International Journal of Applied Home Science *RESEARCH ARTICLE*
Volume 2 (1&2), Jan. & Feb. (2015) : 8-15 ISSN : 2394-1413
Received : 27.12.2014; Revised : 08.01.2015; Accepted : 14.01.2015

Value addition of mulberry silk with Indian willow (*Salix tetrasperma)*

MINTI GOGOI* AND BULBUL BARUAH
Department of Textiles and Apparel Designing, College of Home Science, Assam Agricultural University, Jorhat (Assam) India
(Email : mintigogoi@gmail.com)

ABSTRACT

Silk is a highly populous fiber due to its luster, fineness and its exotic beauty. The use of natural color on silk is an important aspect which can add more qualities to the silk yarn and fabric. Though the use of natural color on textiles are increasing day by day, its used on silk is a challenging task and it is very limited due to non-availability of standard procedure of its application and colored catalogue. Dyeing of silk with Indian willow through optimization process and chemical analysis of color compound present in the dye were carried out by TLC, column chromatography,UV,IR, NMR and MS spectroscopy.The color obtained from the plant were variation of browns with different mordants. The basic chemical group responsible for the color yielding properties of plant was identified a" salicin".

Key Words : *Salix tetrasperma*, Mordant, Fastness

INTRODUCTION

Color being the most striking and attractive things in the world, human are close to the color of Textiles more than anything. Every bright color flora is being considered as a source of color for natural dyeing for different material such as food, beverage and textiles. Among all other textiles, silk yarn due to its equisetic qualities like natural sheen, inherent affinity for dyes and vibrant colour, high absorbency made an important fiber for dyeing. As India is the second largest producer and highest consumer of silk, the qualities of silk can be highly enhance by natural dyeing process. The North East India, particularly Assam is the treasure house of natural resources, mainly the herbs and plants, the extraction of plants or vegetables dye and pigments through various scientific methods are now becoming more demandable mainly due to the need for sun protectionas it is only the natural dyed clothing which can protect human body from harmful effect of UV ray (Sarker, 2004). As name implies allnatural dyes are produce from renewable and bio degradable sources and always give a very uncommon, soothing and soft shades as compared to the harmful synthetic dye. Natural dyeing also symbolizes craft practices which reflect a harmonious, sustainable relationship with the eco system and the local plant reservoir (Cardon, 2010).

The silk being the "Queen of Textiles" is admired by all the people of the world and currentlyit is becoming a key export commodity to Western world, researchers aim was to screen out a new dye source from the plant kingdom of North east India and to find out its color components.Hence

Cite this Article: Gogoi, Minti and Baruah, Bulbul (2015). Value addition of mulberry silk with Indian willow (*Salix tetrasperma). Internat. J. Appl. Home Sci.,* **2** (1&2) : 8-15.

thestudy was designed to extract dye from Indian willow plant and dyeing of silk yarn under optimized condition. Secondly to isolate and characterization of colored compounds present in the dye.

METHODOLOGY

Materials :

Selection of dye yielding plants:

The bark of Indian Willow (*Salix tetrasperma*) had been collected from the mature plants(in summer season)mainly from Jorhat district ($26^{o}4^{/}$ N latitude and $94^{0}12^{/}$ E longitude) of Assam, India (Fig. 1).

Fig. 1 : Indian willow plant

Selection of mordants:

The natural dye require a substance which act as a colour intensifier and help in chemical bonding between dye and fiber, thus help the fixing of colour to the material known as mordant. Eco-friendly mordant alum (aluminum potassium sulphate) and natural mybrobalan (*Termilini achebula*) were used for the study.

Silk yarn :

The mulberry silk material collected from the Sericulture farm of Sivasagar (Gaurisagar) Assam.

Chemicals and reagents :

Reagents used for the study were the sodium carbonate (Na_2CO_3), ethanol (C_2H_6), methanol (CH_3OH), Dichlromethene (DCM) chloroform ($CDCl_3$) and acetone. Hydrocloric acid (HCl)and concentrated Sulphuric acid (H_2SO_4) is also used as LR grade chemicals. The chromatographic material silica gel (Chromatographyic grade 100:200 mesh for column) Solvent like hexane(C_6H_4), Ethyl acetate($C_2H_4O_2$), and TLC plates were extensively used for the study. Distilled water was used for dye extraction procedure and preparation of mordent solution, and the ionized water was used for dyeing and washing procedure.

