

COLORING OF LEATHER USING HENNA - NATURAL ALTERNATIVE MATERIAL FOR DYEING

by

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ABSTRACT

Synthetic dyes are widely used in the leather industry; some of these are carcinogenic in nature. Environmental concern leads one to seek dye products based on natural resources, which have several advantages like less toxicity, biodegradability, etc. The research work presented in this paper focuses on an eco-friendly dyeing process of the leather using aqueous henna leaves extract that contain lawsone (2-hydroxy 1,4 naphthaquinone) the most prominent member of alpha – hydroxynaphthaquinone, natural dye class. The aqueous extraction from the henna leaves was studied and the best extraction conditions were found to be the maceration of the ground henna leaves in water, at temperature (> 80°C) for 60 minutes in a water bath without agitation. The color of the leather had become deeper with increasing amount of henna extract and multiple colors have been obtained depending on the type of mordants used. The henna dyeing had resulted in leathers with good rub (dry and wet) and perspiration fastness characteristics, while the light fastness characteristics were found to be moderate.

RESUMEN

Colorantes sintéticos son ampliamente utilizados en la industria del cuero; algunos de ellos siendo cancerígenos por naturaleza. Preocupación por el medio ambiente conduce a buscar productos colorantes en base a recursos naturales, cuales tienen varias ventajas como menos toxicidad, biodegradabilidad, etc. La investigación presentada en esta obra se enfoca en un proceso de teñido ecológicamente amistoso para el cuero que utiliza extractos acuosos de hojas de Henna [*Lawsonia inermis*], las cuales contienen *lawsina* [ácido henotánico: C₁₀H₆O₃], 2-hidroxi 1,4 naftoquinona, el miembro más promisorio de las clases naturales de colorantes, -Alfa- hidroxinaftoquinonas. La extracción acuosa de las hojas de Henna fue estudiada y las condiciones óptimas se determinaron para las hojas maceradas en agua, a temperatura (>80°C) durante 60 minutos en un baño sin agitación. El color del cuero se convirtió más intenso con la aumentada oferta de extracto de Henna y múltiples fueron obtenidos dependiendo del tipo de mordientes utilizados. El teñido basado en Henna resultó con buenas solidez al frote (tanto húmedo como seco) así como de características de solidez al sudor, mientras que las características de solidez a la luz se encontró moderada.

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INTRODUCTION

Many plants and some animals have been identified as potentially rich in natural dye contents. Normally natural dyes are extracted from the roots, stems, barks, leaves, flowers and fruits of various plants¹. There are two general groups of dye materials, substantive and adjective dye. Substantive dyes are permanent when heated with fibers while adjective dyes require addition of chemical substance, a mordant to facilitate the absorption of color for increased permanence². The natural dyestuffs can be categorized based on their chemical structures into flavonoids, xanthenes, quinones, carotenoids, chlorophyll, betalains, phycobilins, melanins, o-acylphenols, aromatic ketones, pyrone phenazines, phenoxazinones and indigo, which are important natural coloring matters³. Most natural dyes are yellow and brown; some with multicolor capacity have chemical structures similar to vegetable tanning materials like catechol and pyrogallol⁴. Dyes from vegetable sources are interesting for two main reasons. First, the color of the dyes is very brilliant and second, the toxicity of dye is very low⁵. The various advantages of natural dyeing materials include lower toxicity, non allergic and non carcinogenic, higher compatibility with the environment, and their better biodegradability⁶. The natural materials develop colors in combination with inorganic mordants for example Fe, Co, Ni, Al, Cu, and Sn^{7,8}. The natural dyes require mordants to fix to fibers and to prevent the color from either fading with exposure to light or washing out. These compounds are of three types, metallic mordants, tannins and oil mordants. The mordants help binding of dyes to the fiber by forming chemical bridge between the dye and the fiber, thus improving the staining ability of a dye along with increase in its fastness properties. Poor light fastness of some of the natural dyes is attributed to photooxidation of the chromophore. Photo oxidation can be prevented or minimized by the formation of a complex between dye and transitional metal. The wash fastness of natural dyes can be improved by treatment with mordants such as aluminum sulphate, ferrous sulphate, copper sulphate, and zinc sulphate.

