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Assessment of dyeing properties and quality parameters of natural dye extracted from *Lawsonia inermis*

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ABSTRACT

In India, traditionally some plants were used for dyeing the fabrics. According to dye yield and fastness properties the plant was chosen for fabric dyeing. Most of the synthetic dyes showed strong allergy and pollutant to the environment. So, for the current need we are chosen the plant Lawsonia inermis to colour the fabrics in this work. First, the dye was obtained through the extraction methods and studied the physicochemical properties of the dye. The dyeing properties were optimized for colouring the cotton and cotton thread. Different colour fastness parameters were also noted. In this work, we found the red dye obtained through alkaline extraction has better colour strength i.e. the maximum colour intensity at 3M NaOH. The peak at 1.907 from HPLC result confirmed the lawsone compound present in the dye. During dyeing the fabrics showed brighter colour after 24 hours. In fastness studies while washing with soap and dried under sunlight for a day and 2 days later, the colour was slightly faded in all fabrics. Still, due to its nontoxic and eco-friendly features the henna dye can be an effective natural dye for clothing.

Keywords: *Lawsonia inermis*, Alkaline extraction, Physicochemistry, HPLC, Dyeing, Colour fastness.

INTRODUCTION

The art of making natural dyes is one of the oldest known to man and dates back to the dawn of civilization. India's expertise in natural dyes dates back to ancient times[1].In India, there are more than 450 plants that can yield dyes. In addition to their dye-yielding characteristics, some of these plants also possess medicinal value. The plants exhibit a wide range of colours, not all of these pigments can be used as dyes. Some do not dissolve in water, some cannot be adsorbed onto fibers, whereas others fade when washed or exposed to air or sunlight. It remains a mystery, why plants reward us with vibrant dyes. Due to lack of availability of precise technical knowledge on the extracting and dyeing technique, it has not commercially succeeded like the synthetic dyes[2].

Henna is widely used in the cosmetic industry as dyeing agent. It consists of fresh or dried leaves of the plant *Lawsonia inermis*. It has medicinal importance along with dyeing property [3].In addition, when properly applied, natural dyes are fast, resisting fading due to exposure to sunlight. Natural dyes from crushed fruits, berries and other plants, which were boiled into the fabric and which gave light and water fastness (resistance), were developed.

The textile industry produces and uses approximately 1.3 million tonnes of dyes, pigments and dye precursors, valued at around $23 billion, almost all of which is manufactured synthetically. Natural dyes are produced from plant tissues and fungal species. Natural dyes have been known for centuries, the reasons synthetic dyes have been

so popular are: They are simple to produce in large quantities, and can be manufactured at a reasonable price then it can provide a variety of colours that are demanded by today's consumers. They provide high colour-fastness (i.e. the dye is very strongly bound to the fabric and does not detach after repeated washing cycles). It is clear, however, that if natural dyes are to be considered as an alternative to the synthetic dyes used today [4].

The present work was planned to extract more colouring component with keeping the environment friendly extraction procedure excluding the extensive application of organic solvents. The solvent was chosen to view the acidic chemical nature of lawsone. This will help to make natural dye as co-partner of synthetic reactive dyes. Furthermore, the dyeing and mordanting characteristics of colouring matter on cloth and thread have also been studied.

MATERIALS AND METHODS

Sample Collection

The leaves of *Lawsonia inermis*(Henna) was collected from the Botanical Garden, VHNSN College, Virudhunagar. The collected plant was botanically authenticated by the Department of Botany, VHNSN College, Virudhunagar. The plant leaves were washed with water and shade dried for 1week then ground into the powder. The powdered sample was used for further studies.

Dye extraction

To prepare the particular natural colour on the gathered trial, four types of extraction technique had been followed. In aqueous technique, 10g of *Lawsonia inermis* leaf powder had been added with 100ml distilled water along with boiled in 100°C regarding 30 minutes. In acidic technique, 10g of *Lawsonia inermis* leaf powder had been added using 1% of HCl with 100ml distilled water along with boiled in 100°C regarding 30 minutes. In alkaline technique, 10g of *Lawsonia inermis* leaf powder had been added with 100ml aqueous remedy of sodium hydroxide in 0. 05M, 0. 10M, 0. 15M, 0. 20M, 0. 25M along with 0. 30M levels and also the henna to liquor percentage (M: L) of 1: 10. This extraction had been accomplished in 100°C regarding 30 minutes [5]. In alcoholic technique, 10g of *Lawsonia inermis* leaf powder had been added using 50% ethanol along with boilingat 50°C for 30 minutes. [6].

