the vibrant colours obtained on hair, wool, and silk. Hence, a mordant is needed for obtaining deep shades with good colour fastness on cotton dyed with natural dyes.

As a result, mordanting is unquestionably more crucial for cotton than it is for viscose and other fabrics like hair, silk or wool that takes colour quickly due to the protein fibres' wide availability of anchoring sites for natural colour attachment provided by the amino and carboxyl acid groups. There are three different kinds of mordants: tannins, metal salts or metallic mordants, and oil mordants each of which is explained below.

Metallic Mordants

Metallic mordants are widely and frequently used for dyeing textile fibres with natural dyes. Metal salts of aluminium, magnesium, iron, copper, and tin are employed commonly used as mordants to fix traditional natural dyestuff. Some metal salts, such as chromium salts, copper salts, cobalt salts, nickel salts, etc., are not environmentally friendly because they contain objectionable/hazardous heavy metals and have been listed under eco-mark regulations. As a result, these mordants shouldn't be used to preserve the environmental friendliness of naturally dyed fabrics and to adhere to pollution control board norms regarding the permitted/non-permitted limits of eco parameters of discharged effluent. Copper is also in the objectionable group of metals, but it may be admissible in specific situations maintaining eco norms that say up to 5 mg/litre or 5 ppm copper may be allowed. Hence copper can be used to some extent in small quantities that does not exceed the admissible limit of copper on copper-mordanted natural dyed cloth. However, presence of chromium in dyed fabric is not permitted at any concentration and its presence in effluents is also undesirable as per environmental pollution control norms. Iron and alum might be regarded as eco-friendly mordants.

Different metallic mordants can be used to create various colours using a same dye due to hypsochromic or bathochromic shift in hues under influence of metal moieties in the dye-mordant-fibre complex formed after dyeing the fibre with natural colorants in the presence of suitable mordants and corresponding colour and tone vary depending on mordant type and its concentrations used. The colour tones and colour fastness of colour complexes made with various metallic mordants can vary. When mixed with aluminium, alizarin creates a red lake or complex, and when paired with an iron mordant, it creates a violet lake. Additionally, the naturally extracted golden colour of onion skin will turn orange with stannous chloride mordant instead of alum, and it turns slate-grey with ferrous sulfate. Because mordanting reduces the tensile strength of textile materials, the amount of mordant employed should be kept to a minimum.

Oil-based Mordants

Despite being the most commonly used mordant alum is unable to produce the dark red colour with madder. Oil-based mordants mostly find use in dyeing cotton with madder for obtaining this dark red colour. *Til* (sesame) and castor oils were historically employed to get the red colour of madder on cotton, but in more recent years, Turkey red oil (TRO), a sulphonated castor oil, has taken their place as a reliable mordant for producing a specific shade and tone. Turkey red oil when used as a mordant allows alizarin in madder to get fixed onto the cotton by forming a complex with the Turkey red oil, forming a deep red colour.

Tannins rich Bio-mordants and Tannic Acid

Tannins rich natural agents like gallnut, *harda* and other naturally occurring substances are currently employed as bio-mordant [9,14] to assist in the formation of vegetable-tannin mordant-cellulosic fibres-natural dye complex with or without alum as metallic mordants. Such tannin or gallotannin containing substances available for natural dyeing generally include myrobolan (*Terminalia chebula*), oak galls/gallnut, sumac-gall, babul bark, and pomegranate peel. Mordanting with vegetable tannins is cheaper and the tannins can be obtained by gathering parts of the plant such leaves, fruits, and galls as done in the case of natural dye.

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Myrobolan contains two types of tannins, ellagi-tannins and chebulinic acid that are substantially found in the fruit peels. Additionally, a little yellowish-brown colouring substance is also present, giving the fabric a yellowish tint on pre-mordanting with it. Myrobolan/*harda* is widely used in mordanting for all types of natural dyeing of cotton as well as for producing deep shades by anchoring of colour component in natural dyes with the tannin present in the bio-mordant i.e *hard*a. Moreover addition of *harda* as bio-mordant with ferrous salt as metallic mordant helps to produce grey to black colour tones on cotton.