Lawsonia inermis (Henna) is a member of the family Lythraceae which consists of about 500 species, widely spread in tropical regions with relatively few species in temperate regions⁹. *Lawsonia inermis* is generally considered as a native of Africa and Asia. It is widely cultivated in tropical regions of the world in Sudan, Egypt, China, and India. Major producing countries include Sudan, Egypt and India¹⁰. Henna plant grows on any type of soil, from light loam to clay loam, but does best on heavy soils, which are retentive of moisture. It tolerates a little alkalinity in the soil. Propagation is carried out through seeds and cuttings¹¹. Henna leaves have been extensively used for centuries in the Middle East, the Far East and Northern Africa as dye for nails, hands, hair and textile¹². Henna is also used in treating skin problems, headache, jaundice, amebiasis and enlargement of the spleen¹². Leaves of *Lawsonia inermis* provide an important cosmetic dye. *Lawsonia inermis* has been well

investigated phytochemically by various researchers. The occurrence of β - sitosterol flavonoids¹², glucoside¹³, quinoids¹⁴, gallic acid¹⁴, naphthalene derivatives¹⁵, coumarins¹⁶ and xanthenes¹⁷ in *Lawsonia* leaves has been reported. Earlier work establishes the use of henna as an alternative vegetable retanning agent¹⁸.

Aqueous solutions of henna are orange in colour and show green fluorescence. The principal coloring matter is lawsone (Figure. 1), 2-hydroxy 1,4 naphthaquinone ($C_{10}H_6O_3$, m.p 190°C) which is present in dried leaves at concentration of 1.0 – 1.4%. Besides lawsone, the other constituents present in henna are gallic acid, glucose, mannitol, fats, resins (2%), mucilage and traces of alkaloid. The flowers of henna have a strong aroma and on steam distillation yield 0.01 – 0.02% of essential oil. Henna oil has been used in perfumery, it consists mainly (90%) of α - and β - ionones (ratio 1:4), nitrogen compounds and resins¹⁰. Henna extract contains lawsone together with a high proportion of another colorant; when applied to wool and nylon 6.6, both henna extract and lawsone behaved as acid leveling, non-metallized acid dyes in so far as dye uptake increased with decreasing pH¹⁹. Also, henna has been used for dyeing tails and manes of horse¹². Henna was once extensively used for dyeing silk and wool. A wide range of colours may be imparted by treating henna dyed fabrics in acid baths containing potassium dichromate, ferrous sulphate, stannous chloride, or alum. Henna extracts are useful for dyeing in acid baths; alkalis intensify the colour but destroy the dyeing properties.

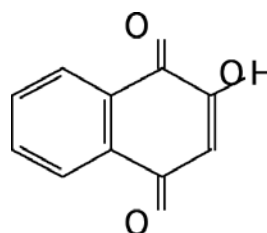


Figure 1: Structure of Lawsone (2- hydroxy – 1, 4- naphthaquinone)¹³

Leather is a difficult substrate to dye to level and consistent shade due to the unique nature of the raw material that has variations within the matrix²⁰. The effluent liquor generated from the dyeing process using synthetic dyes is a major concern because of its poor degradability. Earlier Burkinshaw and co-workers had shown that the use of a natural biopolymer, chitosan facilitating the enhancement of the exhaustion of different types of dyes²¹⁻²³. Natural dyes have been used for textiles for a long time, but their application to leather has been extremely rare. Such an attempt will result in the development of natural leather dyeing process. Leather can be dyed with natural dyes using, either dye alone in acidic or alkaline conditions or with a mordant, which may be applied before or after the dyestuff or simultaneously. In this work, an attempt has been made to study the influence of leather dyeing process using henna extract of *Lawsonia inermis*.

EXPERIMENTAL

Materials

Chrome tanned goat nappa crust (undyed) leathers were utilized to carry out the dyeing trials. Henna extracted from dried henna leaves collected from Sudan were used for dyeing experiments. Aluminum sulphate, ferrous sulphate and zinc sulphate used for dyeing process with henna extract as premordants and postmordants were of laboratory grade.

Preparation of Aqueous Extraction of Henna Leaves

The required amount of ground henna leaves was soaked in water (1:10 w/v) at temperature above 80°C in water bath for an hour, filtered through a piece of cotton cloth and the volume of the henna extract noted. Part of the henna extract was filtered through Whatman no.1 filter paper and 10 mL of filtrate was used for the determination of percentage total solubles.

