Chapter

A Review on Application of Natural Dyes on Textile Fabrics and Its Revival Strategy

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Abstract

A comprehensive review on application of natural dyes on textiles and earlier research findings has been discussed in this chapter. Moreover, recently the consumers have become very much conscious about the environment, renaissance of eco-friendly products and process like dyeing textiles with natural dyes, which has thus become also important now. Thus, revival of natural dye application on textiles and summary of earlier researches on standardization of its method of extraction, mordanting, dyeing process variables and even natural finishing, etc. have been elaborated in this review. Characterization of natural dyes and chemistry of its dyeing, etc. are equally important and hence are discussed here critically. Thus this part has become a unique readymade comprehensive chapter for information on chemistry and application of natural dyes on textiles and its revival strategy.

Keywords: natural dyes, natural pigments, extraction, mordanting, natural dyeing methods, characterization of natural dyes, revival of natural dyeing and textiles

1. Introduction

Natural dyes are known to be used since historic times for coloring food substrate, leather, as well as common textile fibers like cotton, wool and silk. However due to the advent of synthetic dyes and their good fastness properties in comparison to natural dyes, the use of natural dyes have suffered drastically. In the present scenario there has been a rise in concern of eco-friendliness and sustainability of the products used by the consumers for which natural dyes are again starting to experience slight rise in popularity. A study has been conducted by Samanta and Agarwal [1] which reports the characterization as well as chemical/biochemical analysis of various natural dyes available, the different types of mordants as well as different mordanting techniques, the different conventional and non-conventional method of natural dyeing of textiles. The different natural dyes used for the study are madder, henna, held, indigo and others such as annatto pulp, *Rubia tinctorum*. Different methods of extraction are employed such as aqueous extraction, non-aqueous method as well as by acid and alkali. Different types of mordant and method of mordanting significantly affect the rate of fading. For cotton the best mordant combinations used in this study are harda and tartaric acid, followed by tannic acid and harda. Double mordanting is employed by using harda and aluminum sulphate. The various process variables to be considered for dyeing with and extraction of

natural dyes are concentrations of dye source material, extraction time, dyeing time, mordant concentration, pH and concentration of salt used.

Another study conducted by Daberao et al. [2] gives us a concept about dyeing with palash or tesu flower petal (*Butea monosperma*) *as* natural dye sources. The dyes were extracted from *Butea monosperma* or in other terms flame of the forest and they were applied on 100% cotton. A different method of extraction by boiling was employed and alum was used as mordant. The fabric was then tested for all color fastness tests. The cotton sample was scoured and bleached for better color uptake. The washing fastness results were observed where the natural dye having not too much affinity with the fiber but by the application of mordant could withstand at least five washes. Also wet rubbing fastness of the dye was found to be poorer in the experimental results. However it was observed that *Butea monosperma* has good perspiration fastness since it is unreactive to acidic and alkaline perspiration.

The use of natural dyes has further started to be increased substantially over the current years for its slow but growing revival phase at present, due to people's concern over reducing environmental pollution and hence to avoid chemically more hazardous synthetic dyes and intermediates. Day by day in export market, demands for natural dyed natural textiles are being increased. Different institutions/organizations and Govt. have started multifold revival strategies for increasing the use of natural dyes as not only as an employment opportunity for several NGOs, weaver and dyers society, designers, industries, small scale cottage industries, etc. but mainly for adopting green technology dyeing. The handicraft industry in India uses local talents to dye yarns and fabrics with natural compounds, where several products are famous worldwide like Kalamkari print. Different countries other than India like Turkey, Korea, Mexico, several countries of Africa have embraced the uses of natural dyes. A study has been conducted by Gulrajni [3] to understand the scope of natural dyes and its present status in the world in addition to different application techniques, extraction of different natural dyes as well as varying mordanting techniques. Different problems associated with such natural dyeing are also highlighted there.

The Tharu tribes of the Devipatan [4] division have found a new source of natural dyeing from the local leaves and stems of *Jatropha curcas L.* The dyes are extracted by simple boiling the leaves in water and then evaporating the extract to dryness. The extract obtained is of yellowish olive syrupy color and when applied to the cotton fabrics the different shades of tan and brown color are obtained.

Another state in our country, Manipur has been considered to be a source of a natural dye namely extracts from *Strobilanthus flaccidifolius* for uses in handicrafts, handlooms, fine arts, etc. Other tribes of Manipur like the Meitei community have been using species like *Parkia javanica*, *Melastoma malabathricum*, *Pasania pachyphylla*, *Solanum incidum*, *Bixa orellana*, *Tectona grandis*, etc. these plants are combined with other plants for extraction and then dye is prepared by indigenous sources. This study is a report by Potsangbam et al. [5] of the dyes extracted from the above sources, the method of extraction as well as their application.

Now from the forest of Chhattisgarh, different dye yielding plants have been identified and collected. A study was conducted by Tiwari and Bharat [6] on the diversity of dye-yielding plants of Chhattisgarh, the indigenous method of dye extraction and ethnic uses of dyes. These colors are being used by tribal folks of this region for different purposes such as ornamentation, cosmetics, decorating houses and coloring home utensils made up of mud.

From the state of Goa [7] natural dye-yielding plants like *Cassia fistula*, *Garcinia indica*, *Tectona grandis* are obtained and studied where Goa is said to house more than 3000 different species of flowering plants. Natural dyes were extracted from

various parts of plant like fruits, seeds, bark, flowers, roots, etc. The extraction processes are studied and dyes of different shades are obtained. This study can also encourage the small scale industries to use natural dyes from these sources to be applied on cotton and silk fabrics.

A report by Gaur [8] shows the extensive description of survey, collection of botanical information and review of relevant literature on the vegetable dye yielding resources of Uttarakhand Himalayas. Of which, very little known dye yielding plants are considered like *Acacia nilotica*, *Agrimonia pilosa*, *Careya arboraea*, *Averrhoa carambola*, etc. Different extraction processes for each plant are carried out and subsequently dyeing is done by using different mordants. Some of these plants also have high medicinal values and has no toxicity.

1.1 Sources of different natural dyes and their characterization

Various natural products are being used for dyeing these days in order to fulfill the demand of consumers for sustainable environment. The reviews below are for the various natural dyes used on textile materials.

