OPENING MINDS:
RESEARCH FOR SUSTAINABLE
DEVELOPMENT

Dyeing 100% Cotton Plain Fabrics with Natural Dye Extracted from *Thespesia populnea* (Gan Suriya)

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1 INTRODUCTION

Natural dyes are known for their use in the colouring of food substrate, leather as well as natural protein fibres like wool, silk and cotton all of which have been major areas of application since pre-historic times (Samanta *et al.,* 2009). Natural dyes can be extracted from many natural sources. Among them, plant dyes can be extracted from the leaves, seeds, flowers, fruits, barks, roots and wood (Allen, 1971). *Thespesia populnea* is an example of a tree, which contains tannin in its bark. The common name of this tree is Gansuriya. *Thespesia populnea* is a small to medium sized evergreen perennial tree grown in the coastal, tropical and sub-tropical areas. It is a flowering plant belonging to the Malvaceae family.

In Sri Lanka, this plant is not used for any special purposes. However, the bark and leaves of *Thespesia populnea* are ideal for skin diseases.

The main objective of the study is to extract a natural plant dye from this commonly available plant source to dye cotton fabrics. To meet this objective, the study provides the information on suitable extraction methods, dyeing procedures to dye cotton fabrics and the fastness properties of the extracted dye for light and washing.

2 METHODOLOGY

The experimental procedure includes the dye extraction from the stem bark of Gansuriya, mordanting, dyeing and assessing colour fastness of the dyed 100% cotton samples.

2.1 Materials used in dye extraction, dyeing and mordanting

Several materials and equipment were used for the extraction process as given below.
Dried Gansuriya bark chips, water, a scale to measure, pH papers, a thermometer and a burner, Sodium Hydroxide (NaOH), Sodium carbonate (Na_2CO_3), lemon juice, vinegar, de-sized and bleached 100% plain cotton fabric of 6cm x 7.5cm, Potassium Aluminium Sulphate (KAI $(SO_4)_2 \cdot 12H_2O$), Copper Sulphate ($CuSO_4$), Ferrous Sulphate ($FeSO_4$) and Sepalika flowers *Nectanthes arbor-tristis*.

2.2 Dye extraction by changing pH

Aqueous extraction was carried out using 1l of clean well water. The cut bark chips were dried under shade and 100g of dried bark chips was added to the water. The water bath was heated to 1000C over 75 minutes. To observe the extraction efficiency and colour yield by the changing pH at the extraction, several acids and alkalis were added to the water

bath. Vinegar and lemon juice were added separately to adjust the pH of the extraction liquor in to the acidic state to keep the pH level as 5 and 3 respectively. NaOH and Na_2CO_3 were added separately to adjust the pH into the alkaline state to keep the pH as 13-14 and 8-9 respectively.

2.3 Dye extraction by changing temperature and duration

To determine the most suitable temperature and duration for dye extraction, experiments were carried out using 30 minutes, 45 minutes and 60 minutes and two dyeing temperatures of 500ºC and 1000ºC while the other conditions were kept constant. The extracted liquor was filtered.

2.3 Pre mordanting process

The fabrics to be dyed are subjected to mordants before dying. Here, 3 metal salts; alum, $CuSO_4$, $FeSO_4$ and as a natural mordant Sepalika flowers were used.

2.4 Dyeing process

Dyeing was carried out according to the dyeing curve in Figure 1 to dye non mordanted and pre-mordanted samples using dye liquors extracted in neutral, acidic (pH 4-5) and alkaline (pH 12-13) conditions.

2.5 Experiments to evaluate the optimum dyeing duration

To determine the optimum dyeing duration, experiments were done using four different dyeing durations while the other conditions were kept constant. Furthermore, below dyeing curve has been used as the base and improved to evaluate the optimum dyeing duration.

500 mL of neutrally extracted dye liquor to dye, 1.10 g non mordanted fabric sample and 10 g of common salt were used. The temperature of the dye liquor was raised to 500ºC and the fabric samples were immersed in the dye liquor. They were kept at that temperature for 15 minutes. Within about 5 minutes, the temperature was raised to 950ºC. It was kept for another 10 minutes at that temperature to dye over 30 minutes. To dye over 40 minutes, 60 minutes and 75 minutes, the samples were kept for 25 minutes, 40 minutes and 55 minutes at that temperature respectively. Then the temperature was reduced to room temperature over 10 minutes. Dyed samples were left steeped in the dye baths for a further 6 hours at room temperature. Finally, the samples were washed with cold water and dried in the shade.