Sumac or the colourful Rhus species contain 15 to 20 types of gallotannins in their leaves and outer growths. This gallotannin in the mordant gives an olive-green colour. Sumac cannot be used for light and brilliant tones due to the presence of some natural colourants [9,10]. When used with gallnut or *harda*, they serve as a tannin-rich bio-mordant or mordanting assistant cum catcher/fixer of metallic mordants. Gallnut is well-known for its exceptional antioxidant and antibacterial properties.

Additionally, they are known for significant medical benefits, such as those related to detoxification, antifungal activity, antimicrobial activity, anti-cancer, anti-allergic and anti-diabetic responses. Extract of gallnut has been utilised for tanning and mordanting and is also being researched as agents for finishing to produce functional textiles, such as antibacterial and/or antioxidant medical textiles.

Natural Potash Alum as a Natural Metallic Mordant

Natural potash alum (*fitkari*) is also used as a metallic natural-mordant [10], which also gives a neutral colour on natural fibre based cotton textiles, without tonal changes in colour development on cotton. Mordants are required to improve a fibres' affinity for a natural dye in order to get dyeing with good depth of shade and to produce stronger dye-mordant-fibre bonds.

Alum has been used as a mordant since 2000 B.C. Natural alum is potassium aluminium sulphate compound having the chemical formula, $KAl(SO_4)_2$. A fine white powder called potassium alum is sold both as a seasoning and as antibacterial after-shave cubes.

Soda alum used in incinerating powder and also as an acidulant in food. Ammonium alum is used in tanning of leather, dyeing of textiles, making fire-retardant fabrics, in the manufacture of porcelain cements and also finds application in purification of water and production of some deodorants. Chrome alum is used in tanning of leather and can used grow lavender. Ferric alum is used in lower-grade paper for size and weighing. Aluminum sulfate is a typical metallic salt used as an environmentally friendly mordant for natural dyeing. Because the selenium-containing alums are potent oxidizers, they can be utilised as potent antiseptics.

Dyeing Processes for Application of Natural Colours

Natural dyes are distinct from synthetic dyes in terms of their chemical makeup, functional group content, and dyeing techniques, yet their fundamental dyeing methods may be compared to those of their synthetic counterparts based on application-based categories. While the ideal temperature, duration, and pH for dyeing may vary, the initial steps always remain the same. Natural dyes can be applied to pre-mordanted or simultaneously mordanted cotton by carefully heating the aqueous dye bath to a temperature that is close to boiling or to a recommended temperature suitable for a particular dye.

Although cotton may require higher dyeing temperature for dyeing, hair, wool, and silk can be coloured at lower temperatures with the same dye. The pH of the extracted dye liquor of most natural colourants may be neutral, acidic, or alkaline depending on the dye molecular structure, their associated functional groups, and whether any other chemicals coming out with the colourant during extraction. As a result, some natural colourants have an alkaline pH and others an acidic pH. In order to get the proper pH while dyeing protein fibers/textiles like hair, pashmina, wool, and silk, 10–20g/l of acetic acid is often used. To obtain proper pH, use of formic acid and hydrochloric acid may be needed.

It is typical to use 20-30% of the natural dye based on the weight of the dried source because the colour content in the raw natural material is unknown and typically low. On the other hand this amount can be reduced to 2-3% when purified colour powder (purified by soxhleting or by vacuum distillation) is used. The quantity of mordants is also estimated on the basis of percentage shade of the natural source materials selected for dyeing; for a darker shade or colour tone, a large amount of mordants is required. To improve reproducibility of shades, the dyeing conditions for each mordant-dye-fibre system should be separately optimized.

The fibrous material to be dyed by natural colourants must first be pre-mordanted; if not, it must be introduced into the dyeing extraction bath (simultaneous extraction, mordanting and dyeing) at room temperature. After the colour (extracted dye solution of natural colour) has been added, the temperature must be raised gradually and the salt added in two instalments to achieve uniformity in dyeing. The textile material that is to be coloured must be maintained in a dye bath at the highest dyeing temperature for at least an hour in order to give the fibre time to absorb, diffuse and fix the dye on surface as well as well inside the fibre. To achieve dyeing consistency, the fabric movemnet in the dye bath is crucial. The fabric must be continuously moved or rotated with stirring in the dye bath. This can be achieved effectively by dyeing in machines like jigger, winch, etc. In any case, the cloth should not be stirred continuously as it may damage the fabric especially when delicate textiles like silk, pashmina, or any other finer fabrics are dyed with natural dyes. In such circumstances, it is desirable to use a material-to-liquor ratio of at least 1:10 to 1:30 in beakers or lab dyeing and 1:5 in jiggers so that the entire fabric is immersed in the natural dye liquor resulting in uniform dyeing. The material dyed should be cooled in the dye bath itself