Bechtold et al. [9] have studied on the quality of Canadian golden rod plant material as a natural dye. Aqueous solutions of the material containing the extracted flavonoid dyes were characterized by means of direct photometry, absorbance after addition of $FeCl_2$ is measured, total phenolics (TPH) in the extract and dyeing on wool yarn are analyzed where only relatively small differences in color depth and shade were noted amongst the major parts of the different materials collected.

A study on natural dye henna was conducted by Rahman Bhuiyan et al. [10]. Henna is a red-orange pigment that has long been used for the coloration of skin and hair as well as textile materials. A large number of studies were carried out on extraction as well as application of henna dye in textile fibers and the standardization and simplification of dyeing techniques were determined. Due to burgeoning environmental conditions and growing awareness on sustainability there has been a renewed interest in expanding the scope and applications in the coloration of textile fibers with some successes and promises. Henna shows an acidic nature due to the presence of polar groups, which promotes its use in the textile dyeing process.

Dyeing of natural dye extracted from *Liriope platyphylla* fruit on silk fabrics have been studied by Huang et al. [11]. From which it has been observed that the total phenolic content (1109.13 ± 69.02 mg), total flavonoid content (530.60 ± 89.44 mg), and total anthocyanin content (492.26 ± 77.79 mg) were measured in 100 g fresh weight of *L. platyphylla* fruits. A broad variation in color shade and color depth has been achieved with mixtures of different combinations of dye extracts and metal mordants. Purple, blue, and pale green were main color shades obtained when dyed with the extracts. The fastness of dyed silk fabrics against light, washing, and rubbing were noted to be acceptable with at least a gray scale rating of 3.

A study has been conducted on orange peel by Hou et al. [12]. Orange peel is an easily available agricultural byproduct and it is cheap as well as abundant. The variation in effects of dyeing methods and conditions, including pH value, temperature, time and concentration of OP extracts on the colors of the dyed wool fabrics, were studied. Eco-friendly mordants of aluminum and iron were used. The optimum dyeing conditions were noted which included dyeing temperature of 100°C, dyeing time of 120 min, pH of 3 for direct dyeing and pH 7–9 for one-bath mordant dyeing. Good colourfastness to washing with soap, good colourfastness to rubbing and acceptable colourfastness to light were displayed by the tested specimen.

Hibiscus is a major source material for natural dyeing. It belongs to the family Malvaceae. Aqueous extracts of these flowers have shown good fastness properties according to the study conducted by Shanker and Vankar [13]. The dye has been

found to have a good scope in the commercial dyeing of cotton, silk for garment industry and wool yarn for carpet industry. In the present study dyeing with hibiscus has been shown to give good dyeing results. The material is pretreated with 2–4% metal mordants, keeping M:L ratio as 1:40 on weight of the fabric to plant extract. The dye is cheap and has good commercial value if dyed with cotton, wool and silk.

Another natural material has been found by Vankar et al. [14] to be a good source of natural dyeing which is *Mahonia napaulensis DC*., common name taming, from the family Berberidaceae. The natural dye is from the stem and has been used by the tribes of Arunachal Pradesh. The fastness properties for dyed cotton, silk fabrics and wool yarn were show to increase substantially when pretreated with metal mordant (2% w/w with respect to the fabric).

An attempt has been made by Kamel et al. [15] in dyeing of wool fabrics using lac as a natural dye in both conventional and ultrasonic techniques. The dye extraction was compared between conventional method and ultrasonic technique and the data were evaluated. Accordingly the effects of dye bath pH, salt concentration, ultrasonic power, dyeing time and temperature were compared. The result of fastness properties obtained was fair to good.

Montazer and Parvinzadeh [16] have dyed wool with marigold as a source of yellow color. At first the wool yarns were premordanted with alum, dyed with marigold and then treated with different percentages of ammonia solutions. After washing with standard soap after color hue alters and there has been no effect of ammonia after treatment on washing fastness however the samples show lower light fastness.

A study on the dyeing properties of woolen yarns using gallnut extract as a natural dye was conducted by Shahid et al. [17]. A conclusion that gallnut extract can be applied on woolen yarn with or without mordants to produce bright ivory to light brownish yellow color with good fastness properties against light, washing and rubbing was obtained from the test.

Natural dyes have been slowly garnering popularity all over the world. So a study was carried out by Mirjalili et al. [18] by extraction of dyes from weld using soxhlet apparatus. The natural dyes were extracted and isolated and the colored substance obtained was used for dyeing of wool fiber. Finally a comparison was made with the synthetic colorants on the color fastness tests. It can be concluded from the study that weld can be used as a non-toxic dye. Good fastness properties were obtained from this natural extract.

An attempt has been made to dye the wool fabric with Limoniastrum monopetalum stems by Bouzidi et al. [19]. Extraction parameters were optimized. The optimization of extraction results obtained were dye concentration of 60 g/l, a temperature of 90°C and time duration of 100 min. The best results were obtained of pH 2, dyeing temperature of 100°C, and time duration of 60 min. Metal mordants were used in this process. The extract has ample natural tannin and polyphenol compounds which are considered as mordants since they have the ability to fix the dyes in bath to the fabric.

Indigo carmine is another renewable resource based blue dye which can be used to color protein fibers. Komboonchoo and Bechtold [20] have worked on use indigo carmine in combination with other natural dyes in a one-bath procedure as a hybrid dyeing concept. Optimum dyeing parameters of pH in the range of 4–5 and temperature between 40 and 60°C were obtained.

A new concept of few natural dyes as dye sensitized solar cell (DSC) was brought forth by Hao et al. [21–24]. Amongst all these photochromatic natural dye-extracts, black rice extract dye shows best results, perhaps due to high interaction between carbonyl [—C═O] and hydroxyl [—OH] groups of anthocyanin present

in such dyes. Because of the simple preparation technique, these are considered as widely available and low/cheap cost natural dyes as photo sensitized color of natural dyes, having photo-sensitized solar cell type character. Other materials like achiote seeds, rosella, blue pea flowers, spinach and ipomoea were also reported for such natural dyes having in built photo-sensitized solar cell in it.

Cochineal is an insect species of scientific name *Dactylopius coccus.* Carminic acid is the natural colorant obtained from the dried female body of such insects. This finds application in cosmetics, foods, pharmaceutical sectors as well as textile and plastic industries. The study has been conducted by Borges et al. [25] and the study on the newer process of extraction will be discussed in the extraction methods section.