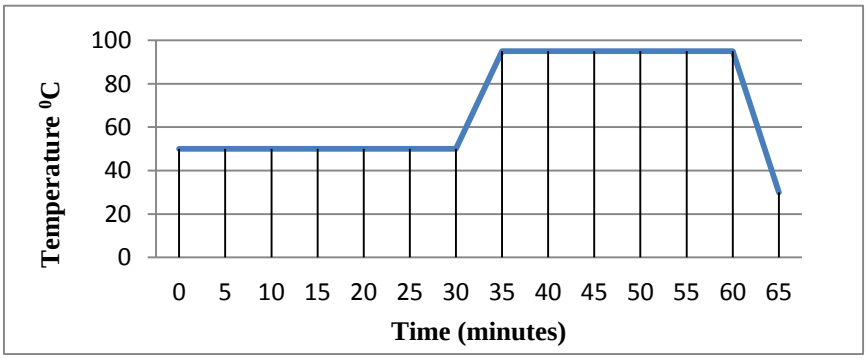

Figure 1. Dyeing curve

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2.6 Experiments to evaluate the optimum dye steeping duration

To determine the optimum dye steeping duration, experiments were done using three different steeping durations while other conditions were kept constant. 500 mL of neutrally extracted dye liquor was used to dye 1.10 g of non-mordanted fabric samples. The temperature was raised to 500°C and the fabric sample was immersed in the dye liquor and was kept for a further 30 minutes. Then within about 5 minutes, the temperature was raised to 950°C. The dye bath was kept for another 25 minutes at that temperature. Then the temperature was allowed to reduce to room temperature. The dyed sample was left steeped in the dye baths for an additional 30 minutes at room temperature. According to the same method, the dyeing was carried out for other samples but the steeping duration was changed to 6 hours and overnight respectively. Finally, all the samples were washed with cold water and dried in the shade. The differences between the colours were visually assessed under the colour assessment cabinet using the grey scale (ISO 105 AO3: 1993).

2.7 Experiments to evaluate the dye affinity at different pH

The dye affinity of the dye under different pH values from acidic to alkali was evaluated. Neutrally extracted dyes of both dyes were taken as the dye liquors. De-sized and bleached cotton and mercerized cotton fabrics were dyed. The temperature of the dye liquors was raised to 500C and the fabric samples were immersed in the dye liquors. Then they were kept at that temperature for 30 minutes. Within about 5 minutes, the temperature was raised to 950°C. To improve the alkalinity of the dye liquors, NaOH was added during dyeing and pH was adjusted to 13. To improve the acidity, lemon juice was added during dyeing and the pH was adjusted to 4. It was kept for another 25 minutes at that temperature. Then the temperature was allowed to reduce to room temperature. The dyed samples were left steeped in the dye baths overnight at room temperature. Finally, all the samples were washed with cold water and dried under shady conditions. The differences between the colours were visually assessed under the colour assessment cabinet using the grey scale (ISO 105 AO3: 1993).

2.8 Colour fastness evaluations of the dyes

Colour fastness tests were done for dyed samples to assess their fastness properties to light and washing. Non-mordanted and mordanted samples dyed at neutral, alkaline and acidic conditions were subjected to these fastness tests.

(a) Wash fastness test

Wash fastness was tested using the beaker dyeing machine. The dyed sample fabric size was 10cm x 4cm and the weight of a single sample was 1 g. First, non-dyed cotton fabric pieces of 5cm x 4cm were stitched to each dyed samples. 50 mL of water was added to each beaker in the beaker dyeing machine. 5 g/L common liquid detergent was added to each beaker. The temperature of the beaker dyeing machine was adjusted to 500°C. The dyed samples were attached to the hooks of the beaker lid and washed for 45 minutes. After washing, all the samples were dried in an oven at a temperature below 600C. Changes in colour were visually assessed under the colour assessment cabinet using the grey scale (ISO 105 AO2: 1993).

(b) Light fastness test

Light fastness was tested using the light fastness tester. The sample fabric size was 1cm x 5 cm. All the samples were exposed to the UV light for a time period of 53 hours until the standard sample was getting in to the 3rd class of the grey scale.

Finally, the changes in colour were visually assessed under the colour assessment cabinet using the grey scale (ISO 105 AO2: 1993).