Methods :

Preparation of yarn for dyeing :

Silk contains sericin which interfere the dye absorption, hence degumming of silk was done with

soap and detergent (Deulkar, 1973).

Preparation of dye powder :

The bark of the Indian willow plant wasair dried and grinded with an electrical grinder to make fine particle as the smaller particle gives better result in dyeing of silk textiles (Fig. 2).

Fig. 2 : Bark of the plant and powered dy

Size reduction:

During dyeing process dye molecule has to be entered in to the inner molecular space of the fibre in which it has to be fixed .A decreased particle size shorten the time required for dyeing and can provide improved dispersion in dyeing process and there is increase color strength in equilibrium. The size of grinded powder from the bark of the plant were passed through a Sieve Analyzer to keep the size of the grinded powder at acceptable range between 50-100 mesh (Gulrajani, 2001).

Extraction of dye :

Alkaline medium was selected as the best medium for dye extraction (Bansal *et al.*, 2006 and Bhuyan *et al.*, 2013) The dye was extracted by alkaline method at 0.5% Na_2CO_3 (sodium carbonate) at optimized condition (Devi *et al.*, 1999, AICRP).

pH of the dye :

The pH of the dye solution was measured before carry out the dyeing process as the alkaline pH should be avoided for dyeing of protein fibers (Bhuyan, 2004).

Dyeing of silk :

The wet silk samples were dyed in the extracted dye solutions for 45 minutes at 80^0C using the M:L 1:30 at a pH- 7. To obtain different colour shades mordanting was done simultaneously with two different mordants. The dyed yarn was washed in clean water allow it to dried in room temperature (Fig. 3 and 4).

Determination of absorption (%) and color strength :

The absorption of dye solution was recorded by measuring by optical density of the dye liquor at the wavelength 510nm (where it showed maximum OD value)for each case. The percentage dye absorbance were calculated as given below :

$$\text{\% of dye absorption:} \frac{\text{OD of the liquor before dyeing} - \text{OD of the liquor after dyeing}}{\text{OD of liquor before dyeing}} * 100$$

Fig. 3 : Dye solution before dyeing

Fig. 4 : Dye solution after dyeing

Further the K/S value of the dye sample and the dyed yarn were evaluated by light reflected technique and the value were assessed using the Kubelka-Munk equation :

$$\frac{K}{S} = \frac{(I-r)^2}{2R}$$

where, R is the observed decimal fraction of the reflectance of dyed yarns

K is the absorption coefficient and S is the light scattering coefficient.

Measurement of color value :

The Hunter co-ordinates CIE L, a and b, values were ascertained for two mordants and it was calculated from the tristimulus values (x,y,z) and were converted to CIELab co-ordinates.

Color Fastness properties:

The other criteria, the depth of colour, coloureveness and colour fastness to washing, and sunlight were tested by standard test methods (ASTM Standard,1968).

Isolation of dye compounds :

For isolation of color compounds the 50 gm of ethanolic extracted of dye was concentrated through a rotary evaporator at 45^0C to get a semi dry mass of the components. Initial screening was performed by TLC. Then the sample were injected in chromatographic column packed by silica gel (100-200mesh) eluted with mixture of ethyl acetate and hexane and two pure compounds compound 1 and compound 11 were isolated.

Characterization of isolated compounds :

The isolated dye compounds weresubjected to wavelength scan in the UV visible spectrophotometer (Specord-1) at a wavelength 200-800 nm .For identification of its chemical nature and functional groups present in the dye (FTIR)spectras of extracted compounds were recorded with $CDCl_3$ using spectrophotometer. Further, ^{1}H NMR and ^{13}C NMR spectra were measured at 25^0C on Bruker AV-DPX-300 MHz FT and AV Avance III 500 MHz FT NMR respectively. Then the MS Spectra of the compounds were recorded through GC-MS system, model Trace DSQ GC-MS spectrophotometer.

RESULTS AND DISCUSSION

The finding of the study was discuss below :

Color of the dye :

Mordant play an important role in imparting color in the yarn (Kamel *et al.*, 2009) hence variation of color were found in different mordants. The dye from Indian willow bark gave a very pleasing reddish brown color in presence of myrobalam and slightly light brown color in silk in presence of alum. Both the mordants were applied by simultaneous mordanting method.