A study of natural eco-friendly dye extracted from *Plumeria rubra* is carried out by Vettumperumal et al. [26]. Due to the existence of highly delocalized systems absorption spectrum shows a broad absorption in the range of 292–590 nm. This plant also encourages usage of waste lands, afforestation of wasteland and provides consequent additional source of income to rural population.

Rubia tinctorum is commonly known as madder produces anthraquinone pigments in its roots, one of them being alizarin (1,2 dihydroxy anthraquinone) which has been used for dyeing textiles since ancient times. Angelini et al. [27] has evaluated four madder genotypes for their agronomic characteristics as well as for their industrial value and to assess its value as a new industrial dye crop. Industrial assays demonstrated good performance when using dry powder 30% of the weight of material to be dyed for dyeing cotton, wool and silk yarns. Resistance to fading appears to be fairly good for dyed wool when using madder.

Different coloring plants from New Caledonia were considered for research by Toussirot et al. [28] amongst which *Hubera nitidissima*, an Annonaceae, showed an intense yellow color on fibers. Color was extracted from the leaves of the said plant on linen, silk and wool. The color fastness results were obtained where it was concluded that *H. nitidissima* appears as an excellent source of light-fast yellow dye with interesting antioxidant properties. These days natural dye extracted from mangrove bark was also used as a dyeing material.

1.2 Application of natural dyes on different textiles

Punrattanasin et al. [29] applied selectively extracted few natural dyes to a silk fabric by an exhaustion dyeing process where aluminum potassium sulfate, ferrous sulfate, copper sulfate, and stannous chloride were used as mordants. Dyeing was carried out in three different stages of the fabric-premordanted, meta-mordanting and post-mordanting. Color fastness values of each were reported. Dyeing conditions were optimized as dyeing temperature of 90°C, dyeing time—60 min and dye bath pH of 3 was fixed to be optimum. In this work, natural silk textiles were dyed with and without mordants using $SnCl_2$, KAl SO_4, $FeSO_4$ and $CuSO_4$ providing varying degree of color/tone/shade, where $FeSO_4$ produced darker and blackish brown shade, $CuSO_4$ produced lighter to pale reddish brown shade, both showing poorer washing fastness but very good water soaking, perspiration, light and rubbing fastness.

The various physical tests were done and tensile strength, tearing strength and stiffness of the fabrics before and after dyeing were also compared.

Shukla et al. [30] has reported dyeing of woolen textiles with extract of *acacia pennata* plants. The color was extracted from barks of the said plant and applied on wool. *Acacia pennata* is a thorny shrub found throughout India and Burma. Experiments were carried out where acacia pennata was used in conjunction with banana stem. When compared without banana stem it was observed that dye

fastness without the banana stem was poorer than when stem was used. It was concluded that banana stem acted as a good mordant thus eliminating the use of metallic, carcinogenic mordants.

An attempt has been made by Gulrajni et al. [31] to dye nylon and polyester with annatto. Annatto also known as *Bixa orellana* has a color component namely carotenoid dye bixin. It has been observed that both of these fibers show good affinity for this dye but moderate fastness to washing and poor light fastness.

An attempt has been carried out by Gulrajni et al. [32] to extract dyes from ratanjot also known as *Arnebia nobilis* for application on cotton, wool, silk, nylon, polyester and acrylic. The process conditions such as pH and temperature were recorded. It has been noted that dye exhibits acute sensitivity to pH in terms of solubility and color and is found to be thermally stable upto 80°C. The different colors shown by various fabrics were noted such as pink color for polyester, blue for nylon and other substrates acquiring a purple hue under similar dyeing conditions.

A study by Gulrajni et al. [33] on the kinetics and thermodynamics of dye extracted from *Arnebia nobilis* on woolen textiles was reported. Physicochemical and dyeing kinetics parameters of this natural dyeing using aqueous extract of *Arnebia nobilis* applied on woolen textiles were reported as compared to the same for other natural colorants like juglone, lawsone and *Rheum emodi*, etc. The results showed here that anthraquinonoid based these natural colors do not form desired coordinated complex with wool and rather are absorbed on wool substrate by partition mechanism following Nernst isotherm like absorption of disperse dye on polyester.

Chakraborty and Chavan [34] reviewed on dyeing of cotton denim with Indigo, which gives the information on the newer application techniques of indigo dyes applicable for natural indigo. Since indigo has negative affinity for cotton conventional methods cannot be applied. The details of indigo reduction, solubilization and dye application has been studied in this reference.

Deo et al. [35] had attempted dyeing of ecru denim with onion extract as natural color using Potash-alum in combination with harda and tartaric acid as mordants. Any of the single mordant did not produced desired shade. Amongst combined mordants used, Potash-alum + harda combination was found to be better than potash-alum + tartaric acid for producing desired depth of shade, but potash alum + tartaric acid (5%:5%, that is, 1:1 combination of each 5% application) post mordanting showed best overall color fastness results.

A study has been carried out by Samanta et al. [36] on standardizing dyeing process variables for its application on bleached jute fabric with aqueous extract of tesu (Palash flower petal). It is observed that higher amount of pre-mordanting with 20% myrobolan (Harda containing chebulinic acid) followed by 20% aluminum sulphate in sequence and dyeing at pH −11.0 produced optimum color yield and all round good color fastness. Improvement in wash and light fastness was also achieved with suitable chemical post-treatment using suitable agents.

Gray jute fabric bleached with hydrogen peroxide in conventional method was mordanted with different concentrations of ferrous sulphate and dyed separately with natural dyes extracted from deodara leaf (*Cedrus deodara* L.), jackfruit leaf (*Artocarpus integrifolia* L.) and eucalyptus leaf (*Eucalyptus globulus* L.). Pan et al. [37] have observed the interdependency of color yield and color fastness properties on dosages, that is, concentrations of mordants ($FeSO_4$) used, higher iron-mordant concentration lead to higher color yield, darker color and better overall good color fastness. But they have not studied loss of strength of due to mordanting and which is essentially needed to be assessed also.

