Optimization of dyeing parameters for natural dyes on cotton fabrics

Saurabh Garg[1] and D P Chattopadhyay[2]

[1]Department of Textile Design, National Institute of Fashion Technology, Kangra, India
[2]Department of Textile Chemistry, The Maharaja Sayajirao University of Baroda, Vadodara, India
saurabh.garg@nift.ac.in

Natural dyes, as the name signifies, have been used by mankind from prehistoric times. The first written records of natural dyeing were found in China dated 2600 BC. The dyes were used to colour the caves, textiles, wood, body parts and other daily use objects. As per records, these have been used in Europe during the Bronze age and Indus valley civilization. It is believed that the madder was used to dye the textiles at Mohenjo daro & Hadappa in 3500 BC[11]. Indigo dye, which is still the favorite dye for many, is as old as 5000 years and used by our ancestors in India, East Asia and Egypt. Many dyed textiles found in Egyptian tombs prove natural dyeing methods[9]. Madder, which is widely available in Europe and Asia, is found on the cloths of mummies and confirmed by chemical tests[1].

Many popular dyes and pigments like Patang, Saffron, Catechu, Dhao, Myrobalan, Madder, Pomegranate etc. were used during the Mughal reign (1556-1803) using metallic mordants with good fastness properties[13]. In a study, seventy samples of traditional textiles were collected from Calico museum, Ahmedabad, India

to identify natural dyes using Thin Layer Chromatography (TLC) and enquire about the type of natural dyes used for their respective materials. It was found that suranji, manjeet, pomegranate, myrobalan, catechu, indigo etc were used to dye the fabric[5].

Traditional Indian textiles are appreciated for the use of natural dyes in the form of dyeing, printing and weaving. Telia rumal, Ikat textile, is produced in Chirala town of Telangana state in terracotta red and black colours using natural dyes. Alizarine, iron and alum were used to create the red and black colour. Patola is a prestigious weaving from Gujarat state where warp and weft yarns are tie-dyed to produce many colours and patterns. A traditional scarf (chunri) called piliya from Rajasthan is a costume for young mothers. The fabric is dyed using turmeric to produce yellow colour and anti-inflammatory properties.

> **Traditional Indian textiles are appreciated for the use of natural dyes in the form of dyeing, printing and weaving**

Table 1 : Details of the common brown dyes[8,11]					
Source	Colour	Habit	Part used	Botanical name	Pigment
Mycrobalan	Brown/Yellow	Tree	Root, seeds, fruit, bark	Terminalia chebula	Allagic acid
Black Catechu/ Catechu	Brown	Tree	Bark	Acacia catechu	Catechin
Bark of Acacia/ Black Babool	Black/Brown	Tree	Seed pod	Acacia nitotica	Catechin
Himalayan Rhubarb/ Rhubarb	Yellow/Brown	Herb	Rhizomes and roots	Rheum emodi	Chrysophanic acid
Walnut	Brown	Tree	Hulls, shells, bark and leaves	Juglans regia L	Juglone

Indigo is widely used in tie-dyed, solid and printed traditional textiles in Rajasthan, Gujarat and other parts of India. Kalamkari is a block printed cotton textile from Andhra Pradesh that uses alum, jaggery, alizarine, myrobalan, cow dung, seeds etc[12].

In recent years, a segment of environmentally conscious consumers has created a demand for environment-friendly products. This has led to increased demand for natural dyes, and many industries have started using them. Over the last two decades, research in the area of natural dyeing has been going on. Besides dyeing textiles, natural dyes are used in food, medicines and dyeing of leather and other items.

Brown natural dyes

Mother nature gifts various tones of red, yellow, brown, and other colours. Amongst these, red and yellow are available in abundance. Myrobalan, catechu, Acacia nilotica, rhubarb and many other materials provide brown colour. Some of these materials are rich in tannins and provide good fastness properties. Some of the brown dyes are mentioned in *Table 1*.

This paper discusses the properties, usage and colour analysis of these three brown-colour dyes - Acacia nilotica, Himalayan rhubarb and catechu.

Acacia catechu

'Catechu' is obtained from the heartwood of a cutch tree (Acacia catechu) and belongs to the Leguminosae family. It is majorly found in Indian sub-Himalayas and is called catechu in Hindi. The chief colouring component in catechu is 'catechin' with molecular formula $C_{15}H_{14}O_6$ and contains many similar compounds, including gummy matter. Its high tannin content and colouring component make it suitable for textile dyeing. Due to its high tannin content, it can be used as a mordant for cellulosic fabrics. It is known for its brown colour along with different hues and tones when used with different mordants and dyeing conditions. It produces a dark brown, fawn, or golden brown with good wash fastness properties. It imparts yellowish brown colour on cotton, wool and silk materials, and turns grey-brown with iron and olive brown with copper mordant[14]. Catechu is known for its medicinal properties. As per traditional Chinese medicine, catechu has astringent, neutral and bitter properties. It has antimicrobial activities, anti-inflammatory, anti-cancer, UV protection and many other properties on dyed textiles[7].

Rheum emoji

The botanical name for Himalayan rhubarb is Rheum Emodi and dolu in Hindi. It is a stout found at an altitude of 2800-300 m in India. It is majorly used in medicines, especially in Ayurvedic and Unani systems of medicines. Its roots are used for textile materials' colouration and contain several anthraquinone derivatives. Besides colouration properties, Rhubarb is known to exhibit anti-oxidant, UV protection and anti-microbial properties[15].

Rhubarb can be used to colour various textiles, including silk, wool, cotton and some manmade textiles. The dye is pH sensitive and changes colour with pH, mordant type and mordant concentration. It produces brown, yellow, yellowish green and reddish brown shades[3,4]. The fastness properties obtained depend on the type of mordant used and are generally in the moderate to good range.