3 RESULTS AND DISCUSSIONS

It was observed that different shades can be obtained when dye extraction is done in alkali condition and tints can be obtained under acidic conditions. The highest depth of shade was observed in the samples dyed with dye liquors extracted with NaOH (pH 13-14). The depth of shade improved with the time and temperature; for example, a low depth of shade with 500^{o}C and high depth of shade with 1000^{o}C over the dyeing duration of 60 minutes. However, at 1000^{o}C dyeing the temperature was kept at about 900^{o}C and slowly raised to 1000^{o}C when the duration was reaching 60 minutes to prevent decreasing liquor volume due to vaporization. Different colour depths and shade differences can be obtained by using different mordants and different dyeing conditions. The sample mordanted with $FeSO_4$ and dyed in neutrally extracted dye liquor and the sample mordanted with $FeSO_4$ and dyed in acidic extracted dye liquor produced very deep shades. Comparatively light hues were observed in the samples mordanted with alum and *Sepalika* flowers for all three different dye liquors. Non mordanted samples have exhibited similar hues compared to the mordanted samples. Table 01 shows the results of the optimum dyeing duration and the optimum dye steeping duration. Table 02 shows the effect of pH on dye affinity of different fabrics and table 03 shows the changes in colour after the light fastness and wash fastness tests.

A small influence of dyeing time can be seen when the dyeing is done for 75 minutes. Natural dye extracted from annatto seeds also proved to produce a good colour depth on cotton after around 45 minutes of dyeing time (Prabhavathi *et al.,* 2014). Staining was best when the sample was steeped overnight. Good staining was observed when dyed using de-sized and bleached cotton, and mercerized cotton. The results of wash fastness test show that the least degree of change in colour in the samples was visible in the samples that were dyed in neutrally and alkaline extracted dye liquors mordanted with alum, $CuSO_4$ and $FeSO_4$. *Sepalika* mordanted samples also show moderate values for a change in colour. The results of the light fastness test show moderate values in samples dyed with alkaline extracted dye liquor. Samples mordanted with alum and dyed in neutrally and acidic extracted dye liquors have shown changes in colour.

Table 01: Results of the optimum dyeing time and optimum dye steeping time

Steeping time	Staining value	Dyeing duration	Staining values
30 minutes	3	30 minutes	2
6 hours	2-3	45 minutes	2
Overnight	2	60 minutes	2
		75 minutes	1-2

Table 02: Effect of pH on dye affinity of different fabrics

Fabric type	Staining value (pH 13-14)	Staining value (pH 4-5)
De-sized and bleached cotton	2-3	2
Mercerized cotton	2	2

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Table 03: Change in colour values due to light and washing of dyed samples

Nature of the dye liquor	Non-mordant sample	alum	$FeSO_4$	$CuSO_4$	Sepalika
Neutrally extracted liquor	Light 3 Wash 4-5	Light 2-3 Wash 4-5	Light 3 Wash 4-5	Light 3 Wash 4-5	Light 3 Wash 3-4
Alkaline extracted liquor	Light 3-4 Wash 3	Light 3-4 Wash 4-5	Light 3-4 Wash 4-5	Light 3-4 Wash 4-5	Light 3-4 Wash 3-4
Acidic extracted liquor	Light 3 Wash 3	Light 2-3 Wash 3	Light 3 Wash 3-4	Light 3 Wash 3-4	Light 3 Wash 3-4

4 CONCLUSIONS AND RECOMMENDATIONS

The present study has shown that it is possible to use the bark of *Thespesia populnea* (Gansuriya) to extract natural dyes using aqueous extraction method for colouring 100% cotton fabrics. The depth of shade in extraction can be improved by the addition of an alkali during the extraction of the dye. The results have shown low colour yield for the dye when the extraction was done at the temperatures about 500°C. The colour yield was apparently improved when the extraction was done at temperatures close to 1000°C over 30 minutes. In the case of dyeing, the duration of dyeing did not heavily affect staining. However, the duration of steeping affected staining. According to the results, deep shades can be obtained by steeping the samples for 6 hours or more in the dye bath. The use of mordants has changed the hue of the dyed samples. $FeSO_4$ and $CuSO_4$ mordants have contributed to improve the original hue while alum and *Sepalika* flowers (natural mordant) contributed to lighten the original hue. The increased pH has contributed to improving the dye affinity of cotton. Further, the pH difference, whether the dyeing liquor is alkaline or acidic, allows for the colour changes in the dyed samples. The use of mordant has improved the wash fastness of the dye as the changes in the colour was least apparent in the wash fastness test, but the light fastness properties were not very satisfactory as they gave moderate results. Samples dyed in alkaline extracted dye liquor have shown somewhat good fastness to light. Non-mordanted samples have shown moderate wash and light fastness.

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