ISOLATION AND PURIFICATION OF LACCAIC ACID DYE FROM STICK LAC AND STUDY OF ITS (COLOUR FASTNESS) PROPERTIES AND REFLACTANCE ON SILK FABRIC DYED WITH HEAVY METAL MORDANTS

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Abstract:

Dye from stick Lac was extracted, isolated and purified to remove wax and shellac which were used as by products. Dye extracted was purified and applied on silk fabric using four different heavy metal mordants (Potash alum, potash chrom alum, copper sulfate and ferrous sulfate) by using metamordanting method of dyeing. Colour fastness properties (colour fastness to washing, rubbing, light and heat) were studied with the aid of grey scale for metamordanted and control silk fabrics. Reflactance of all the dyed fabrics was also noted. λ_{max} and absorbance of dye were determined by spectrophotometers. Results showed that mordant dyeing increased the uptake of dye on silk fabrics. The results of colour fastness properties were from best to excellent.

***Key words*:** Mordant, reflectance, Isolation, Wax, Shellac.

Introduction

A dye can generally be described as a coloured substance that has an affinity to the substrate to which it is being applied. It adheres to the fabrics like cotton, silk, linen, woolen, leather and many other materials. Dyes are of two types i) synthetic dyes and ii) natural dyes. As synthetic dyes cause environmental problems in textile and leather processing industries which are big environmental polluters in the World. It is the need of hour to use dyes and chemicals which are environment friendly and are less polluting (Nisar et al., 2007; Waheed and Alam, 2004; Mahanta and Tiwari, 2005).

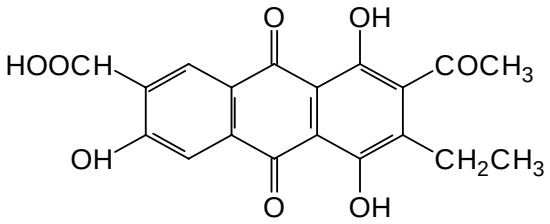
Natural dyes have been known for a long time. These dyes not only come from flowering plants and vegetables but also from animal and insect sources like fungi, lichens and insects like shellfish (Padma and Vanker, 2007; John and Cannon., 1994).

Natural dyes produce different suitable and soft colours to the brightness colours on the yarn and fabrics and are gaining momentum day by day (Win and Swe., 2008; Bechtold et al., 2008).

Insect dyes are obtained from the body of the insects. They live on different plants to get food colour or dye produced in the abdomen of the insect. The ancient dye stuff lac is obtained from the dried bodies of an insect coccus laccae (Laccifer Lacca) found on twigs of certain trees native to South Asia like India and Cyclone which secretes Lac resin (Chairat., 2009., Khalid et al., 2008, Bechtold et al., 2008., http//wikipedia/Lac).

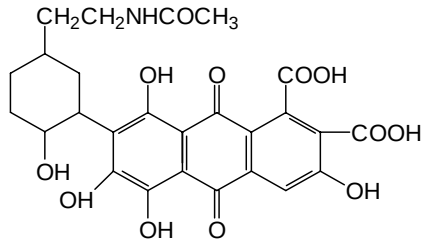
The word Lac is presumably derived from Hindui word Lakh meaning “one hundred thousand” or “A great Number” having reference to the fact that a great, many of these insects had to be gathered and collected in order to produce any quantity of dye (http://Scribed/Lac 1). In general preparation of the dye from the insect it took 70,000 insects to make one lb of dye commercially it was provided by Mexcer.

Lac is chemically very similar to carmine, carminic acid kermes or kermesic acid based partially on these findings, the following structural formula for laccaic acid has been purposed.