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according to some traditional methods of applying natural dyes. The dyed fabric must be then removed from the dye bath and the excess dye that has adhered to the fabric surface thoroughly removed by rinsing in water and light soaping for 15 minutes using 2 g/L soap. The dyed material should be washed twice and dried outside. Soaped of the dyed fabric at 40-50°C using non-ionic soap produces excellent wash and rubbing colour fastness. Hydro extractors maybe employed to compress the washed and soaped fabrics to remove surplus water. However, if mordanting is not done first, the dyed materials may occasionally be post-mordanted without being washed or soaped; in this instance, soaping must be done after the post-mordanting and further washing are finished.

When pre-mordanted cotton fabric is to be dyed using colourants such as to madder that have no affinity for the fibre, the pre-mordanted and dyed material may be subsequently post-mordanted to get different tones and improvements in fastness. Post-dyeing after-treatments with natural tannins, UV absorbers, and chitosan, among others can aid to enhance or improve the colour fastness to wash and light [9]. Treatment with cationic agent improves the fastness to wash for anionic natural dyes and treatment with UV absorbers increases light fastness for many natural dyes [9], although these treatments result in subtle variation in the colour tone.

Significant Studies on the Natural Colours Used for Textiles

Excerpts from some significant studies on the standardisation procedures for colour extraction, mordanting, and dyeing using natural colourants have been briefly described mentioning the key findings with focus on advancements in the field of natural coloration of textiles.

Mortazavi et al. [15], studies colouring of natural hair using saffron (*Crocus sativus* L.) flower petals obtained from a perishable herb belonging to the Iridaceae family. Iran, Greece, Spain, Turkey, India, and Morocco are the main saffron producers. Several methods of dyeing with saffron petals using various mordants have been investigated to understand the effects of different mordants on colour tone/tinge, light and wash fastness of coloured materials. Using saffron flower powder, the dyeing process was carried out while preserving the time, temperature, and MLR, and dried at room temperature. Dyeing was carried out using powder of saffron petals maintaining the time, temp and MLR and the fabric was dried at ambient temperature. Colours obtained varied from light-yellow to light-brown under effect of different mordants where $Na_2Cr_2O_7$ and $FeSO_4$ were found as the best mordants that rendered good wash and light fastness properties.

Shin et al. [16], carried out one-step dyeing of cotton with natural indigo using infrared dyeing machine followed by oxidation and washing. Darker shades were obtained with increase in the percentage of indigo used and mercerized cotton showed better (two times higher) uptake of colour than the untreated cotton. Maximum K/S value was observed with the use 4g/L of sodium hydrosulfite. It was possible to achieve good wash fastness (Grade 4-5) with nearly no staining of the adjacent fabric, and good dry rubbing fastness at reduced colour strength. Additionally, the decrease in microorganism growth on cotton cloth coloured was visible rendering the dyed fabric as antibacterial.

Veríssimo et al. [17], dyed cellulosic materials with annatto seeds (*Bixa Orellana* L.) using different methods of dye extraction, pre and post mordanting processes and dyeing under different process conditions. *Bixa Orellana* L. is most commonly available in the tropical regions like South America, India and Africa. KOH based method of extraction was followed to determine bixin content in the extracted dye liquor. The powdered extracted dye used for dyeing cellulosic materials was found to be 4% on the weight of the seeds, which corroborates with earlier studies. Extraction in ethyl alcohol proved to be better than other solvents. Reflectance values of dyed fabrics revealed that the colour in the dyed material changed from orange to red when dyed at different intervals of time (30, 60, and 90 minutes), and with the mordant used. The level of exhaustion of the dye remained constant after the first 30 minutes of dyeing time. Spectrophotometric reflectance curves of the dyed samples showed that several mordants, such as potash alum, tannic acid, and citric acid, improved the fixing of the dyes.