Measurement of colour strength

Spectrophotometric sizes have been performed applying SHIMADZU 1650-PC visible spectrophotometer fitted that has a 10mm path length quartz portable and also built with UV Probe software program to the rating with the coloration durability.

The particular produced natural color by *Lawsonia inermis* had been dissolved with acidified methanol to create the actual stock answer while using attention involving 1mg/ml and also aliquots involving only two and also 4ml of these have been transferred to sample tubes, evaporated to be able to dryness, and also dissolved with 20ml involving double distilled H_2O give you a remaining attention to be able to 0. 1 mg/ml. The particular sample pontoons have been closed together with Para film and it also had been saved. UV–Vis spectra have been noted dependant on intake maxima involving 620 and also 280nm with spectrophotometer built with UV Probe software program and a pc device. Colour intensities have been measured while absorbance worth pertaining to *Lawsonia inermis* and also indicated while molar absorptivity while not too long ago defined pertaining to natural dyes [7]. Pertaining to measures reproducibility, each and every try had been done with triplicate.

Analytical HPLC

Analytical HPLC analysis of the NaOH dissolved (1:16) *Lawsonia inermis*dye was carried out using a model SHIMADZU, LL-10ATVP ternary pumping system with integral variable volume autosampler, column oven and model 1040 series II photodiode array detector with HP workstation. The column used was a 250-4.6mm HRPB 5mm (octyl-octadecylsilane, fully endcapped14% carbon loading; HiChrom,) and the mobile phase consisted of solvent A (0.4% v/v acetic acid in acetonitrile) and solvent B (0.4% v/v aqueous acetic acid) delivered at 1 ml/min. For isocratic elution, the mobile phase was fixed at a ratio of 65%A : 35%B, while for gradient elution the starting mobile phase ratio was 50%A : 50%B which was increased linearly over 30 min to 20%A : 80%B and held for 10 minutes [8]. All samples were filtered through a 0.2mm membrane syringe filter prior to analysis.

Dyeing procedure

Cotton, polyester, cotton thread and silk thread were collected from weavers. The cotton cloth, polyester cloth, cotton thread and silk thread were used for the dyeing studies. The cloths were cutted into small pieces (Approximately 2cm sq.) and the threads (Approximately 10-15 cm length) were used for this experiment.

Scouring of cloth and thread

The clothes and threads were washed in a solution containing 0.5g sodium carbonate, boiled at 80°C for 25minutes and keeping the material to liquor ratio at 1:40. The scoured material was thoroughly washed with tap water and dried at room temperature. The scoured material was soaked in clean water for 30minutes prior to dyeing and mordanting.

Dyeing

The cloth and thread samples were dyed with extracted natural orange colour dye from *Lawsonia inermis* keeping M:L ratio as 1:20; however for cloth dyeing it was used directly while in the case of thread dyeing the pH was maintained by adding a buffer solution (sodium acetate and acetic acid) for plant extract. The dye extract was prepared by adding 10ml of natural dye extract in 20ml water (M:L - 1:2). Dyeing was done by the conventional dyeing method. After dyeing, the dyed material was washed with cold water and dried at room temperature [9].

Mordanting

The scoured cloth and thread samples were treated with different metal salt, only premordanting with metal salts was carried out before dyeing. The mordant such as potassium dichromate, copper sulphate and ferrous sulphate 2%, 4% and 6%, were dissolved in 100ml distilled water in different 250ml beaker. Then wetted samples were dipped into the mordant solution and then brought to heating temperature of the dye bath was raised up to 80°C for 30minutes. Then cooled for 15minutes and washed with water and air dried. Further the dyed samples were used for dyeing process.

Fastness properties of natural dyed cloth and thread

Fastness test is one of test to check the dyeing property of the materials. Wash fastness and light fastness tests were studied. The results were compared with different parameters studied for fabric dyeing.

Wash fastness test

The dyed clothes and threads were washed with cold water for 30minutes. After 30minutes, excess water was removed and air dried at room temperature. The fastness was observed [8].