Narayana Swamy et al. [38] have studied the use of *madhuca longfolia* as a dye source. The dried leaves of the said plant are taken as dye source for silk dyeing. The optimum conditions under which the dye has been extracted are pH of 10,

time (60 min) and temperature (95°C). Varying range of shades is obtained using different methods with or without using mordants. The dyed samples have been evaluated for color measurements and standard wash, light and rub fastness tests. Eco-friendliness of the dye has been kept into account. The dyed samples are also tested for antimicrobial activity against Gram-positive and Gram-negative bacteria. The dyed silk fabrics show acceptable fastness properties and the results show that *Madhuca longifolia* leaves are promising as a natural colorant, which can thus open new doors towards environment friendly products.

An attempt has been made to color silk using barberry, a cationic type natural dye by Pruthi et al. [39] Barberry bark also known as *Berberis aristata* DC. was used for dyeing of degummed pure silk yarn using four selected mordants; alum, chrome, copper sulphate and ferrous sulphate in different ratio, that is, 1:1, 1:3 and 3:1. Optimized results was obtained for aqueous extraction of barberry as 60 min time, 8% dye source material and optimized dyeing conditions were observed to be pH-of dye bath-4.0, dyeing time-45 min for standard mordanting with chrome + ferrous sulphate (1:3) and chrome + copper sulphate (3:1) produced the higher degree of color fastness properties. Varying shade percentages and color tone were obtained by using varying degree/percentages of different combination of mordants.

Das et al. [40] have worked on the application of *Bixa orellana* on protein textiles viz. on wool and silk. Seeds of annatto have been extracted first and then have been employed on silk and wool in absence and presence of magnesium sulphate, aluminum sulphate and ferrous sulphate. Effective colouration has been achieved at pH 4.5 commonly in the absence and presence of such inorganic salts. Color uptake for wool is found to be more than that for silk under all the conditions studied. When both the substrates are treated with such salt prior to application of annatto there has been significant increase in color uptake. Colored protein fibers, in general, produce light and wash fastness ratings of 2–3. Ferrous sulphate in turn, improves color fastness properties and color retention on washing of wool and silk fibers.

Another study has been conducted by Das et al. [41] on the application of *Punica granatum* on wool and silk textiles. *Punica granatum* also commonly known as pomegranate rind was used on wool and silk fabric in the presence and absence of environment-friendly mordanting agents. Both the dyeing of silk and wool with pomegranate solution is found to be effectively accomplished at pH 4.0. With the application of ferrous sulphate and aluminum sulphate during pre- and post-mordanting has shown improvement in the color uptake, light fastness and color retention on repeated washing. The use of such mordants, however, does not show any improvement in wash fastness property of dyed substrates.

An application of *Terminalia bellerica* fruit extract dyeing on woolen textile under different conditions of pH, concentrations of natural coloring matter, time of extraction/durations and temperatures were studied by El-Zawahry and Kamel [42]. The results evaluated were mainly surface color strength and color depth. The study shows that optimum color yield was obtained using following extraction and dyeing conditions:

Extraction: source coloring matter—5%, temperature of extraction bath—near boil, that is, 90–100°C at pH—7 (neutral).

Mordanting: (i) potassium dichromate + lactic acid-application 0.5 gpl, and (ii) chromic chloride + lactic acid-application −0.5 gpl,

Dyeing: time—60 min and temperature—near boil (95°C),

Shade obtained: moss green with mordanting system (i) as above, mustard yellow with mordant system (ii) as above and muster brown with both copper acetate and ferrous sulphate or ferric chloride as mordant. Thus for such natural dyes-color tone and shade depth are much dependant on type of mordant and its concentration

used. Overall color fastness results to washing, to acid or alkaline human perspirations and rubbing/crocking fastness were found to be almost the same for said premordanted and dyed wool fabrics. In case of light fastness, longer the duration of exposure to light, darker the shade and better light fastness were obtained. There has been no change of color strength and fastness properties despite of use of standing dye bath (50 g T.b. fruits/100 ml water) for 8 times.

A new approach of dyeing was undertaken by Naz et al. [43] where Eucalyptus (*Eucalyptus camaldulensis*) bark powder (without any further treatment/irradiation) using gamma ray irradiated natural colorant of dry powder of eucalyptus leaf extract, for producing natural colored textiles of soothing brown color with improved color fastness by required pre and/or post mordanting. Thus, when this fabric was therefore dyed in this case using gamma ray irradiated powder of eucalyptus dry leaf, it showed noticeable improved overall color fastness properties.

A paper was presented by Ferda Eser et al. [44] on dyeing of polyester and polyester/viscose blends dyed with walnut shell extracts. Different extraction conditions were considered such as material—liquor (M:L) ratio, extraction temperature, extraction time and pH in order to obtain highest color depth. Optimal extraction of natural dyes from walnut shells (*Juglans regia*) was obtained at temperature—80°C, time of extraction—75 min using MLR as 1:30 at pH 2. For dyeing polyester and polyester/viscose blends with said extract of wall nut shell using $AlKHSO_4$ or $AlK(SO_4)_2$ or $FeSO_4$ for separate mordanting for 90 min time and subsequent dyeing was studied and found that pre mordanting with $FeSO_4$ offers best dyeing results with good color depth and overall good color fastness, which can be used for future applications for ecofriendly dyeing of polyester and its blended textiles.

A detailed study was carried out by Samanta and Agarwal [45, 46] on dyeing of jute and cotton fabrics with binary mixtures of jackfruit wood along with other natural dyes in combination for producing compound shades after study of their compatibility. Conventionally hydrogen peroxide bleached jute and cotton fabrics were taken and was pre-mordanted with 10–20% harda (myrobolan) followed by 10–20% $Al_2(SO_4)_3$ or $FeSO_4$ salt in sequence as sequential double mordanting as a most prospective mordanting system for subsequent dyeing with aqueous extract of jack fruit wood. Study of dyeing process variables showed that optimum dyeing results were obtained for 90 min dyeing time, 70–90°C dyeing temperature, 11.0 pH, 1:30 material-to-liquor ratio, 20–30% mordants concentration, 30–40% source dye concentration, and 15 gpl common salt. In conventional method, for test of compatibility of these selected binary pairs of natural dyes, in order to obtain progressive depth of shade, two sets of five different samples were produced and tested after dyeing with 1:1 mixture of two dyes at 1% fixed shade depth with varying time and temperature profile in one set as well as by varying total concentrations of the binary pairs of dyes (using varying shade depth with 1:1 equal proportion of mixture of two dyes) keeping time and temperature fixed for second set were obtained and their color parameters of K/S vs. DL and DC Vs. DL were compared to judge compatibility by graphical comparison method. However, in this work, a newer method of compatibility rating procedure with calculation of Color Difference Index data (a newly defined useful color difference parameter) was described and adopted here for easy determination of compatibility rating between two dyes of any binary pairs of selective natural dyes used for applying that binary mixture of natural dyes in the same dye bath for compound shade. Moreover, they have shown methods of improving color fastness to washing by using separate post treatment with cationic agents like CTAB (n-cetyl-N-trimethyl ammonium bromide), or cetrimide, etc. Similarly separate post treatment with 1% benztriozale as an UV absorber had also shown an improvement in light fastness results.