Optimization of conditions for dyeing with henna

Goat nappa crust leathers were weighed, wet back overnight, washed, fatliquored, dyed with natural dye (aqueous extract) of henna leaves, fatliquored, fixed, washed and dried using the process mentioned in Table I. In order to optimize the dyeing with henna, parameters such as concentration of henna, pH of dyeing, and temperature of dyeing had been studied.

Concentration variations of Henna extract

Goat nappa crust leathers were weighed, wet back overnight, washed, fatliquored and dyed as per the process given in Table I (Experiment I) at different concentrations viz., 2.5, 5, 10, 15, 20 and 25% (based on crust weight) of aqueous extract (concentration) of henna leaves at constant room temperature (30°C) and pH 5. The process liquors were collected and estimated for the exhaustion of henna.

pH variations

Goat nappa crust leathers were weighed, wetted back overnight, washed, fatliquored, adjusted to different pHs viz., 4, 5, 6 and 7 and dyed with constant concentrations of henna (20%), at 30°C using the process given in Table I. The process liquors were collected and estimated for the exhaustion of henna.

Temperature Variations

Goat nappa crust leathers were weighed, wetted back overnight, washed, fatliquored, adjusted to pH 4; then dyed with 20% of henna using the process given in Table I at varied temperature viz., 30 and 60°C. The process liquors were collected and estimated for the exhaustion of henna.

Experimental trial with Henna dyeing using pre and post Mordants

The pre and post mordant experiments trails were carried out on goat nappa crust leathers using 2.5% metal ions viz., aluminum sulphate, ferrous sulphate, zinc sulphate with 20% henna at pH 4 and 60° C following the process mentioned in Table I.

Analysis of Henna Extract Exhaustion in the Process Liquor

Spent henna extract liquor was collected and analyzed for the dye concentration using a spectrophotometric method by measuring the absorbance value at 267nm (λ_{max} of the henna used), after suitably diluting the spent dye liquor using Cary 100 Perkin-Elmer UV-visible spectrophotometer.

% Henna extract exhaustion = $[(C_o - C_s)/C_o] \times 100$
where C_o is the concentration of henna extract offered and C_s is the concentration of henna extract in the spent liquor.

Conditioning of Leather

Leather samples for fastness characterization were cut from the official sampling position²³ and conditioned at temperature 20 ± 2 °C and 65 ± 2 % relative humidity for a period of 48hrs.

Determination of Fastness to Wet and Dry Rub

The resistance of the color of leather specimens to dry and wet rubbing was measured using standard method²⁵. The leather specimens were rubbed by dry or wet wool felt pads which were moved backward and forward under constant force. After a defined number of rubs, the damage to or transfer of color from leather specimens is assessed using grey scale.

Determination of Fastness to Perspiration

The resistance of the color of leather specimens to the action of artificial perspiration solution was measured using standard method²⁶. The leather specimens were kept in contact with standard undyed multifiber strip and immersed in artificial perspiration solution for 30 minutes so as to thoroughly wet the test assembly. The test assemblies were maintained at 37 ± 2 °C for 4 hours before separating and drying. The degree of staining to the individual components of the multifiber fabric was then assessed using the grey scale for assessing staining.

Determination of Fastness to Light

Leather samples dyed with henna extract without mordant, premordant leathers and postmordant leathers were cut from the official sampling position. The resistance of the color of leather specimens to an artificial light source, Xenon lamp, was measured using standard method²⁷. One side of the leather was exposed to light from a Xenon arc under prescribed conditions for 20 hrs, along with dyed eight blue wool standards having increasing levels of fastness. Black panel temperature was maintained at 63 ± 1 °C and the relative humidity was 30 ± 5 %. Fastness was assessed by comparing the fading of crust leathers with that of standards, from standard 1 (very low light fastness) to standard 8 (very high light fastness), with each standard being approximately twice as that preceding one. Rating was given on a scale of 1- 5 points, where higher points indicate better fastness.