Acacia nilotic

Babool tree (Acacia nilotica) bark is rough, dark brown or reddish brown wood. It has golden yellow flowers with long white thorns. The tree's bark produces brown dye, which is used to dye many textile materials. Acacia nilotica dye contains catechin as the colouring component and has 9%-15% tannins[2]. The Acacia nilotica bark is found very useful in the dyeing of cotton, silk, wool and nylon fabrics with good fastness properties[6]. It produces black, brown and khaki shades using different mordants and dyeing conditions[10]. Acacia nilotica dye on cotton has been successfully used with the padding technique.

Materials and methods

Materials

Cotton fabric

Ready for dyeing (RFD) bleached

60s cotton cambric fabric, obtained from RMP Fab Sourcing Pvt Ltd, Faridabad, Haryana, India was used for the present study.

Chemicals, dyes and auxiliaries

Citric acid, sodium carbonate, alum (aluminium potassium sulphate) and non-ionic detergent was used in this present work. All these were laboratory grade chemicals from Fisher Scientific and Loba Chemie Pvt Ltd. Dye powder for Acacia nilotica, Himalayan Rhubarb, and catechu dyes were procured from Jaipur, India.

Instruments and apparatus

Electronic pH meter, electronic weighing scale, HTHP beaker dyeing machine (R B Electronic & Engineering Pvt Ltd) laundrometer and crock-o-meter (Ramp Impex Pvt Ltd).

Methods

Mordanting

Ready for dyeing (RFD) bleached cotton cambric fabric was mordanted using alum with MLR of 1:60, temperature 90°C for 60 min. After the complete process, the samples were washed twice to remove the excess alum particles on the surface to make them ready for dyeing.

Dyeing

The dyeing of pre-mordanted cotton fabric was done at MLR of 1:90 with a dye percentage 30% owf at a range of pH, time and temperature to find the optimum dyeing condition for each. The details of the experimental setup are indicated in *Table 2*. 17 samples of each dye were dyed at different dyeing conditions as per *Table 3*, with a total of 51 samples.

All 51 samples were dyed as per the conditions mentioned above and repeatedly washed with hot and cold water. Then, the samples were subjected to soaping with 1 gm/l soap solution at 60°C for 15 min and then dried.

One sample of each dye was dyed with the same method as mentioned above at optimum dyeing conditions for

Table 2 : Experimental setup for optimization of dyeing parameters	
Parameter	Values
Control Factors	
pH	3, 5, 7
Temperature (°C)	65, 80, 95
Time (min)	30, 60, 90
Response Variable	
Surface Colour Strength (K/S)	

colourimetric and fastness properties analysis.

Testing and evaluation

Colorimetric analysis

Surface colour strength parameters were determined using Konica-Minolta spectrophotometer, Model-3600d. K/S is the measure of surface colour strength of dyed samples, and CIEL*, a*, b* values signify the lightness/darkness, redder/greener tone, and yellower/bluer tone, respectively. K/S, CIEL*, a*, b*, values were obtained with the help of relevant software for all the dyed samples.

Optimum dyeing conditions, graphs of K/S vs pH, K/S vs temperature, and K/S vs time were obtained using the Box-Behnken Design of experiment

for all the three dyes under analysis. Comparative analysis of the maximum K/S dyed sample was also done for all the three dyes. L*, a* and b* values were used to determine the tonal variation and lightness/darkness for each highest K/S sample for each dye.

Colour fastness evaluation

Colour fastness to wash and rubbing was evaluated on the samples dyed at optimum dyeing conditions for each dye. Colour fastness was evaluated using a laboratory laundrometer with AATCC test method 61-2010. Dry and wet rubbing fastness was evaluated using laboratory crock-o-meter with AATCC test method 8-2007.

Results and discussion

Analysis of colour strength

Table 4 shows the actual K/S for all the 51 dyed samples; the response surface plots for K/S vs pH, temperature and time for the three mentioned dyes are given in *Figs 1, 2 & 3*. It is evident from *Fig 1* that the K/S values for Catechu are sharply increasing with an increase in dyeing pH

Table 3 : Dyeing conditions for Catechu, Acacia nilotica and Rhubarb as per Box Behnken Design of experiment					
Catechu (Katha, K)	Acacia nilotica (Babool, B)	Rhubarb (R)	pH	Temperature (°C)	Time (min)
K1	B1	R1	5	65	30
K2	B2	R2	3	65	60
K3	B3	R3	7	65	60
K4	B4	R4	5	65	90
K5	B5	R5	7	80	30
K6	B6	R6	3	80	30
K7	B7	R7	5	80	60
K8	B8	R8	5	80	60
K9	B9	R9	5	80	60
K10	B10	R10	5	80	60
K11	B11	R11	5	80	60
K12	B12	R12	3	80	90
K13	B13	R13	7	80	90
K14	B14	R14	5	95	30
K15	B15	R15	7	95	60
K16	B16	R16	3	95	60
K17	B17	R17	5	95	90

and maximize at pH 7. The K/S values are increasing slowly with the increase in dyeing temperature and maximized at 95°C. The impact of dyeing time is increasing with a minor increase with time. The impact of pH may be due to non-formation of ionic bond between dye-mordant-fibre complex in acidic pH and formation of hydrogen or co-ordinates at neutral pH. It is believed that increasing the temperature enhances the fibre swelling and the dye diffusion inside the fabric, and hence increased the colour strength of the dyed fabric. Impact of time may be due to increased chances of more dye diffusion onto the fabric before saturation.