Another attempt of dyeing ratanjot on nylon and polyster was studied by Gulrajni et al. [47] where the observed results indicated that this dye has a good substantivity for both nylon and polyester fibers, probably due to less polar structure of this dye and Nernst partition isotherm of absorption of this dye on these two fibers. However, deep color shade and better fastness to light and washing was obtained.

Sagarika Devi et al. [48] have studied about *Alternaria alternata* for textile dyeing and printing where reddish brown natural pigments which was obtained after extraction of colors from dry mycelium of *Alternaria alternata* in methanol solvent media. At pH −6, this Fungus produce the said extractable colored pigment, which can be applied on cotton for light color with medium grades of color fastness results using pigment dyeing process. This natural color is antibacterial and antifungal as evidenced in this work by AATCC100 test method for both gram positive and gram negative bacteria species for test, showing its antimicrobial nature.

Ke [49] studied the dyeing properties of natural dye extracted from *Rhizoma coptidis* on acrylic fibers. Acrylic fiber was dyed with *Rhizoma coptidis* aqueous solution and its dyeability was studied in terms of the thermodynamic and kinetic properties and dyeing process conditions. This study showed that effect of dyeing temperature is positive, that is, color yield and dye diffusion rate increases with increase of dyeing temperature up to a limit, indicating dyeing temperature and mordant concentration as important critical variables in such dyeing of acrylic with extract of *Rhizoma coptidis*. Color fastness to washing and rubbing are found to be grade—4.

Haque et al. [50] extracted ubiadin dye from *Swietenia mahagoni* and studied its dyeing characteristics onto silk fabric using metallic mordants. Metallic mordants such as $MgCl_2$ and $FeSO_4$ were used and dyeing properties were evaluated. $FeSO_4$ as compared to that of $MgCl_2$ showed good result for color yield and color fastness results.

Mahale et al. [51] studied about natural dyeing of Silk yarn skeins using extract of *Acalypha wilkesiana* leaves using varying concentrations of mordants like potash alum, potassium dichromate, copper sulphate and ferrous sulphate. Potassium dichromate and copper sulphate are not ecofriendly mordant. Potash alum though gives good fastness but considering color yield and fastness both, $FeSO_4$ offers best results of color yield and color fastness.

A study was conducted by Poorniammal et al. [52] for natural dyeing with extracted and purified natural fungal pigment from *Thermomyces* sp. to apply on different textile fabrics to optimize and dyeing process parameters for silk, cotton and woolen fabrics. This extracted pigment color obtained from *Thermomyces* sp. indicated good affinity towards silk fabrics than others, with good light fastness (rating 4), color fastness to washing (rating 4–5) and color fastness to rubbing (rating 3–4). The optimum conditions for dyeing was found to be dyeing temperature—30°C, dyeing pH—3, myrobalan mordant—5%, and dyeing time—20 min duration were suggested. The pigment also gave a reasonable extent of bacteria reduction in such silk dyed sample against *Salmonella typhi* (51.05%).

An attempt is made by Onial et al. [53] on utilization of *Terminalia chebula* Retz. fruits pericarp as a source of natural dye for textile applications. *Terminalia chebula* Retz. of Family-Combretaceae, trade name-Myrobalan fruits pericarp powder was taken for the utilization as a dye. The dried fruits constitute one of the most important vegetable tanning materials and have been used in India for a long time. This fruit pericarp thus can be used as a raw material for natural dyeing.

Das et al. [54] made an attempt to dye wool and silk with *Rheum emodi*. Silk and woolen fabrics, which were dyed with colorant extracted from *Rheum emodi* in the absence and presence of metallic mordants such as magnesium sulphate, aluminum sulphate and ferrous sulphate for producing shades of different colors, ranging from yellow to olive green. Study of dyeing isotherms and kinetics of dyeing process indicated that this dyeing mechanism do not follow coordinated complex formation

amongst fiber-mordant-dye, rather follow Nernst type isotherm showing pattern of partition mechanism, for this anthraquinonoid-based colorant where the dye molecules are adsorbed by silk and woolen fabrics as a disperse dye.

However, rate of dyeing is found to be higher for silk than that of wool and that color depth is increased by use of both aluminum sulphate or ferrous sulphate as mordant and considering color fastness test results, the later, that is, ferrous sulphate as mordant is found to be superior (offering wash fastness grade as 3 to 4 or 4) than use of same dosages of aluminum sulphate.

Thus ferrous sulphate is preferred as mordant for obtaining an improvement in the color fastness properties and color retention on washing of both wool and silk fabrics further.

Goodarzian and Ekrami [55] used brown dry rind of pomegranate as dyestuff. Extraction was carried out by solvent extraction method. Woolen fabrics were dyed with both raw and extracted dyestuffs using variations of concentrations. Spectrophotometric evaluations as well as colorimetric studies were carried out to compare the color strength of raw and extracted dye stuff on woolen fabric. It was concluded that color strength of extracted dye from pomegranate rind was more than raw dye stuff.

As mentioned by Garfield and Mauve [56], 'Mauviene' was the first synthetic dye synthesized by William Perkin in 1856. Like every scientific invention with man-made materials, advantages and disadvantages coexist, and the synthesis of synthetic-dyes is no exception. With the present growing global concern for environment protection and use of eco-friendly and bio-degradable materials, the trend of application of natural dyes have once again gained the momentum and for growing concern of consumers on eco friendliness of textile products, application of natural dyes on textiles is slowly being revived again.

Advantages of natural dyes over synthetic are manifolds [57] as they are eco-friendly, safe for body contact and are harmonized as reported by Brian [58]. Many scientists have also suggested and reported the medicinal and antibacterial importance of natural dyes [59, 60]. Yellow dye from rhizome of turmeric has been reported to be traditionally used in medicine as an anti-inflammatory drug [61]. Most of the natural dyes are proved to be non-toxic and eco-friendly, although there are some exceptions.