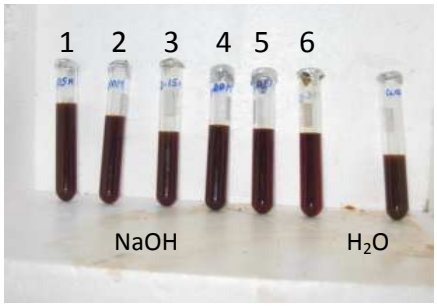
Light fastness test

The dyed clothes and threads were washed with distilled water for 30minutes. After 30minutes, excess water was removed and dried in direct sunlight for 2hours.In next experiment, the dyed samples were taken and washed in soap solution keeping liquor to material ratio 1:50 for 30minutes. After that time, excess water was removed and washed with cold water then dried in direct sunlight for 2hours.Further, the colour fastness was tested in dyed clothes and threads by exposing it to sunlight for a day and 2days. Finally, the observed results were compared to the unwashed fabrics, sunlight exposed and unexposed areas of the fabrics [10].

RESULTS AND DISCUSSION

India is being one of the countries which possess the natural wealth in the form of plantation in plenty. This has provided relatively better opportunity for the development of industries in this country. The raw materials for the production of natural dyes were plentifully available. Natural dyes do not cause any harm to human skin and no hazards. Some of the natural dyes act as a health cure [11]. The toxic chemical nature is almost absent in the manufacture of natural dyes and no pollution problem in the environment. The extraction efficiency of colourant compounds present in plant and microbes, based on the media dye (aqueous / organic solvents or acid / alkali), pH of the media and conditions of extractions such as temperature was major role in colouring.

Fig 1: Showing the extracts of *Lawsonia inermis*yield red colour



1 – 0.05M, 2 – 0.1M, 3 – 0.15M, 4 – 0.2M , 5 – 0.25M, 6 – 0.3M

The leaves of *Lawsonia inermis* was collected, air dried and powdered. The powdered materials were used for the experiments. For the dye extraction, organic solvents such as HCl, NaOH and alcohol were used. It was observed that the yield of the dye extract was better in 3M NaOH in comparison with other solvents (Fig 1).

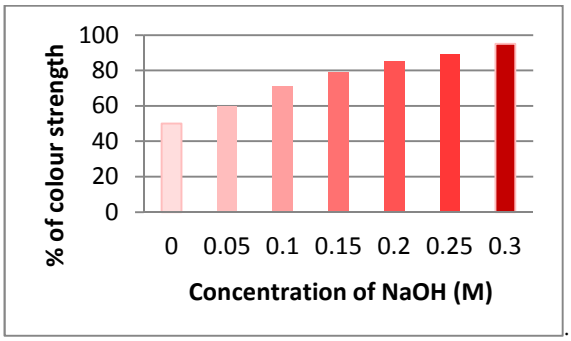
The figure 1 indicated that the colour strength of *Lawsonia inermis* while using different concentrations of sodium hydroxide. The quality and quantity of the resulting extract might vary depending upon the amount of pigments present in the plant materials taken. The alkaline extracts of *Lawsonia inermis* have better colour strength than the dye extracts obtained through distilled water and 50% alcohol. Red colour dye from *Lawsonia inermis* leaf extract was used for the further studies. The UV–Visible spectrum of the extracted natural dye was characterized by the major absorption maxima. The molar concentration of NaOH variation affects the colour intensities of the natural dye from *Lawsonia inermis* was studied.

The maximum colour intensity for the studied natural dye from *Lawsonia inermis* reached in the alkaline region (Fig 2, Table I) giving a deep red colour at 560nm. The results representing the intensity of the visible absorption maximum began to increase of NaOH concentration and continued with the increase of pH.The colour strength of the natural dye extracted from *Lawsonia inermis* showed the colour strength as concentration dependence increases based on NaOH concentration.

Table I: Absorbance maxima of natural dye extracted from *Lawsonia inermis* in water extract

S.No.	Wavelength (nm)	Absorbance
1.	620	0.5
2.	600	0.8
3.	580	1.0
4.	560	2.3
5.	540	1.5
6.	520	1.6
7.	500	1.8
8.	480	1.0
9.	460	1.1
10.	420	1.3
11.	400	1.5

Fig 2: Colour strength of the natural dye extracted from *Lawsonia inermis* using NaOH