TABLE I
Formulation of the dyeing process

Process	%	Product	Temperature (°C)	Time(min)	Remarks
Wetting back	250	Water	30	Overnight	
	0.4	Wetting agent			
	0.2	Ammonia solution			
Washing	300	Water (2 times)	30	20	
Fatliquoring	100	Water	60	40	
	2	Lipoderm liquor SCP* (Synthetic fatliquor)			
pH adjustment		Sodium bicarbonate/ formic acid		60	Adjusted to the required pH viz., 4, 5, 6 and 7
Dyeing	X	Henna extract	30/60	30	X= 2.5, 5, 10, 15, 20 and 25%
pH adjustment		Sodium bicarbonate/ formic acid		60	Adjusted the pH to 4.5
Fatliquoring	3	Lipoderm liquor SCP* (Synthetic fatliquor)	60	40	
	2	Lipoderm liquor SAF* (Synthetic fatliquor)			
Fixing	2	Formic acid	60	3X10 + 30	pH 3.5
Washing	200	Water		10	

* procured from BASF, India

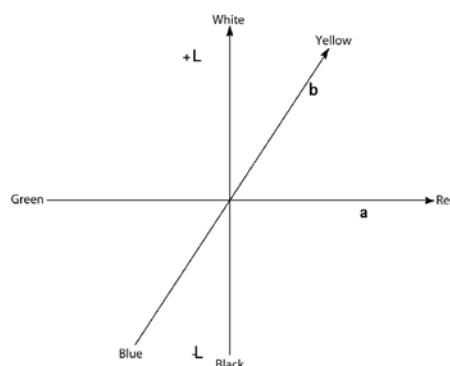
The formulation above is used for pre and post mordant experiments using 2.5% metal ions viz., aluminum sulphate, ferrous sulphate, zinc sulphate with 20% henna dyeing, pH 4 and 60° C

Premordanting: 2.5% mordant treatment (1 hour) followed by dyeing with 20 % henna

Postmordanting: 2.5% mordant treatment for 1 hour after dyeing with 20 % henna

Color Measurement

Goat nappa crust leathers (undyed) and leathers dyed with henna (with and without mordants) were subjected to the reflectance measurements. The color measurements parameters viz., *L*, *a*, *b*, *h* and *C* were recorded using Milton Roy Color Mate HDS for the crust leather samples, where *L*, represents the clarity; *a*, represents the chromatic component green –red (red and green axis); *b*, represents the chromatic component blue – yellow (yellow and blue axis); *C*, represents color saturation: $C = (a^2 + b^2)^{1/2}$; *H*, represent the tonality (angle of color): $H = \arctan(b/a)$



Evaluation of Dyeing Characteristics

Three experienced tanners evaluated the dyeing characteristics viz., color uniformity, penetration of dye and differential dyeing of henna leathers with and without mordants on a scale of 1 - 10. Higher values indicate better dyeing characteristics.

RESULT AND DISCUSSION

Dyeing is a very important process in leather making, which facilitates in improving the aesthetic value of the leather. However, most of the dyes that are currently used pose threat to the environment as they are poor biodegradable materials. It is important to seek natural alternative materials for leather dyeing. Hence, in the present work an attempt has been made to study the dyeing characteristics of the natural material, Henna of *Lawsonia inermis*.

Concentration of Henna Extract

The exhaustion of henna at different concentrations of henna extract is given in Table II. From the Table, it is seen that the uptake of henna dye by the leather slightly decreases with increasing offer of henna extract. The 20 and 25% concentration of henna extract gave a good shade compared to other concentrations at the room temperature. However, no significant difference in shade observed between leathers dyed with 20 and 25% of henna and hence 20% of henna had been considered to be sufficient for the dyeing process.

Effect of pH

The pH of the leather plays a significant role in the absorption of the dye. The exhaustion of henna extract dyed at different pHs viz., 4, 5, 6 and 7 is given in Table III. From the table it is seen that the exhaustion of the henna is high at pH 4 compared to the exhaustion at other pH's and further increase in pH resulted in decrease of the exhaustion of henna. Hence pH 4 is found to be optimum for the dyeing using henna.

Effect of Temperature

Henna dyeing was carried out at two different temperatures viz., 30° and 60°C at 20% henna offer and pH 4. The exhaustion is determined to be high, 78% for the henna dyeing carried out at 60°C compared to the one carried out at 30°C (59%). The increase in temperature has facilitated the penetration of dye and hence the exhaustion is found to increase in the case of dyeing at high temperature. Hence, temperature of 60°C appears to be effective for the dyeing of henna and the same has been taken as optimum for the dyeing process.