Sr. No.	Name of mordant	Dyed silk yarn
1.	Myrobalam	
2.	Alum	

Fig 5 : Indian willow with myrobalam and Alum

Color strength :

The color strength value (K/S)was found higher (29.74%) in case of myrobalam mordant and less (24.91%) while mordanting with alum.

Brightness of color :

The table shows that higher value of L* Shows lighter shade and lower value showed deeper shade and the higher value of a* and b* indicated brightness due to red and yellow and negative value of b* indicated greenness and blueness. According to Vankar (2009), alum, being alkaline in nature, removes the color and yields lighter shades with

Table 1 : Brightness of Indian willow dye on mulberry silk

Mordants	L*	a*	b*
Myrobalam	18.89	70.73	7.98
Alum	49. 75	7.94	-51.91

Fastness properties of dye :

The color fastness of silk fabric to light fastness and washing and c were found to be good, after series of assessment done by a comparison with International Gray Scale of grading sample from 1-5.

1-very poor, 2-poor, 3-fair, 4 -good, 5- very good

Good wash fastness was achieved in simultaneous mordanted silk between fastness grade of 4 and 5 whereas rubbing fastness ranging between 3.5 - 4 was good. Average to good light fastness was observed in dyed silk with this dye . It was noted that the auxochrome of the dye, rate of the diffusion of the dye and the state of the dye inside the fiber are responsible for fastness properties of each dye. (Jothi, 2008).

Characterization of compounds:

The yellowish colored compound I , as isolated through column chromatography showed UV/

Table 2 : Color fastness properties of dyed samples

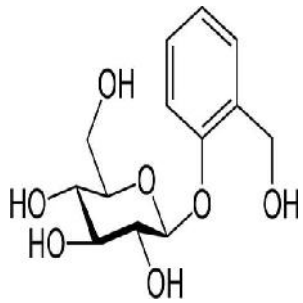
Sr. No.	Mordant used	Sunlight	Washing	
			CC	CS
1.	Without mordant	3	4	5
2.	Myrobalan	5	5	5
3	Alum	5	5	5

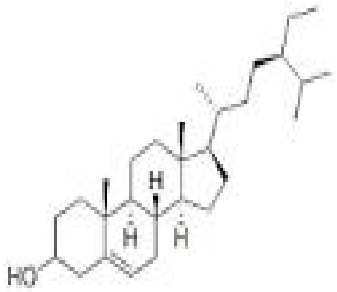
CC :Colour change; CS : Colour staining;
CC Ratings: 1 = very poor, 2 = poor, 3 = fair, 4 = very fair, 5 = good, 6 = very good
CS Ratings: 1= heavily stained, 2= considerably stained, 3= noticeable stained, 4=slightly stained,5= negligible or no staining

vis absorption spectra at 260 nm. These value falls in the spectral range of Flavonoids**.** The IR spectrum of isolated compound showed peak at 1400 $cm^{-}1$ which indicate the presence of aromatic -C=C-.The ^{1}H NMR ($CDCl_3$)spectra display Peaks in the region 5(1H,d,J-13.1Hz H-.7),4.76(1H, J-12.6,Hz,H-7), 7.01 (1H, t.J-7.4Hz, H-4),7.24 (1H t.J-6.8Hz.H-5). Whereas the mass of the compound was m/z 286(M+). for On the basis of above data the colored compound I was identified as salicin with its chemical formula $C_{13}H_{18}O_7$ .The similar finding was Salicin, the major phenic glycoside present in the bioactive extracts in Salixspecies(Karawya *et al.*, 2010).

For compound II, UV spectra showed peak at 285 nm., IR max 3490 cm^{-1} which indicate the presence of (-OH) group, and the absorption at 1660 cm^{-1}(-C= C-,stretching). The ^{1}H NMR spectra shows peaks at ä 1.39(3H *J* 6.5Hz 29 Me) 2.75(IH ,m, 6H) 4.9(1H,m,28H)which indicating the presence of CH_3 group .The mass spectrum of the compound showed the molecular ion peak at m/z 424(M+).All these confirmed compound II is β –Sitosterol[2],with molecular formula $C_{29}H_{50}O$.