The natural dyes are the colorants extracted from the vegetables matters, minerals or insects [62]. Although most of the natural dyes have poor to moderate light fastness and the synthetic dyes represent a full range of colors with light fastness properties ranging from moderate to excellent [63], the use of natural dyes on textiles have been reported by many scientists. Dyeing of cotton with leaf-extract of Beilschmiedia fagifolia was reported by Vankar et al. [64], who has used sonicator method to dye cotton with aqueous extracts of *B. fagifolia*. The authors have reported that pre-treatment of cotton with 1–2% metal mordant and dyeing with 5% plant extract produced optimum results with good fastness properties.

In another report, Shah and Datta [65] used floral dye extracted from marigold flower to dye cotton fabrics. Gahlot et al. [66] used colorants extracted from Jatropha integarrima flowers for dyeing of cotton, wool and silk. Dyeing of silk with Onosma echiodes (Goldendrop) was reported by Sidhu and Grewal [67]. Mahale et al. [68] dyed cotton with Arecanut palm extract. Ultrasonic dyeing of cotton and silk with *Nerium oleander* flower has also been carried out [69]. Purohit et al. [70] reported use of natural color from waste leaves of Arotocarpus beterophyllus on different textile substrates like cotton and silk to get standard reproducible shades of golden yellow color.

The application of natural dyes such as turmeric, madder, catechu, Indian rhubarb, henna, and tea and pomegranate rind on manmade fiber nylon has been reported by

Teli et al. [71]. Some studies have also been conducted on application of Lac dyes [72] on different fibers. Application of a natural dye, annatto, on mulberry silk was carried out by Javali et al. [73]. Some studies [74–76] on natural dyeing of silk textiles have been reported in literature for use of Indian madder, *Spathodea campanulata* and lac dyes as natural sources. Patel et al. [77], have reported environmental-friendly and cost-effective method to create various shades on silk with few natural dyes.

There are many historic books documenting the literature on the use of natural dyes or natural dyed materials (textiles, candles, food, furrs, etc.) dating to as far back as the eighteenth century. The significant literary document on natural coloring matter was made available for the first time by Perkin and Everest [78].

Sahid and Mohammad [79], and Mayer and Cook [80] have also reviewed details of chemistry asnd application of natural dyes and more recent report on the structures of quinonoids natural colorants is described in Thomson's book [81]. Recent reviews in this area also include work undertaken by Parris [82] and Hofenk de Graaff [83] the latter includes information on fastness properties and history of use. Studies in the analysis of natural colorants in textiles are a fascinating subject which started as early as 1930s.

Recently, Samanta et al. [84–86] have described thermodynamic analysis of rate of dyeing, half dyeing time, enthalpy, free energy, etc. as a physico-chemical parameter of dyeing jute with red sandal wood, jackfruit wood and tesu as natural dyes.

The analysis of mass spectrometry of textile fibers dyed with indigo has been reported by McGovern [87]. However, Wong [88] was not able to detect 6,6-dibromoindigotin by direct analysis, but only after it had been separated by reductive extraction with sodium hydrosulphite.

Thin layer chromatography (TLC) was used by many workers to identify natural dyes in textiles [82]. Dyes detected were insect dyes and vegetable dyes viz., yellow, red and blue colors. Koren [89] also analyzed the madder and indigoid dyes by HPLC. Guinot et al. [90] also used TLC chromatography analysis to carry out a preliminary evaluation of plants containing flavonoids (flavonols, flavones, flavanones, chalcones/aurones, anthocynanins), hydroxycinnamic acids, tannins and anthraquinones, which are the phylo-compounds (color compounds) found in the plants.

Physicochemical dyeing parameters of red sandal wood as natural dyes and its compatibility with other dyes were analyzed by Samanta and Agarwal et al. [91, 92]. Neem bark [93] colorant showed two absorption maxima at 275 and 374 μm; while beet sugar showed three absorption bands at 220, 280 and 530 μm as per study undertaken by Mathur [93]. The visible spectra of ratanjot [47] in methanol solution was observed at both acidic and alkaline pH by Gulrajani et al. [47]. *Gomphrena globosa* flower colorant showed one major peak at 533 μm. The dye did not show much difference in the visible spectrum at pH 4 and 7; however the peak shifted to 554 μm as reported by Shanker and Vankar [94].

Bhuyan [95] observed the amount of dye absorption for extract of *Mimusops elengi* and *Terminalia arjun* varies from 21.94 to 27.46% and 5.18 to 10.78%, respectively, for the said two dye sources. The color components isolated from most of the barks contain flavonoid moiety. Samanta et al. [96, 97] postulated a new index called color difference index (CDI) value which can be calculated by an empirical formula postulated by them and which made determining dye compatibility easier and simpler for a binary mixture, that is, between a pair of natural dyes.

Identification of dyes in historic textiles though chromatographic and spectrophotometric methods as well as by sensitive color reactions was highlighted by Blanc et al. [98], who studied the retention of carminic acid, indigotin, corcetin, gambogic acid, alizarin flavanoid, anthraquinone and purpurin, etc.

A non-destructive method was reported for identifying faded dyes on textiles fabrics through examination of their emission and excitation spectra. Zin and Moe [99] purified and characterized extracted natural agents and colors from mango bark for application in protein fibers like wool.

Walker and Needles [100] carried out the separation and identification of natural dyes from wool fibers using reverse phase HPLC using a C-18 column. Two quaternary solvent systems and one binary solvent system were reported to be used to obtain chromatograms of by the HPLC analysis of plant and insect based red anthroquinonoid and molluscan type blue and red purple indigoid dyes [89]. This method enables the elution process for the determination of different chemical functionality and class of dyes and significantly shortens the time of test. Son et al. [101] reported HPLC analysis of indigo highlighting the structural changes of indigo component, attributing a decrease/increase in color strength with variation of dyeing time.

Balakina [63] also investigated/analyzed the quantitative and qualitative analysis of red dyes such as alizarin, purpurin, carminic acid, etc. by HPLC. High Performance Liquid Chromatography (HPLC) has been also used by several workers to identify synthetic as well as natural dyes.