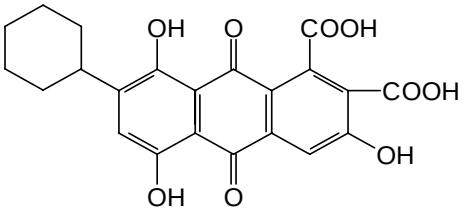


However, this structure has not yet been definitely proven. This formula was studied by Schmidt in 1887 and investigated the colouring matter of the lac dye and gave it the name of Laccaic acid. Latter on it was found that there are four compounds found in lac designated as A,B,C and D. (Chairat., 2009)

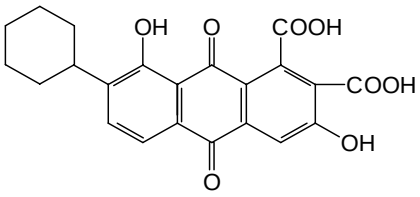
Laccaic acid A is most abundant and the major compound (Kongkachuichay et al., 2002a, 2002b; Zahurul et al., 1998). Laccaic acid A,B & C are very similar but differ at a single point that is illustrated in the following formulas. Laccaic acid D is also called Xanthokermesic acid and closely resembles kermesic acid in structure:



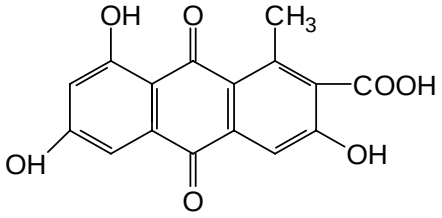
Laccaic Acid A



Laccaic Acid B



Laccaic Acid C



Laccaic Acid D

It is noticed that structures A,B & C differ at a single point only.

Lac dye and Shellac has many applications in different fields. Lac dye is natural food pigment because of it is FDA approval from FDA. Shellac is used to coat apples and other fruits to make them shiner to improve its shelf life. It is used in cosmetics for making lipsticks and eye brow pencils and in sunscreen emulsions (Karim et al., 1997). Lac dye is used for dyeing the fabrics usually wool and silk (Kamel et al., 2005, 2007; Singh et al., 2005).

Shellac is the byproduct obtained and is used in varnishes extensively in food finishing, coating of candies and coating of pharmaceutical tablets to protect against moisture and to seal off active ingredients. It is used to some extent as a leveling resin in some formulations, printing inks due to new packing technology. The electric insulating industry still consume large quantity by shellac is still used for heat-

proofing. It is used in vilons and other varnishes and is soluble in alcohol (http//en.wikipedia.org/wiki/Lac)

So use of natural dyes particularly insect dyes is gaining interest for dyeing and application in biological staining (Kamel et al., 2007). Pure insect dyes are highly valuable. Pakistan policy makers should show interest in propagation and applications of these dyes as they are environment friendly and create source of income for people living in rural area.

Present work includes the extraction and of laccaic acid, study of its applications on silk fabrics and its colour fastness properties (Khalid et al., 2010; Ali et al., 2009; Liu et al., 2005).

As natural and organic mordants applied on silk fabrics do not give so fast colours and colour fastness properties. So heavy metal mordants are used Colours obtained were sharp, bright, soft and colour fastness properties observed were enhanced and increased (Weheed & Alam, 2004; Deo and Desai 1999).

Silk fiber is protein fiber and is produced from silk worm. Most natural red colour dyes are highly soluble in water. So colour fastness to washing of lac dye was found quite low. In order to improve its colour fastness properties heavy metal mordant method was conducted. In which metal ions can act as acceptors to electron donors to form coordinate bonds with dye molecule which is insoluble in water (Kongkachuichay et al., 2007; Waheed & Alam 2004).

Most of the colour of the plant origin are anthrocyanides, flavonoid and caratenoids.

Materials & Methods:

Analytical Equipments Used:

IR spectrometer model “bruker OPUSTUM Nicolet IR 200 (USA). D400 IR dyeing machine (SDL Atlas England). Launderometer (Roaches). Oven Ci 3000 – Xenon. Weatherometer, (Atlas England). Water Bath. Grey scales for staining (ISO 105 A03). Grey scale for change in shade (ISO 105 A02). Crockmeter (SDL Atlas England). Multifiber (DW). Twiglac.

Chemicals:

Potash alum, Potash chrome alum, copper sulfate, ferrous sulphate, detergent, SDC standard soap (without optical brightener), distilled water, sodium carbonate, sodium chloride (AR Grade).

Extraction & isolation of Lac Dye

Source: Fresh twig lac was plucked and collected in the month of June from the branches of beri (Zizzyphous) tree which were found to grow in campus of PCSIR Phase-I, Housing Society, Canal Road, Lahore. Many branches were infested with insect laccifer lacca.

Lac (Shellac containing lac) was separated from the twigs and branches manually with hands and spatula. Fibrous material of branches, twigs, debris and insects were also picked up by hand screening.