Fig 2 plots show that the K/S for the Acacia nilotica dye decreases with an increase in pH from 3 to 4, and then increases sharply and maximizes at pH 7. The impact of both the parameters is a minor increase in K/S value. The maximum value of K/S was obtained at a temperature of 95°C and 90 min time. *Fig 3* plots show the K/S for Rhubarb dye and it is evident that the trend is similar to Acacia nilotica dye. The maximum value of K/S is found at pH 7, temperature 94°C and time 84 min. The effect of dye bath pH can be attributed to the correlation between the dye structures for both, the Acacia nilotica and Rhubarb dyes. Effect of temperature and time may be due to
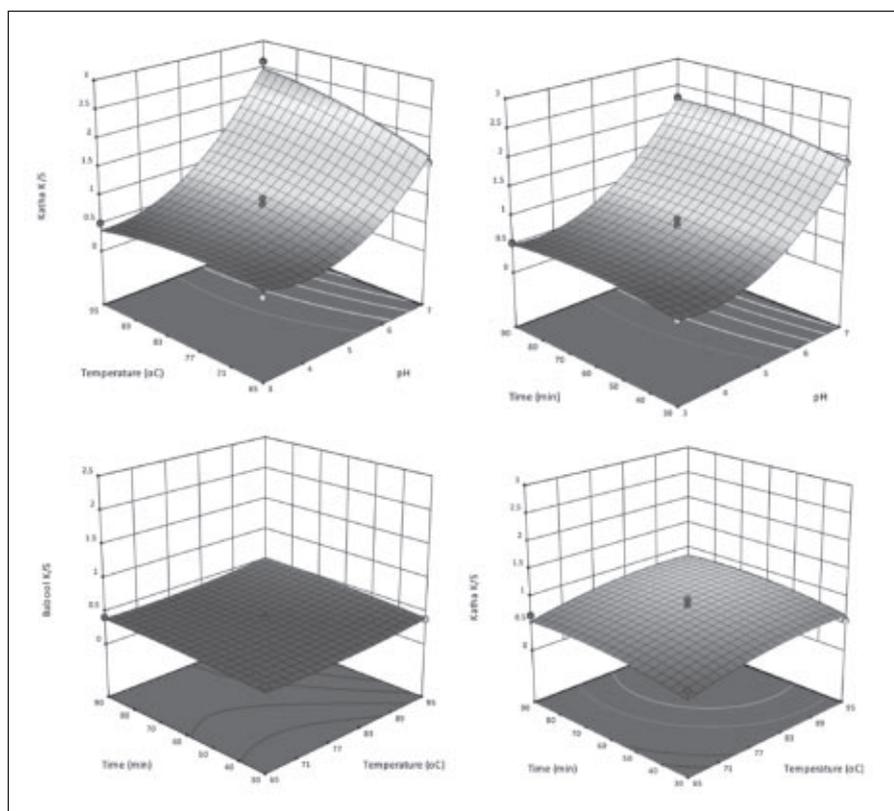

Fig 1 : K/S vs pH, temperature and time graph for Catechu

the same reason as mentioned for the catechu dye.

The optimum dyeing conditions for the three dyes were predicted from the Box Behnken design of the experiment. The predicted optimum dyeing conditions are mentioned in *Table 5*. It is observed that the optimum parameters for the
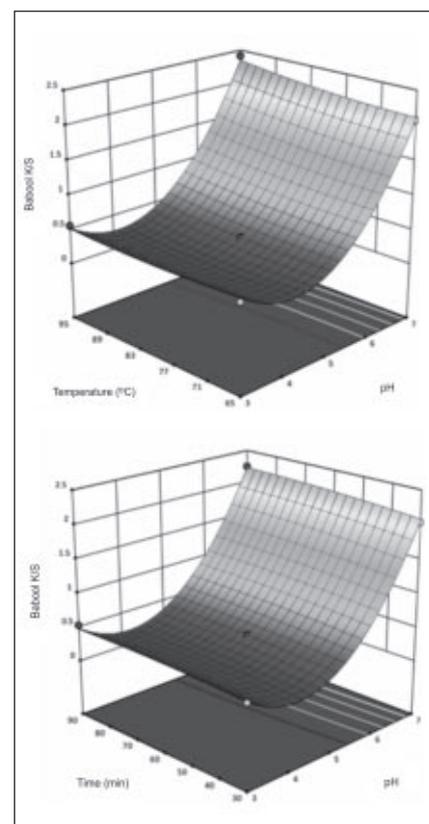

Fig 2 : K/S vs pH, temperature and time graph for Acacia nilotica

Table 4 : K/S values of 51 dyed samples					
Sample number	K/S	Sample number	K/S	Sample number	K/S
K1	0.50	B1	0.40	R1	0.26
K2	0.43	B2	0.46	R2	0.31
K3	1.58	B3	2.08	R3	0.89
K4	0.65	B4	0.41	R4	0.29
K5	1.93	B5	2.04	R5	0.97
K6	0.42	B6	0.41	R6	0.36
K7	0.72	B7	0.40	R7	0.36
K8	0.81	B8	0.39	R8	0.39
K9	0.77	B9	0.39	R9	0.34
K10	0.96	B10	0.39	R10	0.37
K11	0.85	B11	0.38	R11	0.36
K12	0.52	B12	0.53	R12	0.45
K13	2.27	B13	2.25	R13	1.16
K14	0.56	B14	0.38	R14	0.32
K15	2.61	B15	2.41	R15	1.18
K16	0.50	B16	0.56	R16	0.44
K17	0.70	B17	0.44	R17	0.39

being eco-friendly

Ensure the life of your fabrics as well as your planet.

Come to COLORANT.

COLORANT is among the first few Indian companies to be certified for prestigious **ZDHC (Level-3)** Certification.