In another research work by Montazer and Parvinzadeh [18], marigold extract was used to dye wool yarns in a yellow-orange colour. Marigold is most commonly cultivated in South Asia, Mexico, Peru, and India, for various purposes like gardening, to make garlands and for adorning buildings etc. The petals are yellow, and cleaning treatments create structural alterations to the dyes' chromophore systems as a result of pH shifts. Mordanting of wool was done with $AlK(SO_4)_2$ maintaining MLR 1:40 and pH 5 (adjustment of pH was done using formic acid). Dyeing was carried out by gradually raising temperature from 40 °C to 85 °C over 20 min and for 1 hour. After-treatment was done by using ammonia. The study showed that the change in colour hue of marigold dyed yarn decreases with the increase in ammonia concentration. When the sample is exposed to 1% ammonia solution, L* decrease while a* increases. The wicking rate also decreases by ammonia after-treatment as well as the results of the light-fastness and wash fastness are also poor.

A study on dye extraction from the waste barks of Turkish red pine (*Pinus brutia Ten.*) and its application on cotton, flax, wool, silk, tencel, polyamide and acrylic fibers was conducted by Avinc et al. [19]. Mordanting was carried out using 20% mordants like alum and oak ash. The powdered dyestuff was added to the dye liquor at room temperature and the temperature gradually increased to 100°°C. Thereafter dyeing was maintaining 100°C temp for 60 minutes followed by lowering of temperature to 60 °C and washing for 5 min before air-drying. The coloured materials

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had excellent wash fastness, very little staining, moderate light fastness, and good rub-fastness.

Application of curcumin and madder on acrylic fibres was explored by El-Shishtawy et al. [20]. While madder (*Rubia tinctorum* L.) is grown as a source of the deep red dyes found in its root, curcumin is commonly utilized as a food colourant. Acrylic fiber was pre-treated with two concentrations of ammonium acetate and hydroxylamine hydrochloride using MLR - 50:1, temperature – 85 °C and time - 60 minutes. The mordant utilised was alum and ferrous sulphate, and the dyeing was done at a pH range of 2 to 6. By including alum, the coloured samples' washing, rubbing, perspiration, and light fastness qualities were improved. The light fastness rating was enhanced with ferrous sulphate, especially for samples coloured with madder. The natural dye attach to the modified acrylic fibers mainly through ionic and hydrogen bonds.

The study by Kamel et al. [21], determined that a variety of attractive, colourful colours may be produced by using different mordants and mordanting procedures to dye cotton fabrics. Cotton was dyed with saffron (*Crocus sativus*) using both conventional process by heating and an enhanced approach using ultrasonic wave vibration. The study explored how variables like pH levels, temperature, dyeing time, salt concentration, and ultrasonic waves affected the dyeing qualities. Mordanting with potassium aluminium sulphate, copper sulphate, stannous chloride, ferrous sulphate, and tannic acid were done by the pre-mordanting, post-mordanting, and simultaneous mordanting processes and dyeing was done under the following conditions - MLR -1:50, various pH ranges (3–8), various sodium chloride concentrations, varying dyeing durations and varying temperatures. Cotton dyed using ultrasonic wave showed stronger colour retention than fabrics treated using conventional techniques. Pre-mordanting produced better colour strength than post-mordanting and simultaneous mordanting.

Gias Uddin [22], explored the use of onion outer peels as a source for natural colours for dyeing silk. The colour from the onion peel was extracted under hot aqueous conditions maintaining pH - 5. Before mordanting and dyeing, the degumming of silk fabric was done using an aqueous soap solution, and bleached using hydrogen peroxide. A variety of mordants like alum, tin, ferrous sulfate, tartaric acid and tannic acid were used for pre-mordanting. Dyeing was conducted at a pH - 5, MLR - 1:50, temperature – 80 °C and time - 60 minutes. Findings indicated ferrous sulfate to be the best mordant. Fabrics mordanted with metallic mordants (alum, ferrous sulfate, tin) showed higher ΔE values compared to those mordanted with non-metallic mordants (tannic acid and tartaric acid). Fastness properties to different agencies like washing, light, dry cleaning, rubbing, and perspiration were evaluated and in general found to be satisfactory which in some cases also improved.