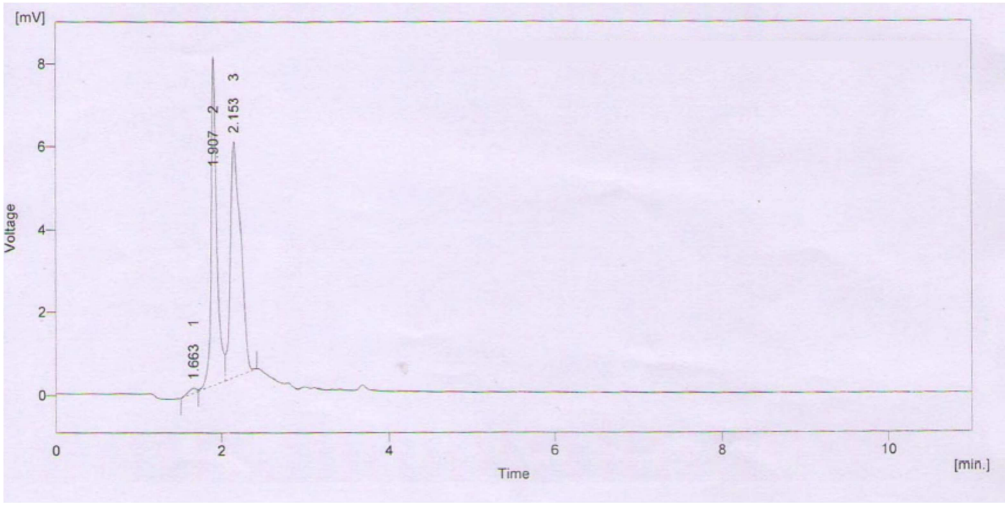


The HPLC study for natural dye henna results showed three different peaks at different time intervals. First peak observed of retention time at 1.663 and second peak showed at 1.907 and third at 2.153. Among three peaks, the second peak observed as major peak (Fig 3).

The UV spectrophotometry and HPLC studies confirmed the physical and chemical properties of the natural dye. The red colour *Lawsonia inermis* contain lawsone compound was confirmed by HPLC analysis.

The effect of dyeing temperature on colour strength was demonstrated. It can be observed as the colour strength increases with increase of dyeing temperature and reaches a maximum at 70°C then it decreases. This increase in dye uptake can be attributed to better dye exhaustion at higher temperatures. However, temperature higher than 70°C results in decrease of colour strength, which may be attributed to decrease in dye molecules stability at higher temperatures.

Fig 3: HPLC result for *Lawsonia inermis*



The longer dyeing time, gives the higher colour strength until dye exhaustion attains equilibrium and there is a decrease in the colour strength on further increase in time over 24hours.After the red colour dye was applied on the clothes and threads, it showed brighter colour in cloths and thread (Fig 4). In 0 minute, it showed palered for natural dye but at the time of 24hours it gives brighter colour.

Fig 4: Direct dyeing of *Lawsonia inermis* dye at different time intervals

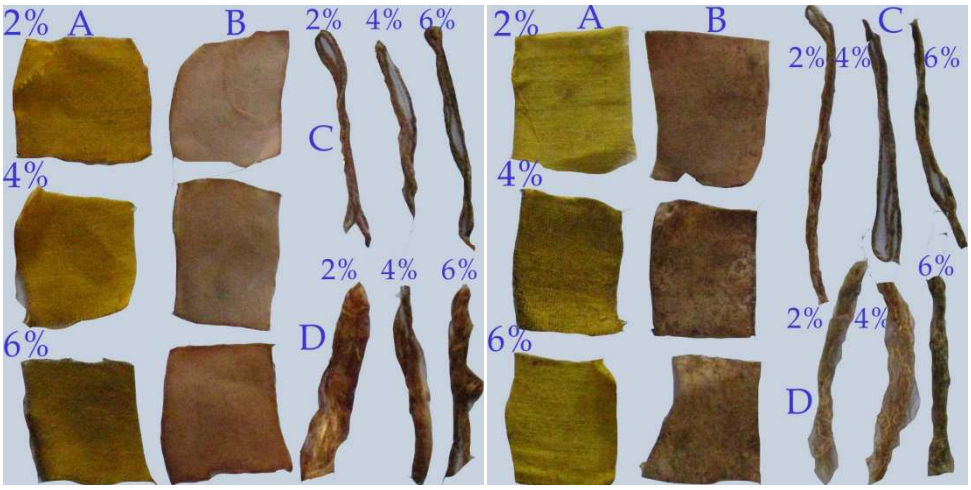
A – 0minute, B - 1hour, C - 3hours, D - 6hours, E - 12hours, F - 24hours

The decline in colour strength is attributed to the shift in equilibrium of colouring component from fabric into dye bath during longer dyeing time, the effect of salt on the dye exhaustion. It can be seen that increase of salt concentration increases the colour strength, which can be attributed to the neutralization of negative charge due to zeta potential of cotton by sodium ions in the dye bath. The effect of liquor to fabric ratio (L:R) on colour strength. It is clear that colour strength increases with increase L:R. Lower relative colour strength value at low L:R may be explained by the overcrowding of dye molecules at lower L:R resulting in reduced dye exhaustion on the fabric.