COLORANT®

Quality is Colorant

ZDHC Level 3 Certified

COLRON® Reactive Dyes

COLORANT LIMITED
Plot No. 116, Phase II, G.I.D.C. Vatva, Ahmedabad 382 445, Gujarat, INDIA
Phone: +91 79 4030 7233 / 4583 • Email: mktg@colorantindia.com

www.colorantindia.com

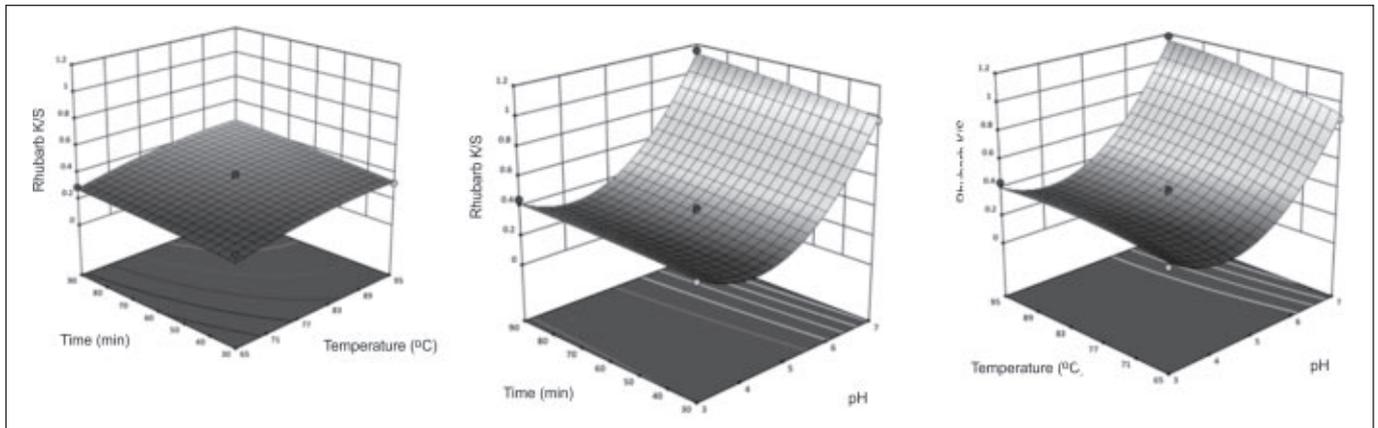*Fig 3 : K/S vs pH, temperature and time graphs for Rhubarb*

Table 5 : Predicted optimum dyeing conditions and K/S				
Dye	pH	Temperature (°C)	Time (min)	K/S
Catechu	7	95	81	2.538
Rhubarb	7	94	85	1.185
Acacia nilotica	7	95	90	2.398

Fig 4 : Predicted K/S for three brown dyes at pH 7, temperature 95°C and time 86 min

Fig 6 : Comparison of b values for the three dyes*

three dyes are similar.

Comparative analysis of three brown dyes

The comparative analysis of the three brown dyes was done in terms of the maximum K/S and CIE a*, b* values to find the best suitable dye for the dyer. Predicted K/S values were obtained from the Box-Behnken design of the experiment for common dyeing parameters of pH 7, temperature - 95°C, and time 86 min. *Fig 4* shows that the Catechu dye produces a maximum K/S value of 2.535, and Rhubarb produces a minimum K/S value of 1.213. The K/S predicted for Acacia nilotica dye was 2.395. It shows that the Catechu dye can obtain the

darkest colour.

It is a known fact that, besides the K/S value, the dyer needs to know about the tone of the dye, and colourimetric parameters a* and b* values are appropriate parameters to judge the tone. These values are
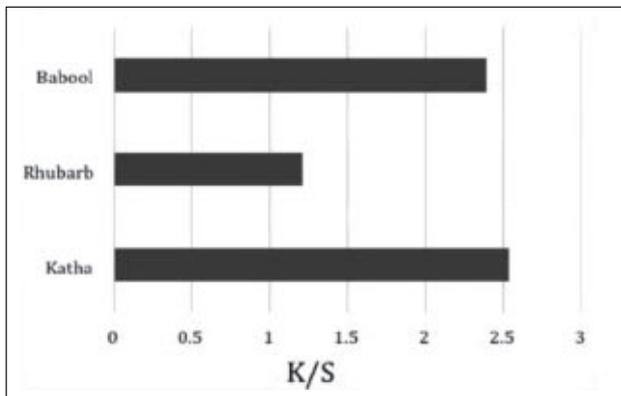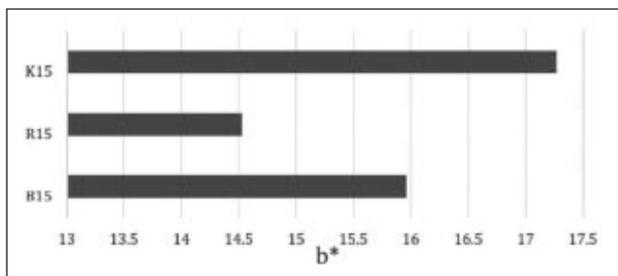

Fig 5 : Comparison of a values for the three dyes*

analyzed for the K15, B15 and R15 (*Table 3*), wherein the dyeing parameters are close to the optimized parameters. *Fig 5* & *Fig 6* show the comparative tonal variation of the three dyes. It is evident that the catechu has maximum a* and redder tone than the other two dyes. In the case of the b* value, the catechu dye shows the maximum value and is yellower than the other dyes. The visual examination of the samples also aligns with the results obtained.