Fastness Characteristics of Leathers

The rub and perspiration fastness of leather dyed with henna at optimized conditions is given in Table IV. The results of rub fastness of the henna dyed samples were found to be good. In case of dry rub fastness, the loss of the depth of shade and change in hue is found between 4-5. Also in the wet rubbing the change in hue is found to be around 4-5,

TABLE II

% Exhaustion of henna using for dyeing of nappa leather at different concentrations, pH 5, 30°C

Concentration % of henna	Exhaustion %
2.5	64±2
5	65±3
10	62±2
15	60 ±2
20	57± 3
25	56±2

TABLE III

% Exhaustion of henna extract (20%) used for dyeing of nappa leathers at different pHs, 30°C

pH	Exhaustion %
4	59±2
5	55±3
6	45±2
7	42±2

which is much better than the standard limits. The perspiration fastnesses of the dyed samples are also found to be good and are within the standard limits. However, the light fastness of the leathers are found to be lower compared to the requisite standard. It is known that pre and post mordant treatment using metal ions will not only improve the light fastness, but also result in generation of different shades/colors. Hence, pre and post mordanting experiments in henna dyeing has been carried out.

Pre and Post Mordanting in Henna Dyeing

The leathers treated with different mordants (aluminum sulphate, ferrous sulphate and zinc sulphate) before or after dyeing with henna leads to change in the depth of the shade produced and variation in colors of the leather. The color deepened markedly according to the amount of natural dye used. Multiple colors were obtained depending on the mordant used (Figure. 2). The color values of the control crust, henna dyed leather and henna leathers dyed with pre and post mordanting are given in Table V.

TABLE IV
Colour, Perspiration and Light Fastness
of the leather dyed using 20%

Property	Henna dyed	Recommendation
Colour fastness		
to rubbing		
<u>Leather colour change:</u>	4/5	
Dry 150 rubs	4/5	
Wet 50 rubs		
<u>Felt- clouring staining:</u>	4	
Dry 150 rubs	4	Min 3
Wet 50 rubs		
Perspiration fastness		
1. Cellulose acetate	3	
2. Bleached cotton	3	
3. Spun nylon	2/3	
4. Spun polyester	4/5	
5. Spun acrylic	4/5	
6. Wash spun wool	2/3	Min 3
Light fastness	3	Min 4

The L value on treatment with henna is found to decrease, which is clearly indicative that the henna treated leather is darker than the undyed crust leather. There is a marked difference in the h value of henna treated leather compared to the undyed crust, which is indicative of the coloring in the case of henna treated leather. Aluminum sulphate (pre-mordant) followed by the henna treatment resulted in highest 'b' value which is indicative that these leathers are more yellow compared to other treatments. The 'a' value of henna treated leather is slightly higher compared to that of undyed crust leather, which is indicative that the shade of the henna treated leather is on red side compared to that of the undyed crust. The 'a' values had been found to be higher in the case

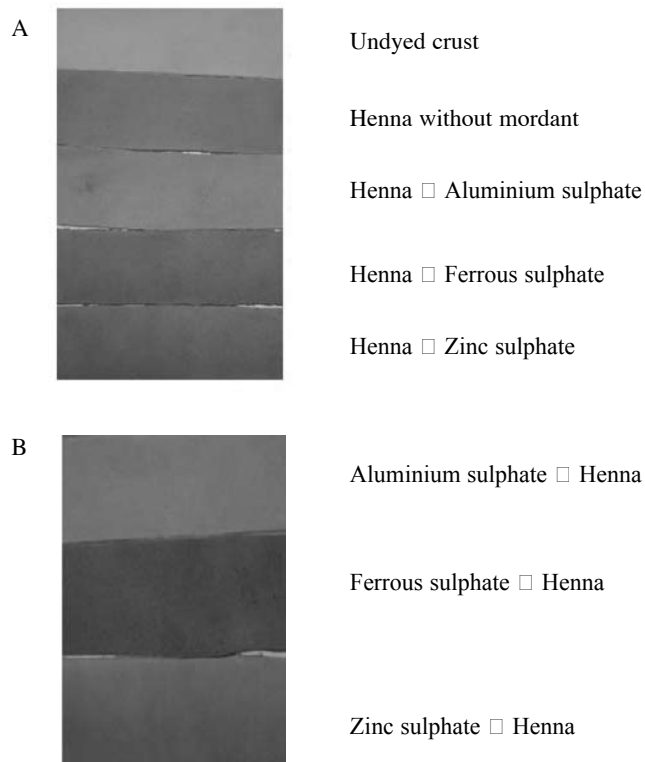


Figure 2: Colour difference of crust leather dyed with henna and different mordants: (A) dyeing of henna with postmordant (B) dyeing of henna with premordant

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of henna mordanting using ferrous sulphate, zinc sulphate and post mordanting with aluminum sulphate. The 'L' values of henna mordanted with ferrous sulphate were found to be low, which is indicative of the darkness of the shade. Henna pre-mordanted with ferrous sulphate was found to be darker than the post-mordanting. The variations in the shade, intensity of color of henna due to post and pre-mordanting using metal ions has been clearly observed from the L, a, b, c and h values given in Table V and the leathers shown in Fig. 2.