In a study by Adeel et al. [23], silk fabric has been dyed with safflower (*Carthamus tinctorius* L.) extract using exhaust dyeing process. Extraction of carthamin, the natural colorant in safflower was carried out in acidic and aqueous media at boiling temperature for 60 minutes maintaining MLR - 1:25. Bio-mordants like lawsone from henna and curcumin from turmeric were used to produce newer shades and improve the colour fastness properties.

Polyester (PET) fabric has been dyed with natural dye like Rhubarb (*Rheum officinale*) by Shahin et al. [24]. Polyester was first treated with NaOH solution to soften its surface and improve some properties like water absorbency, fabric pilling etc. Dye extraction was carried out in an aqueous medium (water). Dyeing process was carried out at pH - 2.5, MLR - 1:100, with use of a carrier i.e. salicylic acid, temperature gradually increased to 40 ˚C to 95 ˚C. Results reveal that alkali etched polyester may absorb dye more deeply and in greater amounts than untreated polyester, which displayed a slightly different colour tone. When polyester was coloured using Dolu, it created a yellow tint and had acceptable fastness qualities when compared to the same cloth dyed with comparable synthetic disperse dyes.

Rossi et al. [25], used eucalyptus bark extract to dye cotton, wool and nylon without traditional mordanting agents. Eucalyptus plant is widely cultivated in Brazil. The knitted fabric (98% cotton and 2% of elastane), wool and nylon 6-6 fabric were dyed using the exhaustion dyeing method without use of metal salts. Dye was extracted using MLR -1:10. The observed results indicated poor colour development with yellow, yellowish-brown and brown colours on cotton, nylon and wool in the absence of mordanting agents though the wash fastness in case of nylon and wool fabrics were outstanding and it was very good on cotton.

Significant Studies on the Extraction of Natural Colorant

Azeem et al. [26], optimized the conditions of dyeing cotton fabrics with four algal species, brown algae (*Iyengaria stellata*, *Sargassum muticum*, *Colpomenia sinuosa*), red algae (*Laurencia obtuse*), were extracted using alkaline (NaOH, KOH, Na2CO3), acidic/organic solvent (ethanol and methanol, acetone, and N-hexane), and under various temperature, pH, and salt concentration conditions (exhausting agent). The standard aqueous extraction procedure involved combining algae powder from each species while maintaining a temperature of 60 °C for 60 minutes. NaCl salt in varying concentrations such as 2-10% was applied as an exhausting agent and dyeing was carried out at different pH levels (3-11), varying temperature (30-90 °C) and varying dyeing time (15-75 min). Extraction using KOH (4%) and acetone (80%) provided highest colour strength on cotton fabric dyed with algal species. Among four algal species L. obtuse provided the highest yield of colorant. Pre- and post-mordanting with iron sulphate and tannic acid gave darker shades on cotton

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fabrics for all the four algal species used. *I. stellata*, *C. sinuosa*, and *L. obtusa* extracts gave cotton fabric a creamy white appearance; *S. muticum* treatment gave fabric a light brown colour, and pre- and post-mordanting of algal powder with tannic acid gave fabric several hues, including light and dark brown.

In their study of a few dyeing techniques to improve the stability of natural colour, Ngamwonglumlert et al. [27], found that choosing a suitable pre-treatment technique is crucial because the ultimate depth of colour depends on it. A suitable extraction method for the extraction of chlorophylls, carotenoids and anthocyanins by irradiation process was studied, though it is not suitable for not-heat-sensitive colourants like betalains. Processes other than non-thermal, such as ultrasonic aided extraction (UAE), maybe employed for betalains. Combination of different extraction can be followed to increase both yield of extraction and colour stability.

Hong [28], dyed cotton fabrics with gallnut extract using infrared (IR) dyeing and pad-dry-cure processes. For extraction, gallnut was dried and ground to powder before immersing in water, boiling for 60 minutes and then filtering to remove insoluble residues. Invesitigation of mechanical properties, antibacterial properties and antioxidant performance of the dyed cotton fabrics were done. The pad-dry-cure process (curing at 120 °C for 15 min.) was found to be superior to the IR dyeing process because it produced less discoloration, whereas the IR dyeing machine produced a notable colour shift that was hastened by cationization during dyeing for IR radiation initiated heating. Gallnut extract also has good antibacterial properties and can be used as a dye-cum natural finishing agent by the less cumbersome and less time consuming pad-dry-cure method of application.