Colour fastness properties

Wash fastness and rubbing fastness to colour were tested for the samples

Table 6 : Rubbing and wash fastness rating of the three brown dyes				
		Catechu	Acacia Nilotica	Rhubarb
Rubbing fastness	Dry	4-5	5	5
	Wet	3	3-4	4
Wash fastness	Colour change	3-4	3-4	4
	Staining	4	4	4-5

dyed in optimized conditions. The rating for both the tests are given in *Table 6*. It was found that the dyes have good to very good fastness properties.

Conclusion

Alum mordanted cotton fabric samples were dyed with Catechu, Rhubarb and Acacia nilotica at a range of pH, temperature and time. It was found that neutral pH, temperature 94°C-95°C and time of 81 min - 90 min were the optimum dyeing conditions for all the three dyes. The K/S sequence of three dyes was found as: Catechu>Acacia nilotica>rhubarb. It is also found that catechu dyed fabric is redder and yellower than other two dyes, and that the dye has good to very good fastness properties. The outcome of the analysis may help the dyer with the appropriate use of dye for the desired results.

References

1 ABEROUMAND A, A review article on edible pigments properties and sources as natural biocolorants in foodstuff and food industry, Journal of Dairy & Food Sciences, 6(1), pp 71-78 (2011)

2 Dhanania Y, Singhee D and Samanta A K Optimization of dyeing process variables for eco-friendly dyeing of cotton fabric with babul bark extract as a natural dye and gallnut extract as a bio-mordant, Journal of Natural Fibers, 00(00), pp 1-18, Available at: https://doi.org/10.1080/15440478.2021.1875955 (2021)

3 Khan M A, Shahid-ul-Islam and Mohammad F. Investigating the Combined Effect of Natural and Metal Salt Mordants on Coloring Potential of Indian Rhubarb (Rheum emodi) Natural Dye, Journal of Natural Fibers, 14(2), pp 196-204 Available at: https://doi.org/10.1080/15440478.2016.1193084 (2017)

4 Khan S A et al, Antimicrobial activity of wool yarn dyed with Rheum emodi L. (Indian Rhubarb), Dyes and Pigments, 95(2), pp 206-214. Available at: https://doi.org/10.1016/j.dyepig.2012.04.010 (2012)

5 Kharbade B V and Agrawal P, Analysis of Natural Dyes in Indian Historic Textiles, Studies in Conservation, 33(1), pp 1-8 (1988)

6 Patel B H, Agarwal B J and Patel H M Dyeing of nylon with babool bark extract, Textile Asia, 33(9), pp 34-42 (2002)

7 Samant L et al, Antimicrobial and UV Protection Properties of Cotton Fabric Using Enzymatic Pretreatment and Dyeing with Acacia Catechu, Journal of Natural Fibers, 19(6), pp. 2243-2253. Available at: https://doi.org/10.1080/15440478.2020.1807443 (2022)

8 Samanta A K, Agarwal P and Datta S, Dyeing of jute with binary mixtures of jackfruit wood and other natural dyes - Study on colour performance and dye compatibility', Indian Journal of Fibre and Textile Research, 33(2), pp 171-180 (2008)

9 Satyanarayana D N V and Chandra K R Dyeing Of Cotton Cloth with Natural Dye Extracted From Pomegranate Peel and its Fastness, International Journal of Engineering Sciences & Research Technology, 2(10), pp 1-6 (2013)

10 Singh P P, Application of Acacia Catechu Natural Dye on Banana Fiber, Ergonomics International Journal, 4(1). Available at: https://doi.org/10.23880/eoij-16000229 (2020)

11 Siva R, Status of natural dyes and dye-yielding plants in India, Current Science, 92(7), pp 916-925 (2007)

12 Textiles and Fabrics of Indian States Ministry of Culture, Government of India. Available at: https://indianculture.gov.in/textiles-and-fabrics-of-india/textilestate (2022)

13 Yusuf M, Shabbir M and Mohammad F. Natural Colorants: Historical, Processing and Sustainable Prospects, Natural Products and Bioprospecting, 7(1), pp 123-145. Available at: https://doi.org/10.1007/s13659-017-0119-9 (2017)

14 Zerin I and Foisal A B M, Effect of Mordanting Process on Cotton Dyeing with Acacia Catechu, Textile Research Conference, 6639(October), pp 29-31 (2016)

15 Zhou Y et al, Simultaneous dyeing and functionalization of silk with three natural yellow dyes, Industrial Crops and Products, 64(1), pp 224-232, Available at: https://doi.org/10.1016/j.indcrop.2014.09.041 (2015). **AD**

New microfibre filtration device

The issue of microfibres from synthetic fibres deteriorates the global plastic problem. Every year, over half a million tons of microfibres end up in the world's oceans due to clothes washing alone. Studies indicate that these synthetic textile microfibres, categorized as microplastics, are the predominant source of primary microplastic pollution in the oceans. Since 2005, incidents of microplastic contamination have surged 10-fold, with over 171 trillion microplastic particles current polluting our oceans.

Xeros Technology, Rotherham, UK, has launched a new product, XF3, an external version of its patented, market-leading washing machine filtration device, XFilter. Xeros' XFilter is recognized as an effective washing machine filtration device in the current market. It efficiently captures over 99% of microplastics and 80% of polycotton microfibres released during a washing cycle, thus preventing them from contaminating global oceans and waterways. The filter offers consumers good ease of use, requiring no cartridge changes and boasts one of the lowest empty frequencies among available filters.

Xeros has designed the XF3, an external filtration device that can be affixed to any make of washing machine. The product is slated for consumer launch in 2024.