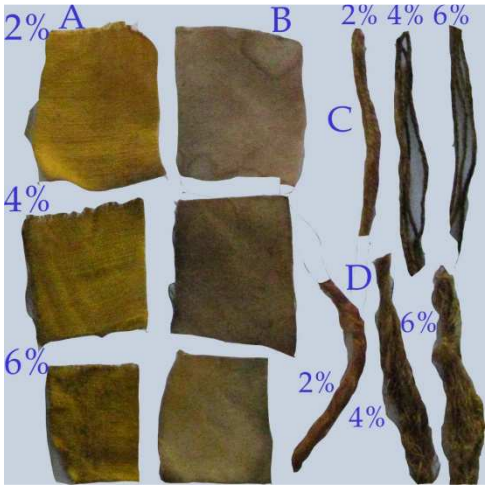
The extracted natural dye was applied directly to fabrics and also to mordanted fabrics (Fig 5). But best result was observed in directly applied cloth compared with chemical mordanted dyed cloth.

Fig 5: Dyeing properties of natural dye with different mordants for clothes and threads

Potassium dichromate **Copper sulphate**



Ferrous sulphate

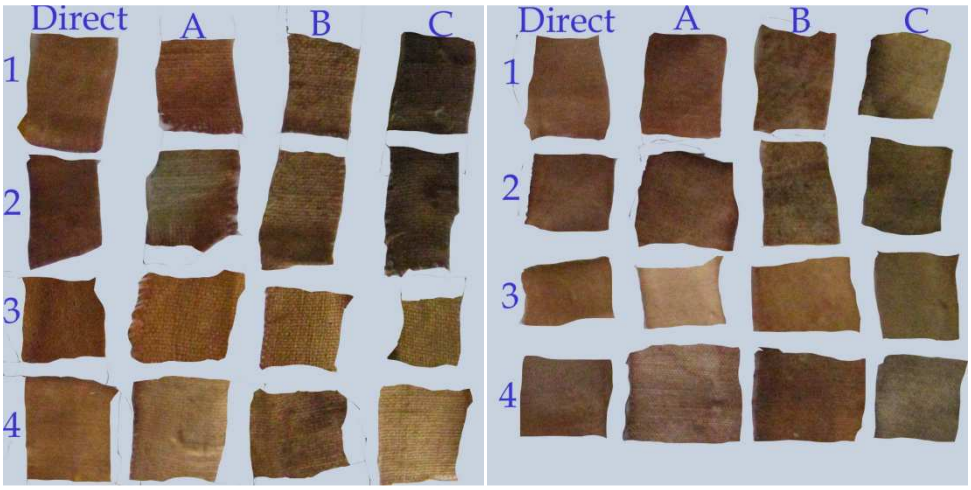


A – Cotton B – Polyester cloth C – Cotton thread D - Silk thread

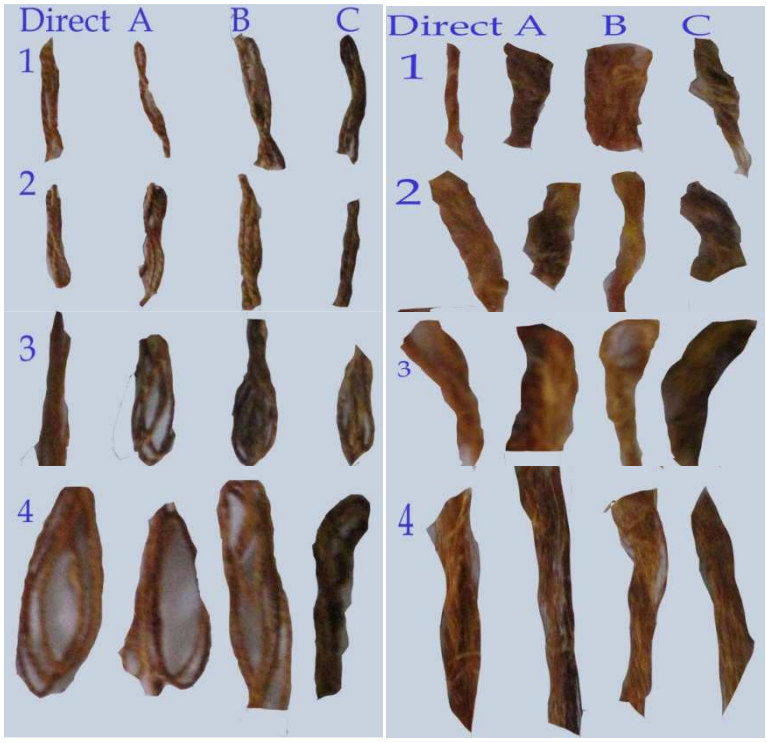
The wash fastness of fabric dyed with and without mordant was analyzed for mordanted fabrics. These results were assessed in the usual way in terms of visual values for the staining of adjacent cotton material and alternation in shade. It is indicated that sample with natural dye extract and iron mordant gave rating good to very good wash fastness in comparison with very good to excellent rating for shade developed with only natural dyes. The very good to excellent wash fastness of sample dyed with natural dyes was due to the strong covalent bonds between the reactive dye molecules and the fabric (Fig 6).

Fig 6: Fastness properties of natural dyed fabrics

Cotton cloth **Polyester cloth**



Cotton thread **Silk thread**



A - Potassium dichromate, B - Copper sulphate, C - Ferrous sulphate

1 – Before washing
2 – After washing and dried under shade
3 – After washing with soap and dried under sunlight for 1 day
4 - After washing with soap and dried under sunlight for 2 days

Light fastness of fabrics dyed with *Lawsonia inermis* alkaline extract with and without mordant results in good to better according to rating. The higher light fastness properties of reactive dyes can be attributed to the strong intramolecular H-bonding, which exists in the form of six member rings. This enhances the stability of the compound by a reduction in electron density at the chromophore. As a result sensitivity of dye towards photochemical oxidation becomes reduced. Here it is noted that sample dyed without mordant has better fastness to light in comparison with sample dyed with iron mordant. It can be attributed to the catalytic effect of mordant for colouring component. The fastness was observed when using soap washed, sun directed clothes and threads.