500 gms of natural shellac thus obtained was coarsely ground in mortar and added 5000 ml of water. Stirred it well for 3 to 4 hours. It was then filtered through coarse mesh sieve and washed the residual lac on the sieve with 100 ml water. The dark red filterate solution was filtered through Buckner funnel, covered with filter cloth. The dye solution was finally centrifuged till clear solution of red dye is obtained. The clear solution is decanted carefully into conical flask (Khalid et al.,, 2010; Bechtold et al., 2008, Oenal et al., 2004;Kongkachuichary et al., 2002; Oparah et al., 2001).

The clear red filtrate was acidified with conc hydrochloric acid to pH 1-2 till red dye was precipitated. It was stirred for 2-3 hours by magnetic stirrer then centrifuged. The precipitated dye was separated from supernatant liquor. The precipitated dye was removed and transferred to petri dish for drying. The dried dye had still waxy touch, so finally it was extracted with hexane to remove any waxy material. Laccaic acid dye 3.4% was obtained by applying above mentioned process.

Isolation of wax, aleuritic acid from free lac.

Dye free lac (residual lac) was extractred with hexane in the soxhlet apparatus. Hexane soluble material containing wax and aleuritic acid was made free from hexane by distillation. The hexane free solid material was taken in diethyl ether and added ethanol drop wise, wax was precipitated and separated from the solvent mixture. The

solvent was distilled off and white fluffy material which may be aleuritic acid was obtained.

Dyeing with Lac Dye:

Dyeing is the process of colouring textile fibres and other materials, in such a way that the colouring matter becomes an integral part of materials (fabrics etc.) rather than a surface coating. Dyeing was completed with 2% lac dye solution without and with mordant (metamordanting technique). The liquor ratio of (fabric and water) during dyeing was 1:20. Before dyeing silk fabrics were wetted with wetting agent. Temperature was maintained at 100^{o}C. Dyeing was completed in 1 hour. After dyeing silk fabrics were washed, squeezed and dried at room temperature.

Dyeing with Mordants:

The processing agent which can make link between cloth and dye is known as mordant which is derived from a Latin word 'mordate' (Sundrarajan M., et al., 2009) to bite. Mordant may be added in three different ways (1) Premordanting when fabric is treated with mordant before dyeing (2) Metamordanting in which mordant is added during dyeing of fabric and (3) Postmordanting when fabric is treated with mordant solution after dyeing. Metamordanting technique was used during this study using heavy metal mordants (Potash alum, potash chrome alum, copper sulphate and ferrous sulphate) (Deo & Desi, et al., 1999, Zaman et al., 1993). Wet fabrics were dipped into 2% lac dye solution. The liquor ratio during dyeing was 1:20. The temperature of dye bath was maintained at 100^{o}C and total time for dyeing was 1 hour. After 45 minutes of dyeing different mordants of 0.1M strength were added to the dyeing bath for fifteen minutes, then fabrics were rinsed, washed and squeezed and dried at room temperature.

Colur fatness to washing

Dyed silk fabrics and multifibre DW were cut into 4x10 cm size pieces and sewed with each other from one side in such a way that their faces were adjacent to each other. Soap solution made by adding 5 gm soap per litre of water, under specified conditions of time and temperature, were added to different steel containers fixed in launderometer having liquor ratio 50:1. Change in colour of the specimen and staining

of adjacent fabrics (DW multifibre) was assessed with the help of grey scale. Results of colour fastness to washing are shown in table-1. (BS 1006; 1990)

Colour fastness to dry and wet rubbing

Colour fastness to dry and wet rubbing of metamordanted dyed silk, fabrics were performed with the help of crockmeter instrument. For this test different silk fabrics of 5 cm x 14 cm were fastened by means of clamps to base board of the testing device. The standard cloth was first rubbed in dry condition and then in wet condition in to and from motion in a straight line along a track 10 cm long on surface of fabrics 10 times in 10 seconds with a downward force of 9 N. Fabrics were dried at room temperature. Change in shade of the dyed fabrics and staining of the rubbing cloth was assessed with the help of grey scale. The results of colour fastness to rubbing are given in table-II. (BS 1006; 1990)

Colour fastness to light

Colour fastness to light was carried out with instrument weatherometer by Atlas according to ISO 105 standard procedure B02, in which Xenon arc lamp was used as an artificial light source, representative of natural day light D65. Silk fabrics of measurement 7 cm x 12 cm were exposed to D65 light and result of colour fastness to light against blue wool were noted with the help of grey scale. Change in shade was noted . Results obtained are tabulated in table-II.