STUDY OF COMPATIBILITY PARAMETERS OF MIXTURE OF NATURAL DYES

D P Chattopadhyay[1] Saurabh Garg[2]
[1]Professor, Department of Textile Chemistry,
Faculty of Technology & Engineering,
The Maharaja Sayajirao University of Baroda, Vadodara ,
[2]Assistant Professor, Department of Textile Design, NIFT, Kangra (HP)
*Corresponding author : Saurabh.garg@nift.ac.in

Abstract

Eco-friendliness is the essential requirement of the present world of textiles and textile processing and its absence is the major cause of various types of pollution. Before the invention of synthetic dyes, natural dyes were the only medium for textile colouration. Natural dyes can contribute towards achieving the eco-friendly textiles. Natural dyes can be used with a completely harmless procedure and give soothing colours on textiles. In spite of many advantages, it has many limitations related to fastness, lengthy dyeing procedure, limited shade range, etc. The issue of limited colour range can be overcome by the use of binary and tertiary combination of these dyes to produce a wider shade range. The combination of natural dyes can be done based on their compatibility with each other. The compatibility of dyes depends on parameters such as optimum dyeing conditions and procedure, colour strength and related colour interaction parameters, metamerism index, etc. This paper presented various factors responsible for the compatibility of natural dyes and reviewed the compatibility aspects of certain natural dyes.

Keywords: Compatibility, Natural dye mixture, Textiles

Introduction to Natural Dyes

Textile materials (natural and synthetic) used to be coloured for value addition, look and desire of the customers. Anciently, this purpose of colouring textile was initiated using colours of natural source, until synthetic colours/dyes were invented and commercialized. For ready availability of pure synthetic dyes of different types/classes and its cost advantages, most textile dyers/ manufacturers shifted towards the use of synthetic colourant. Almost all the synthetic colourants being synthesized from petrochemical sources through hazardous chemical processes pose a threat towards its eco-friendliness.

Hence, worldwide, growing consciousness about the organic value of eco-friendly products has generated renewed interest of consumers towards the use of textiles (preferably natural fibre product) dyed with eco-friendly natural dyes. Natural dyes are known for their use in colouring of food substrate, leather as well as natural fibres like wool, silk, and cotton as major areas of application since pre-historic times. Although this ancient art of dyeing textiles with natural dyes withstood the ravages of time, due to the wide availability of synthetic dyes at an economical price, a rapid decline in natural dyeing continued. However, even after a century, the uses of natural dyes never erode completely and they are being still used in different places in the world. Thus, natural dyeing of

different textiles and leathers has been continued mainly in the decentralized sector for specialty products besides the use of synthetic dyes in the large scale sector for general textiles/apparels.

There are many advantages of the use of natural dyes on textiles including eco-friendly nature, unsophisticated and harmonized with nature and many more. Besides advantages, there are certain disadvantages also such as reproducibility of shade, colour fastness issues, and limited shade range.

Limited shade range issue can be dealt with in different ways. Dyers have been doing the two-step process to get newer shades which is time-consuming and affects the fiber properties as well. In line with synthetic dyes, mixtures of natural dyes in the single bath may be used with several challenges. The essential element for this is that the dyes should be compatible with each other. In this paper, various factors responsible for the compatibility of natural dyes are discussed along with compatibility aspect of some synthetic and natural dyes have been discussed. The compatibility status will determine whether the combination of dyes will be suitable to use and what type of problems can arise. This paper will give insight on the colour parameters like colour fastness properties, exhaustion-fixation and adsorption isotherms for combination of natural dyes. Few examples of compatibility check with newer method for binary combination of natural dyes has also been discussed.

Methodology

Methods to find the compatibility of combination of dyes need to check different aspects of colour parameters. Various methods have been developed to check the compatibility by different authors. These include methods as dip test, plotting ΔL versus K/S or Δ C versus K/S, using diffusion coefficient values and by finding relative compatibility rating (RCR) which is newer method for compatibility check. Also, the proper methods to assess the compatibility of wide range of natural dyes are yet to be studied in detail.

Compatibility of Dyes

Most of the dye producers are providing the different tones and hues of three basic colours. They try to provide as many as tones possible by changing the structure of the dye. To dye a piece of fabric for any given colour standard, three colours are generally required. If the dyer is lucky enough, the same tone may be obtainable by single or combining two dyes but this is a rare phenomenon. Mostly, the dyer would require three primary colours. For the dyer to mix the dyes and bring a shade out of it is not an easy task. The dyer has to check the compatibility of the dyes being mixed. The optimum dyeing conditions may be different for different dyes. The fastness properties for the different dyes may be different or the mixture may change the tone during the dyeing cycle. Reproducibility of the shade is a very crucial matter of concern for the dyer. There is the scope of improvement through using better compatible dyes (Sivaramakrishnan, 2014). All the dyes in the mixture should have the same rate of dyeing. They should have similar rates of exhaustion when used in mixtures under the given dyeing conditions. During dyeing, the colour of the goods will then gradually become deeper, but, since the dyes are absorbed in the same proportions throughout the process, the hue does not change and the goods will always be on shade (Broadbent, 2001).

In this way, several dyeing parameters are mentioned in the literature, which defines the compatibility of dyes. Many researchers have attempted to define the qualitative and quantitative methods for compatibility.

Methods to Assess the Compatibility of Dyes

One of the conventional methods to check the compatibility of dyes is the dip test. In this test, small pieces of cotton of equal weight are dyed in the same bath with a mixture of dyes. At various intervals, a small dyed sample is removed from the bath and replaced by an identical piece of undyed fabric. A series of dyed samples arranged in order of increasing dyeing time will have gradually decreasing colour depth, but the invariant hue, when the dyes used are compatible (Sultana & Uddin, 2007)

Degree of on-tone build-up, rate of dyeing, half-dyeing time, diffusion coefficient, change in hue angle are also used to check the compatibility of binary and tertiary mixtures of dyes (Beckmann, Hoffmann, & Otten, 1972).