Jiang et al. [29], dyed wool with aqueous extract of *Caulis spatholobi* plant under varying process parameters of MLR - 1:40, pH - 12, time - 120 min and temperature – 100 °C, which were optimized. The extract was sieved twice before dyeing. The primary functional groups found in *Caulis spatholobi* were assessed using Fourier transform infrared spectroscopy (FT-IR). Metal mordants such as aluminium sulphate, ferrous sulphate, copper sulphate, and zinc sulphate were used in the dyeing process. Techniques for pre-mordanting, meta-mordanting, and post-mordanting were used.

In another, Sinnur et al. [30], approach to dyed cotton Khadi fabric using aqueous extract of babul bark (*Acacia Nilotica* L.). For extraction, dry and powdered babul bark was boiled under varying conditions such as MLR - 1:30–1:80, pH - 4–10, time periods - 15–90 minutes and temperature 60-90°C and the conditions of aqueous extraction of colour component from babul bark was optimised/standardized MLR-1:30, pH-6, temperature-60 °C and time - 45 min.

In order to provide cotton, silk, and wool fabrics an antibacterial quality, Lee et al. [31], extracted natural dyes from pomegranate rind, cloves, and oak gall under a heated (90 °C) aqueous bath media. It was found that the chemical nature of the colourant, together with the tannin concentration in it and the kinds of functional groups contained in the fibres, greatly influences the dyeing results.

To learn more about the make-up of the dyestuffs and how they age, Ilaria Degano et al. [32], used an analytical procedure based on Na2EDTA/DMF (ethylenediamine tetra acetic acid/ dimethyl formamide) extraction and high-performance liquid chromatography (HPLC) analysis using high-resolution mass spectrometry detection. Because highly polar chemicals, such tannins, typically exhibit poor retention efficiency in traditional reversed-phase (RP) columns, an RP-amide embedded polar group stationary phase was employed to provide the best retention of the polar tannin compounds. These HPLC spectra can serve as fingerprints to recognize this dye in fabrics that have been dyed.

In another study by Sinnur et al. [33], extraction process of natural dye from anar peel (pomegranate rind) calculated 20% on the weight of dye source material was optimized as MLR - 1:50 and pH 5-7, temperature 80°C and time - 45 min.

Some Important Studies on Mordants and Mordanting for Natural Coloration

Ashrafi et al. [34], conducted a research on using myrobalan extract as a natural tannin-based bio-mordant (without any metallic mordants) for subsequent green dyeing with different natural dyes (using only myrobolan mordant coantianing chebulinic acid in it) and it gave red colour on silk and wool material.

Another significant study by Deveoglu et al. [35], investigated at the effects of utilizing gall oak (*Q. infectoria Olivier* containing 10-20% tannins) as a bio-mordant and 6% aluminium sulphate [$Al_2(SO_4)_3$] as eco-safe metallic chemical mordant on tannin pre-mordanted cotton fabrics subsequently dyed with a cationic or basic type of natural dye i.e the roots of barberry (*Berberis vulgaris* L.). Cotton was pre-mordanted with 10% (owf) gall oak at 80 °C for 90 minutes. The fabrics that had been pre-mordanted with gall oak were then re-mordanted with 6% aluminium sulfate and maintaining gall oak concentration at 20% and other remaining process parameters same. After mordanting, the cotton fabrics were dyed with varying amounts of barberry roots extract at 75 °C for 60 minutes with MLR 1:50. The colours present in the sample extracts were identified using a diode array detection method and high performance liquid chromatography. Cotton samples that had been naturally yellow-dyed were calorimetrically analysed, and their colour coordinates' L*, a*, b*, C*, h, K/S, and E* values were assessed. Results indicated that the dyeings without a mordant produced the lowest K/S value, whereas use of 100% root of the barberry plant produced the highest K/S value (2.53). Among all other techniques utilized, the non-mordanted cotton fabric dyed with barberry plant had the more +ve L* value (darker) in lightness/darkness scale.