Colour fastness to heat

Colour fastness to dry heat fastness for all silk fabrics was performed by hand iron. Silk fabrics were pressed for five minutes at specific temperature with and without mordants. Results are shown in table-II.

Reflectance of Silk fabrics

IR spectrometer, model “Bruker OPUSTM Nicolet IR 200 (USA) was used for recording reflectance in the infra region for silk cloths dyed with lac dye with and without mordants. The spectrum of laccaic acid dyed fabrics was recorded on the above mentioned IR spectrometer. (Shamas – Nateri., 2008)

Procedure

Silk fabrics were put on the lens of IR spectrometer and noted the reflectance peaks of the dyed silk fabrics. Results are given in table-III.

Results & Discussions

The grey scale results of the silk fabrics dyed with lac dye (control) and dyed with different heavy metal mordants are given in table-I & II for colour fastness to washing, dry and wet rubbing fastness, light and heat.

Table-I: COLOUR FASTNESS TO WASHING FOR SILK FABRIC DYED WITH HEAVY METAL MORDANT AND WITHOUT MORDANTS (CONTROL)

Sr.#	Observed dyed fabric	Diacetate	Cotton	Nylon	Polyester	Polyacrylic	Wool	Change in shade
1.	Control	4	3-4	5	4-5	4-5	4	2-3
2.	Potash alum	4-5	4	5	5	5	4-5	3-4
3.	Potash chrom alum	5	4-5	5	5	5	4-5	3
4.	Ferrous sulfate	4-5	4	5	5	5	4-5	3
5.	Copper sulfate	4-5	4	5	5	5	4-5	2-3

Colour fastness to washing was observed for control silk fabric (dyed with lac dye on silk) and silk fabrics dyed with four heavy metal mordants using meta mordanting method respectively. For control fabrics the values for colour fastness to washing did not gave good results, because dyeing on fabric does not adhere on the fabric, resulting light colour shades with different concentration of dye. Application of different heavy metal mordants not only increase the depth of shade on fabrics but also change the colour of the dye. Potash alum gave move (purplish) colour. $CuSO_4$ gave grayish move colour while potash chrome alum gave dark pinkish move colour. Reason is that heavy metal mordants increase the binding capacity of the dye to the fabric.

So in metamordanting process of dying the dye is absorbed on the fiber followed by the formation of an insoluble complex with metal ions showing bathochromic shift. So some of the dye is lost because of formation of this insoluble complex in the dye

bath itself. So this phenomena bring about a decrease in the effective dye concentration in the dye bath.

For washing tests all the values for change in stain and change in shades were noted and are shown in table-I.

Change in staining:

On diacetate staining strip of multifibre, (DW) lac dye control gave good (4) rating, while potash alum, ferrous sulfate and copper sulfate mordanted silk fabrics gave best (4-5) rating while excellent results rating (5) were observed with potash chrome alum. On cloth staining strip of multifibre gave rating (3-4) satisfactory results for control (fabric dyed with lac dye) where no mordant was used. Potash alum, potash chrom alum, ferrous sulfate and copper sulfate gave good (4) rating on grey scale. On Nylon strip of multifiber all the five silk fabric dyed with lac dye and silk fabrics mordanted with potash alum, potash chrom alum, ferrous sulfate and copper sulfate gave excellent rating i.e. (5). Polyester strip of multifber showed (4-5) best rating with control fabrics and (5) excellent rating with potash alum, potash chrom alum, ferrour sulfate and copper sulfate mordants silk fabrics. Polyacrylic strip showed same results as for polyester stip, while wool strip gave (4) good rating with control silk fabric and (4-5) best rating with other four metamordanted silk fabrics.