Plotting ΔL versus K/S or ΔC versus K/S is generally used for compatibility check of two dyes for two sets of the progressive depth of shade developed for a binary mixture of dyes. It is done by varying the temperature and time of dyeing for one set and varying total dye concentrations of the binary mixtures of dyes in another set to judge whether the two sets of curves for shade buildup run alike or not.

A plot of ΔL versus ΔC and/or ΔL versus K/S gives better results than other methods assumes that the there is no interaction in dyes with no change in the rate of dyeing in presence of another dyestuff which is not true. For a plot of ΔL versus ΔC and/or ΔL versus K/S would require a precise temperature-controlled machine for progressive shade buildup. This method is time-consuming and is subjective nature (Hoffman, 1988).

Dye compatible matrix (DCM) can be used to define the optimum dyeing profile for exhaust dyeing of dyes, understanding dye migration behavior, level dyeing check and the degree of process control required to achieve shade reproducibility. A simple dip test can check the compatibility of dyes by dyeing the several pieces of cloth dyed successfully for short times in the bath containing dyes. The changes in the hue between the first and last samples are visually judged (Sivaramakrishnan, 2014).

The determination of compatibility in exhaust dyeing has been reported in different ways. A study was done where the dyeing process is interrupted at regular interval and degree of exhaustion is assessed visually. As the visual evaluation gives a qualitative evaluation, photometric methods are necessary for quantitative evaluation of compatibility (Hoffman, 1988). Some of the photometric methods have used theoretical compatibility values and others have evaluated by either extinction coefficient of the dye liquor or reflectance values of the dyed materials when dyeing binary combinations. HPLC combined with spectrophotometry has also been used for improvement in accuracy (Schlaeppi, Wagner, & McNeill, 1982). Harnrnoudeh et al introduced a 'Kombi-Test' by which the dye penetration into a film roll by single dyes is evaluated. This method can be used to determine diffusion values and on-tone diffusion.

Special tests have been developed compatibility check for the continuous dyeing process. In the case where the interaction between the dyes is negligible, tailing indices can be considered as the guidelines for dye selection. A dip test was developed for the continuous dyeing of nylon carpets. The compatibility of dyes is also examined during fixation by measuring fixation curves in combination dyeing for pad-steam dyeing of acrylic fibers.

Compatibility of Synthetic Dyes

Direct dyes vary widely in their dyeing behavior giving compatibility problems. The compatibility of direct dyes depends on the rates of dyeing and migration and the salt sensitivity of the dyes. Hue differences as dyeing proceeds are much more apparent than depth differences. Incompatible dyes also tend to give unlevel dyeings. They have different rates of migration, different degrees of fiber penetration because of dissimilar diffusion rates, and give a change of fabric hue during dyeing because of differences in their overall rates of absorption. For reactive dyes, we need to mix dyes with the same type of reactive group having about the same substantivity. For cationic dyes, the key parameters determining the compatibility of dyes are the fiber saturation value and the combination of constant or compatibility value (Broadbent, 2001). One standard test (AATCC Test Method 141) evaluates a dyes combination of constant or compatibility value (Beckmann, Hoffmann, & Otten, 1972).

The compatibility of certain reactive dyes on cotton in the different textile industry in Bangladesh. Three steps process was used which includes the measurement of the extinction coefficient, measuring the concentration at a different stage of dyeing and measuring the colour strength before and after wash (Sultana & Uddin, 2007).

Determination of compatibility of reactive dyes was done using the chromatic diagram. It was assessed qualitatively from the nature of plots of Chromaticity Co-ordinates (y against x). Compatibility is observed if the dominant wavelength of all the dyed samples taken out at various time intervals during dye fixation falls between 10 or less than 10 nm (Singh, Bhattacharyya, & Gupte, 2006).

The auxiliaries present in the dye bath may also affect the compatibility of dyes. Compatibility analysis of reactive dyes is done by exhaustion-fixation and adsorption isotherm on knitted cotton fabric. Red RR, Blue RR, and Yellow RR dyes were analyzed. The exhaustion % of Red RR and Blue RR was uniform but exhaustion % of Yellow RR was decreasing with the increase of shade %. The difference in their fixation was significant in deeper concentrations. Increasing the amount of electrolyte gives better results especially for deeper shades. It was also found that a decrease in alkali amount in a dye bath and reducing the washing temperature increases the compatibility of these dyes (Naser, Haque, Hannan, & Rana, 2015).

Compatibility of Natural Dyes

The compatibility of natural dyes on Aluminum pre-mordanted woolen yarn, diffusion coefficient has been calculated and compatibility was checked. The dyes were a walnut green shell, cochineal, and weld. The following equation has been used to check the compatibility:

$$log\,\frac{C_{t,1}}{C_{0,1}} = K.\,log\,\frac{C_{t,2}}{C_{0,2}}$$

Where, $C_{t,1}$ and $C_{t,2}$ denote the concentration of each dye in the dye bath at time t. Besides, $C_{0,1}$ and $C_{0,2}$ show the concentration of each dye in the dye bath at the beginning of dyeing or time=0, respectively. K is a constant that defines the degree of compatibility and ranges between 0 and 1 with 0 as incompatible and 1 as excellent compatibility. It was found that the

exhaustion of each dye in single dyeing is different from the exhaustion in the dyeing mixture. The order for compatibility is as follows: cochineal-weld > walnut green shell-cochineal > walnut green shell-weld (Shahparvari, Safi, Safapour, & Gharanjig, 2018).