Jain and Vashanta [102] characterized antimicrobial activity after eco-friendly dyeing with arcea nut using natural mordant/mordanting additives like myrobolan, lodhra and pomegranate rind and found that pomegranate rind renders best antibacterial activity and Lodhar renders highest color fastness to wash amongst all the moderating additives used.

Mondhe and Rao [103] made an attempt to prepare azo-alkyd dyes by the reduction of nitro alkyds, followed by diazotization of amino alkyds and coupling with different phenol compounds present in *Jatropha curcas* seed oil by using IR spectra.

The toxicity [104, 105] data also provide evidence about the adverse effect to human and environment. Of primary concern are the acute toxicity, irritation effects on the skin and the eye and sensitization potential besides environmental pollution in the society. Furthermore, possible long-term effects such mutagenic, carcinogenic or reproductive toxicity is best judged by LD50 test. The crude methanolic extracts of stem and roots stem, leaves, fruit, seeds of *Artocarpus Hetrophyllus* [106] exhibited good rating of antibacterial activity. The butanol fractions of the same root bark and fruit were also found to be the most active.

Mishra and Patni [107] extracted tannins from gall leaf from oak plant (i.e., oak galls containing gallic acid and tannic acid and helps in better dye fixation) from Himalayan region and dyed cotton, woolen and silk textiles with different metallic mordants and obtained better color fast fabrics, which are skin friendly too. The main reason of revival of natural dyes for textiles are its environmental friendliness and skin friendliness too.

1.3 Natural dyes cum natural antimicrobial finishing agents

A study was conducted by Mari Selvam et al. [108] to investigate the antibacterial and antifungal effects of such dyed textiles dyed with Turmeric, Terminalli, Guava and Henna. The results obtained indicated that at a dose level of 50 μl of Terminalli dye was able to inhibit the growth of all the fungi tested. The absorbance rate of natural dyes was analyzed by UV Spectrophotometer. The absorbance rate obtained were high in Terminalli (2.266) and turmeric (2.255). Hence from this study it was concluded that natural dyes were bound with traditional products to give good color and good antimicrobial activity against isolated fungal pathogens.

Another study carried out by Rajni Singh et al. [109] on antimicrobial activity of some natural dyes like *Acacia catechu*, *Kerria lacca*, *Quercus infectoria*, *Rubia*

cordifolia and *Rumex maritimus*, which gives us an idea about to determine their minimum inhibitory concentration (MIC), which was found to be varying from 5 to 40 mg. So, such dyed textile material with these dyes must up take above MIC concentration for effective antimicrobial action in such natural dyed textiles.

Curcumin, a common natural dye used for fabric and food colorations was used by Han and Yang [110] to dye woolen fabric to obtain dyeing and antimicrobial finishing simultaneously showing relation amongst bacterial reduction percentage and dye (curcumin) concentration, and microbial inhibition rate and surface color strength (K/S value). However, durability of antimicrobial action for different nos. of washing cycle after laundering and after exposure to UV light/sun light are also very important criteria, which were also critically discussed in this work.

Shafat Ahmad Khan et al. [111] attempted a work to investigate the antimicrobial action of *Rheum emodi* L. as a potential antibacterial natural dye and they dyed wool yarns with extract of *Rheum emodi* L. as purified dye applying dye concentration of 5–10% with or without mordants like ferrous sulphate, stannous chloride and natural alum for subsequent antimicrobial test against *E. coli* and *S. aureus* following AATCC100 test method. Test results of such *Rheum emodi* natural dyed woolen yarn samples indicated 90% bacterial reduction percentage as well as very high fungal protection showing very effective antimicrobial properties.

Shahid-ul-Islam et al. [112] have studied the use of *Tectona grandis* L. leaves extract plant colorants for dyeing woolen fabrics for simultaneous dyeing and antimicrobial finishing with natural dye cum natural antimicrobial finishing agent. This study indicated that the dyeing woolen yarn with extract of *Tectona grandis* L. is suitable for dyeing cum multifunctional finishing to impart simultaneous dyeing and antioxidant and antibacterial finishing properties to woolen based textile fabrics.

Fatemeh Shahmoradi Ghaheh et al. [113] have found that pre-treatment with aluminum sulphate as pre mordanting and followed by subsequent dyeing with selective natural dyes extracted from green tea leaf, madder route, turmeric route, saffron petals, and henna as natural dye cum natural antimicrobial agents provide moderate to good antibacterial finishing property on woolen fabrics and also led to good durability of the said antimicrobial action even after five cycles of laundering and above 300 min exposure to UV light/sun light.

Mohd Ibrahim Khan et al. [114] have conducted a study on antimicrobial activity of catechu itself and catechu extract dyed woolen yarn. The results indicated to show more than 90% antibacterial reduction as per standard test method. Observed antimicrobial inhibition character indicate that catechu may be a promising natural antimicrobial finishing agent for developing bioactive and antimicrobial dyed textile materials for today's need.

A number of recent studies on simultaneous natural colouration and antimicrobial finishing of different textiles using selective natural dyes/natural agents applied alone or in combination were investigated by several authors as mentioned below for detailed study and further references:

Prusty et al. [115] have studied about simultaneous natural coloring and antibacterial finishing of few natural colorants on silk.

Similarly Gupta and Laha [116] have worked on simultaneous natural dyeing and antimicrobial finishing of cotton fabric using natural tannin-rich extract of *Quercus infectoria* (QI) plant in combination with alum, copper and ferrous sulphate as mordants showing good antimicrobial activity at 12% concentration (owf), inhibiting the bacterial reduction around 45–60% for ferrous sulphate and bacterial reduction increases to 70–90% when mordanted with alum or copper sulphate making it suitable for anti-odor agent for use in medical, sports and home textiles.

Chen and Chang [117] have applied extract of onion skin on plasma pretreated cotton fabric to obtain simultaneous coloring and antimicrobial finishing effect

where the plasma-pre-treated cotton samples subsequent dyed/grafted with extract of onion skin showed measurable inhibition zone against *S. aureus* around 1.1–0.8 cm inhibition for 10 min grafting time with onion skin extract and 0.7–0.5 cm inhibition zone for 30 min grafting time of onion skin extract.

Joshi et al. [118] have reported a comprehensive review on natural product based bioactive agents such as chitosan, natural dyes, neem extract and other herbal products for antimicrobial finishing of textile substrates which is useful for further study.