Change in shade

When change in shade of original dyed fabrics (control) and mordant treated fabric, was observed, it was found that control fabric (dyed with lac dye) gave (2-3) i.e. poor rating potash alum mordanted silk fabric gave (3-4) i.e. satisfactory rating. Potash chrom alum and ferrous sulfate mordanted silk fabrics gave (3) rating while copper sulfate mordanted silk fabric also gave 2-3 poor rating.

Results of grey scale for rubbing fastness, light fastness and heat fastness

Rubbing fastness was performed according to ISO 105 X 12 by ISO method with instrument crockmeter. Both dry and wet rubbing along warp and weft were noted with standard cloth fabric. Similarly light fastness was performed according to ISO 105 standard procedure B02 while heat fastness with hand iron.

Table-II: RESULTS OF RUBBING FASTNESS, LIGHT FASTNESS AND HEAT FASTNESS ON SILK FABRICS DYED WITH AND WITHOUT HEAVY METALS MORDANTS

Sr.#	Name of fabric	Rubbing fastness				Light fastness	Heat fastness (Dry)
		Dry rubbing		Wet rubbing			
		Warp	Weft	Warp	Weft		
1.	Control	4-5	4-5	4	3-4	4	4
2.	Potash alum	5	5	4-5	5	4-5	4-5
3.	Potash chrom alum	5	5	4	4-5	4-5	4-5
4.	Ferrous sulphate	5	5	4	4	4	4-5
5.	Copper sulphate	4-5	5	4-5	4	4-5	4-5

Results of Dry rubbing fastness:

Dry rubbing fastness of lac dye gave best (4-5) results along warp and weft, while with potash alum, potash chrom alum and ferrous sulfate mordanted silk fabrics gave excellent (5) results on grey scale along warp as well as weft. With CuSO4 mordant, dry rubbing gave best (4-5) results along warp and excellent (5) results along weft side of the silk fabrics.

In all the cases where rubbing was performed with control fabric gave low grey scale rating and low quality level of dyeing as compared to rubbing performed by the application of metal mordants.

Wet rubbing fastness:

Wet rubbing fastness of control fabric gave better (4) rating along warp and satisfactory (3-4) rating along weft. Potash alum gave best (4-5) rating along warp and excellent rating (5) along weft potash chrom alum and ferrous sulfate both gave good (4) rating along warp and (4-5)best, (good) (4) along weft respectively. Copper sulfate gave (4-5) best results along warp and 4 (good) along weft side of the silk fabric.

Results of colour fastness to light:

According to table-II colour fastness to light with control fabric gave 4 (good) results. Potash chrom alum and copper sulfate dyed silk fabrics gave (4-5) best, rating for colour fastness to light.

Results of colour fastness to heat

Colour fastness to heat gave (4) better rating for control fabric (dyed with lac dye soln.) and best results for potash alum, potash chrom alum, ferrous sulfate and copper sulfate mordanted silk fabrics. Results of washing fastness light fastness and colour fastness showed low grey scale rating with control silk fabrics dyed with lac dye.

Results of reflectance peaks

IR reflectance peaks of spectra of dyed fabrics with and without mordants are given in Table-III.

Table-III: RESULTS OF REFLECTANCE PEAKS OF DYED SILK FABRICS WITH AND WITHOUT MORDANTS

Dye	Frequency cm^{-1} Dyed silk with mordant				Dyed silk without mordant	Intensity	Assignment
	A	B	C	D			
550	640	665	520	700	724.41	Var	04
1000	1000	1000	1040	1000	1016	WK	Amine
-	1090	1100	1100	1110	1094	-	-
1260	1240	1240	1210	1280	1238	Med	Carboxylic acid
1400	1400	1410	1430	1410	1408	Str	C=O
1630	1690	1690	1590	1520	1505	Med	Arene
-	-	2430	-	-	2360		
2850	2700	-	2890	2890	-		
2950	-	-	2950	2950	-		
3300	-	3000	-	-	-		

A=Chrom alum, B=Copper sulphate, C=Ferrous sulfate and D=Potash alum.

Conclusion

The use of natural dyes, particularly insect dyes is gaining interest again for natural dyeing and other applications in pharmaceuticals and cosmetics. Present results showed that mordanted dyeing with lac was more applicable because colour uptake on silk fabrics increased and results were best to excellent. Lac dye is environment friendly and create source of income for people living in rural areas. So Pakistan policy makers should give interest in propagation of these natural dyes.

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