The structures of above isolated compounds are given below :





Salicin –Sitosterol[2]

Among two different isolated compounds, salicin extracted from dye is mainly responsible for color yielding properties of the plant and it was consider as a most important compound for many of the medicinal products. Salicylate, is an organic compound that was historically derived from the Soaponification of Salicin found in the bark of the willow tree. Salicylate itself only absorbs in the Ultraviolet region of the spectrum, creating a bit of an experimental challenge. Salicylate derivatives like Aspirin, acts as an analgesic and anti inflamatory. Topically it is used against acne, psoriasis, corns and wars. “It works as both a keratolytic and a comedolitic agent by causing the cells of the epidermis to shed more readily, opening clogged pores and neutralizing bacteria within, preventing pores from clogging up again by constricting pore diameter, and allowing room for new cell growth.” Hence it is widely used as an active ingredient in face washes (Wikipedia). Karawya *et al.* (2010) reveals that Salicin, is considered to be the pharmacologically active principle due to its structure

similarity to aspirin and possess a significant anti-inflammatory activity with different degree.Since the color compound salicin have high medicinal value,coloring of Textiles with this compound must have immence importance in the Textiles Science as well as it will help in maintaining a green environment in the world.

Conclusion:

Many of the plants grown in different geographical climate contain coloring compounds in their structure which have higher compatibility with our environment but they are yet to be explore. However in light of the growing demand for natural dye among the Green-minded consumer, large and small-scale industries have begun exploring the use of natural colorants as a possible means of producing an ecologically sound products. In order to move with the ecological movement on earth, while we think about the commercialization of natural dye, only the characterization of chemical components of dye will help to understand the whole mechanism of dyeing on different yarn and fabrics .It also help in structural modification of some of the chromophores groups present in dye molecules to obtain best quality dyeing on textiles.

Acknowledgement :

I am grateful to Dr. D. Ramiah, Director and all the members of Natural Product Chemistry, Cellulose Pulp and Paper Division and the Analytical Division of the NEIST, Jorhat, for their help in isolation and spectral analysis of plants for identification of colored compounds.

REFERENCES

AATCC Technical manual (2008). American Association of Textile Chemists and Colorists, (AATCC), Vol: 83.

Annual Report(2004). All India Coordinated Research Project in Home Science. Assam Agricultural University. Jorhat.

Bhuyan, R and Saikia, C.N (2002). Isolation and Characterization of Natural Colouring material (Natural Dye) from selected plant sources and studies of their Characterization. Ph.D.Thesis. Dibrugarh University. pp. 172-173.

Cardon Dominique (2010). Natural Dyes, Our Global Heritage of Colors, Textile Society of America Symposium Proceedings, Textile Society of America.

CIE Publication 15.2(1986). HunterLab Measure color-measure quality, Insight of color, July 1-15 1996,vol.8, No .7

Deulkar, D.(1983). Household and Laundry work published by Atma Ram and Sons, Delhi, Lucknow.pp 43, 59-61

Gulrajani, M.L.(2014). Technology for production and Application of natural Dyes on Textiles" IWND 2014. A value Chain in Natural Dyes. Pub by Saifabad, Hyderabad A.P, India PP. 89-96.

Jothy, D. (2008). Extraction of Natural dye from African Merigold flower (TagetesErecta) for Textile coloration"Autex Research Journal . Vol.8 No-2 June 2008.

Kubelka, P (1954). New Contributions to the Optics of Intensely Light-Scattering Materials PartII:Nonhomogeneous Layers, *JOSA*, **44** (4) : 330-335.

Karawya, S.M. *et al.* (2010). Some medicinal plants growing in Egypt. *Medical J. Islamic World Academy of Sci.*, **18** (4) : 139-150

Kamel, M.M., Hemly, H.M. and Hawary, N.S. (2009): Some studies on dyeing properties of cotton fabrics with *Crocus sativus* (Saffron) flowers using an Ultrasonic method. AUTEX Res. J.,

Sarkar, A.K. (2004). An evaluation of UV protection imparted by cotton fabric dyed with natural colorants," *BMC Dermatology*, **4** (15) : 1–8.

Singh, O.P. (.2000). Natural Dye; The Pros and Cons" *The Indian Textile J*., **109** (1) : 42-45.

Vankar, P., Shanker, R. and Samudrika W. (2009): Utlizatation of temple waste flower – Tagetuserecta for dyeing of cotton, wool and silk on industrial scale. *J. Textile & Apparel, Technol. & Mgmt.*, **6**: 1-15.

****