It was observed that the use of a mixture of turmeric and madder on cotton in case of simultaneous mordanting shows a synergistic effect in colour development than that for single dye application; 50:50 ratio of turmeric and madder gives the best results (Singh, Jahan, & Gupta, 1996). For the combined dye application, it is observed that in the case of simultaneous mordanting method, turmeric when combined with either madder or red sandalwood gives better colour strength, while myrobolan shows the reverse trend (Samantaa & Agarwal, Application of natural dyes on textiles , 2009).

Single and binary and mixture of aqueous extract of red sandalwood with aqueous extract of the other five natural dyes in different proportions are applied on bleached jute fabric. The other five dyes used are Manjistha (MJ), Jackfruit wood (JFW), Merigold(MG), Sappan wood (SW) and babool(BL). These were used in different proportions with Red Sandalwood (RSW). Colour parameters, colour fastness, and compatibility were checked for these combinations of dyes. In this study, the author has not used the conventional methods of compatibility check but has formulated a newer method of assessing then compatibility using a newer index called CDI. On application of different proportion of binary mixtures of dyes on the same fabric, magnitude of respective ∆E, ∆C, ∆H and Metamerism Index (MI) values irrespective of their sign and direction have been utilized to obtain an empirical index called 'CDI' for the samples dyed with different proportions of binary mixture of dyes by the following relationship.

$$CDI = \frac{\Delta E \text{ x } \Delta H}{\Delta C \text{ x } MI}$$

The closer the CDI values for a different proportion of mixture of dyes applied on the same fabric under the similar condition of dyeing, the higher is the compatibility. A relative compatibility rating (RCR) is proposed in accordance with CDI values. The scale of compatibility (RCR) is 0-5, zero is the least compatible and 5 is excellent compatibility. As per RCR system, the order of compatibility was found to be as RSW:MJ>RSW:MG>RSW:JFW>>>RSW:BL>>>RSW:SW (Samanta, Agarwal, Singhee, & Datta, 2009).

In a similar study as above, varying proportion of binary mixtures of aqueous extracts of jackfruit wood (JFW) with other natural dyes, like manjistha (MJ), red sandal wood (RSW), marigold (MG), sappan wood (SW) and babool (BL), have been used to dye bleached jute fabric pre-mordanted with 20% myrobolan followed by 20% aluminium sulphate. The compatibility was found by conventional as well as the newer method (by finding RCR). The order of relative degree of compatibility of these binary pairs of natural dyes applied on pre-mordanted jute was found to be JFW:RSW ≥ JFW: BL ≥ JFW : MJ >>> JFW: MG >>> JFW : SW (Samantaa, Agarwal, & Datta, Dyeing of jute with binary mixtures of jackfruit wood and other natural dyes — Study on colour performance and dye compatibility , 2008).

Conclusion

Dyers using natural dyes currently either produce shade with single dye or apply different dyes in multi-stage application technique. For the application of more than one dye from single bath require compatibility analysis of the dyes. The study of the compatibility of

natural dyes has been done for a few dyes only. Extensive research is required for other dyes and their combinations. On finding the compatibility of dyes, the optimum dyeing/mordanting conditions can be found out. Natural dye combination for producing combination shade is currently a challenge. Proper methods to assess the compatibility of wide range of natural dyes are yet to be studied in detail.

Bibliography

1. Beckmann, W., Hoffmann, F., & Otten, H. (1972). Practical Significance, Theory and Determination of Compatibility of Dyes on synthetic-polymer Fibres. *Colouration Technology, 88* (10), 354-360.
2. Broadbent, A. D. (2001). *Basic Principles of Textile Coloration* . Canada: Society of Dyers and Colourists .
3. Hoffman, F. (1988). Compatibility of dyes. *Colouration Technology, 18* (1), 56-64.
4. Naser, A., Haque, M. A., Hannan, M., & Rana, M. M. (2015). Compatibility analysis of reactive dyes by exhaustion-fixation and adsorption isotherm on knitted cotton fabric. *Fashion and Textiles* , *2* (3), 1-12.
5. Samanta, A. K., Agarwal, P., Singhee, D., & Datta, S. (2009). Application of single and mixtures of red sandalwood and other natural dyes for dyeing of jute fabric: studies on colour parameters/colour fastness and compatibility . *The Journal of The Textile Institute, 100* (7), 567-587.
6. Samantaa, A. K., & Agarwal, P. (2009). Application of natural dyes on textiles . *Indian Journal of Fibre & Textile Research , 34*, 384-399.
7. Samantaa, A. K., Agarwal, P., & Datta, S. (2008). Dyeing of jute with binary mixtures of jackfruit wood and other natural dyes — Study on colour performance and dye compatibility . *Indian Journal of Fibre & Textile Research , 33*, 171-180.
8. Schlaeppi, F., Wagner, R. D., & McNeill, J. L. (1982). HT Dyeing of Polyester and Polyeter/Cotton. *Textile Chemist & Colorist, 14* (12), 257-268.
9. Shahparvari, M. R., Safi, M., Safapour, S., & Gharanjig, K. (2018). Compatibility of Natural Dyes on Aluminum Pre-Mordanted Woolen Yarns by Determination of Diffusion Coefficient . *Fibers and Polymers , 18* (8), 1663-1669.
10. Singh, M., Bhattacharyya, N., & Gupte, V. (2006). Determination of compatibility of reactive dyes in mixtures by chromatic diagram. *Colourage, 53* (11), 71-76.
11. Singh, N., Jahan, S., & Gupta, K. (1996). Optimization od Dyeing Process for Silk with Natural Dye from Nagris. *Asian Textile Journal, 5* (6), 48-49.
12. Sivaramakrishnan, C. (2014, September). Compatibiliy of dyes. *Colorage* , 42-44.
13. Sultana, R., & Uddin, M. Z. (2007). Compatibility Testing of Reactive Dyes. *Journal of Mechanical Engineering , ME38* , 61-64.