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In the study conducted by Mongkholrattanasit et al. [36], dyeing of silk fabric with eucalyptus leaves extract containing quercetin, rutin, and tannin using various mordants by the pad-batch and pad-dry techniques. 5 g/L of ferrous sulfate was used as a mordant. Yellow quercetin, gallotannins, and small amounts of the brown epicatechin found in eucalyptus are the main colouring agents found in eucalyptus leaf The dyeing pH was maintain 4 using acetic acid. The cloth was soaked in the extract at ambient temperature, padded using the pad-dry process in a two-bowl padding mangle with 80% pick up, and then dried for 3-5 minutes at 90-100 °C. Another technique involved keeping padded cloth in roll form on a revolving drum with a plastic cover wrapped around it at room temperature for 12 to 24 hours rather than drying it after padding. Both samples were washed at 60 °C for 15 minutes using a non-ionic soap before being air-dried in the sun at room temperature. The results showed that the colour strength of silk fabrics dyed with eucalyptus leaf extract using the cold (room temperature) pad-batch method is superior to that of silk fabrics dyed using the pad-dry method. With the exception of silk fabric that had been pre-mordanted with tannin and dyed with eucalyptus extract, the findings for colour fastness to washing were very good, while those for rubbing fastness were fair to good. Wet rubbing fastness for the ferrous sulfate mordant was unsatisfactory. The light fastness rating of the ferrous sulfate-mordanted silk fabric was fair to good, but when quercetin-containing eucalyptus was employed, the results were poor and no mordant was used.

Hosseinnezhad et al. [37], reported green dyeing techniques and suggested use of bio-mordants to enhance the quality of dyeing. They used gall oak extract as a bio-mordant to dye wool yarns with weld luteola and madder as natural dyes. In this study, light, wash, and rubbing fastnesses, as well as K/S values and other colorimetric features of dyed wool yarns, were examined. Wool yarns were cleaned in a nonionic soap solution to improve surface wettability before being mordanted. Wool yarns that had previously been meta-mordanted with a solution of gall oak extract and natural alum in combination was dyed using MLR - 1:40 under varying dyeing conditions. Results revealed that using oak extract along with natural alum produced relatively higher K/S values. The C-N peaks in the ATR FT-IR spectra, which were used to assess the chemical bonds between the mordants and fibre disappeared indicating strong interaction between dye, fibre, and mordant molecules. Use of tannin based gallnut as bio mordant also enhanced the wash, light and rubbing fastness properties.

Zerin and Foisal [38] dyed scoured and bleached cellulosic fabrics with Acacia catechu using different mordanting techniques like pre- , post- and simultaneous. Alum and copper sulphate were used as chemical mordants. The colour characteristics i.e. reflectance percentage, K/S value, the effect of dyeing on colour fastness to wash and rub of cotton fabric dyed with catechu were analyzed. The extracted catechu was used to dye cotton fabric while maintaining a MLR - 1:40, temperature – 100 **°C** and time - 30 minutes. This was followed by washing the dyed fabric in cold water. A wide range of brown colours were obtained. Pre-mordanding with alum and copper sulfate as mordants produces deep shades. Post-mordanting, however, was found to improve colour fastness to washing and rubbing. Reflectance values were higher when no mordant was applied, while K/S value was higher for pre-mordanting process. When dyeing with catechu, alum and copper sulphate both produce results that are similar as evident from the values of reflectance %, K/S, and colour fastness in relevant cases were found to be very close to each other. Hence, future researchers need to extend this study to improve colour yield to obtain deep brown shade on cotton using bio-mordants.

Important Studies on Optimization of Dyeing Process Variables Using Eco-safe and Bio mordants

Sinnur et al. (33) used aqueous extract of *anar* peel to dye Khadi cotton fabrics using dual/double mordanting technique using potash alum and *hard*a (myrobolan), both from natural sources, as well as stannous chloride and aluminium sulfate (chemical mordants) and optimized extraction conditions, mordanting conditions and dyeing conditions. Findings indicate that mixture of *harda* and potash aluminium sulfate (50:50) provided the highest K/S value with good overall fastness than other combinations when applied at a 15% application level. The effects of varying dyeing process conditions on the colour yield and colour fastness were studied and optimized. The process variables/parameters were varied as time - 15-120 min, temperature - 50-95 **°C**, MLR - 1:10-1:50, pH - 3-13 common salt - 3-15% (owf) and pomegranate rind (as natural dye) aqueous extract - 10-50% (owf). The dyeing conditions for dyeing Khadi cotton fabrics with *anar* peel extract were optimized as follows - dye concentration - 20% (owf), pH - 9.0, MLR - 1:30, time - 60 minutes, temperature – 80 **°C** and common salt - 3%.