In areas, where synthetic dyes, mordants (fixatives) and other additives are imported and are therefore relatively expensive [12]. Increased use of synthetic dyes and chemicals in the textile, foodstuff, cosmetic and pharmaceutical industries have resulted in the generation of large quantities of effluent that contain high levels of dyes and toxic materials. The presence of these pollutants in the effluents may pose environmental disposal problems due to their non-degradable and persistent nature. Moreover, the presumed or actual noxious effects of some synthetic dyes on human health, such as skin cancer and allergic reactions, have raised a great deal of concern [13, 14 & 15].Therefore, the environmentally hazardous synthetic dyes must be replaced with cost-effective natural and environment-friendly dyes or pigments.

The black dye produced from herbal resources may be used in wide variety of context including hair colour products. At this juncture, there is enormous need for a method to increase the yield of such dyes from herbal products [16 & 17].

The identified problems associated with the use of synthetic dyes now prompted the search for low-cost dyes, especially of biological origin, that will be effective, easy to use, biodegradable and safe to both the human and environment health [18,19 & 20]. Now-a-days, the use and production of natural dyes is becoming more popular owing to the growing awareness of environmental problems coupled with the toxicity associated with synthetic dyes. As a result, renewable resources are now being reinvestigated as alternative raw material for natural dyes [21 & 22]. Some of them have also been reported to possess anti-UV and anti-microbial properties [23].

CONCLUSION

The red colour dye extracted from *Lawsonia inermis* used with different mordants as natural dyeing for clothes and threads which showed good fastness properties and high dye uptake. It can provide bright hues also. It is noteworthy that it can serve as a source of raw material for fabric dyeing in the future. Natural dyes are not only having dyeing property but also having the wide range of medicinal properties. Now-a-days, there is an increasing awareness among people towards natural dyes and dye yielding plants. Due to their non-toxic properties, less side effects, more medicinal values, natural dyes are suitable for dyeing.

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