1.4 Natural dyes cum natural UV protective finishing agents

A study has been conducted by Salah [119] about antibacterial and UV property of Egyptian cotton fabrics treated with aqueous extract from waste peel of banana fruits after its extraction in 1% NaOH solution.

Chattopadhyay et al. [120] have worked on developing natural dyed jute fabric with improved color yield and UV protection characteristics using harda (myrobolan) as bio mordant (though it is not truly a mordant, it is rather a mordanting assistant having high coordinating power for promoting fiber-mordant-dye complex formation using several —OH and —COOH groups of chebulinic acid present in it) and pomegranate rind extract as natural dye as well as UV protective agent using ecofriendly ferrous sulphate and potash alum as mordants. Very good ultraviolet (UV) protection ratings were achieved in case of dyeing of jute fabric with pomegranate rind. However, Jute fabric treated with manjistha, annatto, ratanjot and baboolas natural dyes cum natural UV protective finishing agents, applied after pre-mordanting with sequential pretreatment with Harda extract as biomordant and Alum as metallic but natural eco-friendly chemical mordant. Observed results indicated that UV protection properties of the said selective natural dyes cum natural UV protective finishing agents applied on bleached jute fabric follows the following order of UV protective performances: babool > annatto > manjistha > ratanjot.

Hou et al. [12] has used in their study waste orange peel as agricultural bye product for obtaining concurrent natural coloring and UV protective finishing on textiles for potential strong UV absorbance character of orange peel applied on woolen fabrics. The results was encouraging and optimum conditions of this concurrent natural coloring and UV protective finishing of woolen textiles is: optimum temperature of 100°C, optimum time—120 min, dyeing cum finshing bath pH—3 for following dyeing cum finishing without mordant and pH is 7–9 for simultaneous mordanting, dyeing and finishing in one bath using aluminum sulphate or ferrous sulphate, that is, iron as metallic eco-friendly mordant, showing great potential of orange peel extract as useful for this purpose.

Grifoni et al. [121] have shown in their reports that UV protection property not only depends on the surface finishing agents applied whether natural or synthetic, but also depend much on fabric construction, type of fibers, type of natural or synthetic dyes and finishes used (absorption criteria of dyes and finishing agents in UV zone). In this study, they measured UPF value of different types of textile apparels, hats, canopy type shade structure made of textiles with varying fabric construction for vegetable and natural fibers based product and finally dyed with different natural dyes cum natural finishing agents using tannin based natural mordants for obtaining maximum level of safest UV protection from sunlight radiation.

Feng et al. [122] have conducted a study on the UV protective properties of hats and clothing against solar ultraviolet radiation and found that *Rheum emodi* and *L. erythrorhizon* shows equally good and comparable UV-absorption protection character as compared to the common known standard UV-absorber compounds like benzophenone and benztriazole, etc.

Sinnur et al. [123] have reported a study on natural colouration and UV protective finishing using aqueous extract of pomegranate rind, that is, commonly known as anar peel. Besides optimization of conditions of natural color extraction from dried anar peel powder, effect of different single and double mordants in different proportions and concentrations on color yield and optimization of dyeing process variables as well as measurement of UV protective action of such dyed cotton khadi fabric with extract of anar peels (pomegranate rind, i.e., *Punica granatum* L.) as a natural colorant has been reported recently as an encouraging work.

The analysis of mass spectroscopy of cellulosic textile fibers dyed with indigo has been reported as a method of its identification, which may be the basis and can be used as a finger print for identifying natural indigo with TLC and UV VIS spectroscopic results in combination. Similarly for assuring any textiles being only dyed with natural dye, need its identification method. Very recently BIS has published two national IS standards on identifying natural Indigo and madder (IS 17084-2019 for natural indigo and IS 17084-2019 for madder) for test and identification of these two natural dyes from such a natural dyed textiles.

2. Conclusions

Thus, still there are many gaps in standardizing dyeing conditions for specific fiber-mordant-dye combinations and there is still need of required scientific and industrial research on the effects of different ecofriendly chemical mordants and bio mordants (tannin based natural compounds) and mordanting assistants (gallic acid or chebulinic acid based natural compounds) for finally standardization/optimization of dyeing process variables for obtaining uniform and repeatability of shades to produce with natural colors. Another side of utilizing antibacterial/antifungal and UV protective action or deodourizing action of selective natural dyes by detailed scientific study of the effects of different after-treating compounds antibacterial/UV absorbers compounds for improving its uses as high valued textiles. Similarly study of different natural and ecofriendly chemical dye fixatives for improving color fastness to Washing and effects of UV absorber compounds on exposure of such natural dyed textiles to the exposure to sun-light/UV light can be improved by suitable after-treatment with UV absorbers. For improving rubbing fastness of such natural dyed textiles, after treatment with natural binders or different natural reactive thickeners and ecofriendly synthetic binding agents are required, besides approaches to improve antimicrobial and UV protection activity of such natural dyed textiles. It is also important to know and understand well the exact fiber-mordant dye interaction and role of different pre and post treatments on promoting color yield (in terms of K/S values), uniformity of color yield (measurable by CV % of K/S values) as well as rating for antimicrobial and UV protection factor for different fiber-mordant-natural dye combination as applicable particularly to cotton, silk, wool and jute fibers when dyed with aqueous extract of any selected natural dye.

Hence application of natural dyes on high value apparel and functional textiles are gaining worldwide interest for its less toxic nature, better biocompatibility, biodegradability, producing elegant hues and highly functional value-added textiles as environment friendly oeko-tech/ecofriendly textiles for gaining popularity for natural dyed and finished as high valued textiles of tomorrow, if its revival strategies are well created and executed with utmost care with back up of sufficient scientific study with time bound growth plan and correct revival strategy.

Some of the revival strategies include (i) availability of commercially standardized process and standard commercial shade cards for developing desired shades

with acceptable repeatability and appreciable color fastness results; (ii) availability of standardized test methods for identifying and assuring customers for proving a dyed textiles is really 100% dyed with natural dye(s) without any synthetic dyes used as adulterant/topping to match shade; (iii) commercial process variables need to be standardized for different dyes for desired shades at economical minimum cost; (iv) to train and educate concern dyers and weavers and any big or large textile industry sector for successful extraction and dyeing with natural dyes and finally (v) future creation of a natural dye mark certification method by suitable national and international bodies for consumer assurance service like khadi mark, silk mark, etc.

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