Ashrafi et al. [34], dyed silk and wool using myrobolan extract as natural mordant and almond dye. Fresh myrobalan was extracted at 50**°C** for15 minutes in water and residues were filtrated out to obtained clear dye solutions. Wool fabrics were dyed under acidic pH maintaining MLR - 1:40. The fabric dyed with bio-mordants, showed positive a* and negative b* values and thus was found to have a blue-red hue. The dyed textiles showed good wash fastness rating of 3-4 and poor light fastness ratings of 2-3.

Bio-mordants such as myrobalan extract, gallnut extract, cactus juice, and sumac extract can be used as an alternative/replacement to metal mordants as they enhance colour characteristics and colour fastness properties and their effectiveness depends on their tannin contents, chemical composition, concentration used [39,40].

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Concluding Summary

Recently processing and dyeing of natural fibre-based textiles with natural mordants (bio-mordants), natural colourants (bio-dyes), and natural finishing agents (bio-finishes) using eco-friendly mordanting, dyeing, and finishing procedures has attracted worldwide attention mainly due to their biodegradable and non-carcinogenic nature.

In order to revive the ancient process of natural coloration of textiles in a more modern sustainable manner, numerous researchers across the world are now working to standardize these natural mordanting, natural dyeing and natural finishing processes using advancement techniques that is less polluting and consumes less energy and less water. The aforesaid brief review paper highlights significant current challenges and advances in this sector with a view to path a way towards sustainability in textile wet processing.

Although the phrase "extraction methodology" is not new, there are presently a number of approaches being studied to extract natural mordants and natural colourants by using both traditional methods like boiling or hot water baths with or without additives, refluxing and sSoxhlet extraction, vacuum distillation and hydro-distillation. Various other newer techniques for extracting natural mordants or colourants by supercritical fluid extraction, pressurized liquid extraction, ultrasound/sonicator-assisted extraction, pulsed-electric field/magnetic field extraction, various radiation assisted extraction and enzyme-assisted extraction, among others, are gradually being explored, and some of them are found to be interesting and exciting over conventional methods due to shorter extraction time, higher colour yield and easy purification process for these natural colouring and finishing agents. Different pretreatment of plant resource materials prior to the extraction step are advantageous to enhance the colour build-up rate improving fixing and stability of these natural colorants and finishing agents.

Bio-mordanting standardized dyeing process variables can produce a wide range of reproducible eco-friendly textiles with shades of acceptable colorimetric parameters and acceptable colour fastness properties, along with or without some functional enhancement to broaden its uses as technical textiles (medical textiles). Future researchers need to explore and extend these studies based on use of tannin-based natural mordants (like harda), colourants such as *anar* peel, babul bark, mango bark, catechu, eucalyptus leaves, and neem leaves, as well as newer combinations of bio-mordants, bio-dye, and bio finishes with or without pre-treatments with enzymes, cationization with nature-derived chitosans with or without antibacterial or UV protective natural agents.

It is clear from this review that some gaps exist and industrial and scientific studies on the effects of various bio-mordants (natural compounds based on tannin), bio-cationization (with chitosan), and mordanting assitants (natural compounds based on gallic acid or chebulinic acid), standardization /optimization of dyeing process conditions as well as the effect of various after-treatment compounds like source of dye fixatives/UV absorbers and antibacterial agents on improvement of colour fastness to wash, sun-light/UV light and rubbing may be undertaken. Additionally, there is a need for methods to enhance the antimicrobial and UV-protective functionality of such naturally dyed and finished textiles by using specific concurrent natural dyeing and natural finishing treatments or by selective post-treatments by pad-dry -cure with various naturally resource-based eco-safe corresponding finishing methods.

It is important to remember that natural colours cannot replace synthetic dyes. They have their own market, thus any expansion of the market for naturally coloured products won't lead to a decline in the demand for fabrics with synthetic colours.

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