Dyeing characteristics of crust leathers dyed with henna and henna pre and post treated with mordants are given in Table VI. Uniformity of color, dye penetration has been found to be good for henna dyed crust leathers; also, there is not much difference in color observed between grain and flesh side as observed from the higher value for differential dyeing given in the table. In the case of henna dyeing pre and post-mordanted with aluminum sulphate resulted in leathers with good color uniformity and dye penetration. Mordanting with other two metal salts viz., ferrous and zinc sulphate, had good color uniformity and dye penetration except for ferrous sulphate pre-mordanting. But the both metal salts resulted in more differences in color between grain and flesh compared to mordanting with aluminum sulphate. In general post-mordanting had resulted in leathers with better dyeing characteristics compared to pre-mordanting.

TABLE V

Color Measurement of crust leather dyed with henna extract using premordants and postmordants of different metal ions

Sample		L	a	b	c	H
Crust leather without dyeing		72.363	4.322	5.192	4.396	349.481
Crust leather dyed with henna		64.363	5.169	15.763	16.927	68.626
Pre mordant	Aluminum sulphate	66.120	3.686	19.061	20.397	79.589
	Ferrous sulphate	55.349	5.570	17.178	19.012	72.963
	Zinc sulphate	60.460	5.380	17.847	19.216	73.742
Post mordant	Aluminum sulphate	65.628	5.157	16.966	17.733	73.092
	Ferrous sulphate	41.649	5.111	14.789	15.647	70.935
	Zinc sulphate	64.469	5.940	15.521	16.619	69.058

TABLE VI

Dyeing Characteristics of crust leather dyed with henna extract using premordants and postmordants of different metal ions

Sample		Color Uniformity	Dye Penetration	Differential * Dyeing
Crust leather dyed with henna		8 ± 0.5	8 ± 1	8 ± 1
Pre mordant	Aluminum sulphate	8 ± 1	8 ± 1	7 ± 0.5
	Ferrous sulphate	8 ± 0.5	6 ± 0.5	6 ± 0.5
	Zinc sulphate	8 ± 0.5	8 ± 0.5	5 ± 0.5
Post mordant	Aluminum sulphate	8 ± 1	8 ± 0.5	8 ± 1
	Ferrous sulphate	8 ± 0.5	8 ± 1	6 ± 0.5
	Zinc sulphate	8 ± 0.5	8 ± 0.5	7 ± 0.5

*- higher rating indicates lesser differences in dyeing between grain and flesh side

TABLE VII

Light fastness grades of crust leather dyed with henna extract using premordants and postmordants of different metal ions

Sample		Light fastness
Crust leather dyed with henna		3
Pre mordant	Aluminum sulphate	3/4
	Ferrous sulphate	4
	Zinc sulphate	3/4
Post mordant	Aluminum sulphate	3/4
	Ferrous sulphate	4
	Zinc sulphate	3/4

The light fastness of henna dyed leathers in the presence of pre and post mordant is given in Table VII. From the table it is clear that the light fastness of henna dyed leather improved on treatment with mordants. Pre and post mordanting using ferrous sulphate resulted in better light fastness compared to other mordanting experiments. Hence, the use of mordant not only resulted in making different shades but also improved the light fastness characteristics of henna dyed leathers.

CONCLUSIONS

The plant extract henna has been used as an alternative natural material for the dyeing of leather. An offer of 20% of henna extract (% based on the crust weight of wet blue leather) is found to be optimum for the dyeing process. The pH and temperature of 4 and 60°C respectively has been found to be the optimum conditions for henna dyeing. Rub fastness (dry and wet) and perspiration fastness grades of the henna dyed leather were found to be good to very good, while the light fastness grades of the same were found to be poor. However, the use of mordant improved the light fastness. The pre and post mordanting of henna dyed leather not only resulted in leathers with good light fastness but of leathers with different shades. Hence, leather dyeing to different shades and good coloring characteristics using a natural material, henna, appears to be a viable option for commercial exploitations.

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