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EXTRACTION OF NATURAL DYES FROM THE STEM BARKS OF GAN SURIYA (*THESPIESIA POPULNEA*) AND MAILA (*BAUHINIA RACEMOSA*)

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ABSTRACT

The study was carried out to use plant sources to extract natural dyes for textiles. It is focused on the stem barks taken from two commonly available trees in Sri Lanka as sources. The selected trees are Gan Suriya (*Thespesia populnea*) and Maila (*Bauhinia racemosa*). Gan Suriya is an evergreen perennial tree belonging to Malvacea family which is spread in many areas. Maila is a deciduous tree grown in dry areas which belongs to the Fabacea family. In the present study, dyes were extracted from the dried cut bark chips using aqueous extraction method. To analyse the best colour yield, extraction was done at different pH conditions, different temperatures and over different time periods. It was found that the best colour yield can be achieved by alkaline extraction at temperatures close to 100°C and time periods over 30 minutes. The extracted dye liquors were used to dye de-sized and bleached cotton fabrics. Dyeing was carried out by pre-mordanting method, using commercially available three metal salt mordants, namely copper sulphate, ferrous sulphate, alum and one natural mordant, Sepalika flowers. Different shades could be obtained by using different mordants. Experiments were carried out to find out the optimum dyeing time and optimum dye steeping time. To evaluate the dye affinity at different pH values, the pH of the dye liquor was adjusted to alkaline and acidic states during dyeing. Results were assessed using a colour assessment cabinet and grey scale for assessing staining. The

staining differences were least between different dyeing time durations. Deep shades were obtained by steeping the samples in the dye liquor over 6 hours and increasing the alkalinity of the dye liquor during dyeing. Fastness to light and washing of mordanted and non-mordanted dyed samples were carried out. Washing was done using the beaker dyeing machine over 45 minutes at 50°C. Colour fading of dyed samples due to both light and washing was assessed by using the grey scales for assessing change in colour. Results revealed that the wash fastness of both the dyes was improved by the mordants and the light fastness of both the dyes was moderate even after mordanting. Finally the suitability of fabrics dyed with Gan Suriya against skin infections, such as eczema was tested and confirmed. The dye extracts from Gan Suriya and Maila have been confirmed as suitable for dyeing cellulose based fabrics.

Keywords: *Thespesia populnea*, *Bauhinia racemosa*, cotton dyeing, mordanting, colour fastness

INTRODUCTION

Natural dyes can be extracted from many natural sources. Among them, the plant dyes can be extracted from the leaves, seeds, flowers, fruits, barks, roots and woods. Some examples for previous research work done about natural dyes can be mentioned such as Use of Neem bark as wool colourant (Mathur et al., 2003), Extraction of natural dyes from African

marigold flower for textiles (Jothi, 2008), Colour intensity, fastness and antimicrobial characteristics of silk fabric dyed with Mahua bark (Sahoo et al., 2012) etc.

In this project, the stem barks of two different trees were chosen to extract dyes using different methods employed in the above projects. *Thespesia populnea* is an example for a tree which contains tannin in its bark. It is well known in Sri Lanka as Gan Suriya. *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 requirements. However the bark and leaves of *Thespesia populnea* can be used for skin diseases. Fruits, flowers and young leaves are edible. Dark red, naturally oily timber is used to carve small wooden food bowls and food utensils in Hawaii. Also they are grown in India to provide shade for tea and coffee plantations. This plant is available in many areas of the country grown in wild and can be easily used to produce dyes (Friday & Okano, 2006).

The other plant of which the stem bark can be used to extract a dye is *Bauhinia racemosa*. It belongs to the Fabaceae family. The local common name of this tree is "Maila". *Bauhinia racemosa* tree can be seen in dry areas of the country in most of times. It is a huge tree which is native to tropical Asia. These trees are distributed in dry and deciduous forests most in India and Sri Lanka. The common use of this tree is that the inner bark is used to make rough ropes in rural areas of Sri Lanka. Instead, the wood is used for fuel, leaves are taken for certain medicinal uses, used to make fodder for goat, sheep and cattle, used to make fences and used as timber in India. The bark of this tree is brownish grey and has tannin (Kavitha et al., n. d.).

Objective of the study

- To introduce two natural plant dyes extracted from commonly available two plant sources to dye cotton fabrics.
- To provide information on suitable extraction methods, dyeing methods to dye cotton fabrics with the extracted dyes.
- To evaluate the fastness properties of the extracted dyes for light and washing.
- To investigate the herbal value of the extracted dye which can be important and beneficial for the end product.

METHODOLOGY

1.1 Experimental procedure

The first step of the study was the extraction of dyes from the stem barks. The materials and equipment used for the extraction process are dried bark chips in both plants are, water, a scale to measure, pH papers, thermometer and a burner, Sodium Hydroxide (NaOH), Sodium carbonate (Na₂CO₃), Lemon Juice, Vinegar, desized and bleached cotton fabric of 6cm x 7.5cm, Potassium aluminum sulphate (KAl(SO₄)₂·12H₂O), Copper sulphate (CuSO₄), Ferrous sulphate (FeSO₄) and Sepalika flowers.

The experimental procedure includes dye extraction from the stem barks, mordanting, dyeing and colour fastness evaluations of the dyed samples.

1.1.1 Extraction by changing pH

Aqueous extraction was carried out for both of plants to extract dyes using 1l of clean well water, without adding any chemical. The cut bark chips were allowed to dry under shade. 100g of dried bark chips were weighed using a scale and added to the water. The water bath was heated to 100°C over 75 minutes. To observe the extraction efficiency and colour yield by changing pH at the extraction, several acids and alkalis were

added to the water bath. There, vinegar and lemon juice were added separately to adjust the pH of the extraction liquor in to the acidic state. Sodium hydroxide and sodium carbonate were added separately to adjust the pH in to the alkaline state. As same as the aqueous extraction process mentioned above, 100g of dried bark chips were added to the water vessel containing 1l of clean well water. During the whole extraction process, pH was maintained by adding acids and alkalis. pH was kept to 3 by adding lemon juice. pH was kept to 5 by adding vinegar. pH was kept to 8-9 by adding sodium carbonate and pH was kept to 13-14 by adding sodium hydroxide. When the extraction was completed, extracted liquors were filtered.

1.1.2 Dye extraction by changing temperature and time

To determine the most suitable temperature and time for dye extraction, experiments were carried out with both plants using three dyeing time periods of 30 minutes, 45 minutes and 60 minutes and two dyeing temperatures of 500C and 1000C. 1l of water and 100g of dried bark chips from each plant were subjected to simmer under the previously mentioned temperatures and durations. After extracting dyes from both plants, extracted liquor was filtered.

1.1.3 Pre mordanting process

Pre mordanting was done before dyeing the samples. The purpose of pre mordanting is to support the dye materials to make bonds with the dyeing substrate and allow a good dye affinity. In my experiments, three metal salts and a one natural mordant were used. The metal salts were alum (potassium aluminium sulphate), copper sulphate and ferrous sulphate. The natural mordant was Sepalika (*Nectanthes arbor-tristis*) flowers. The method of mordanting of fabrics is different from one mordant to the other. The conditions required for different mordants are different, which are explained below:

- Mordanting with copper sulphate (CuSO₄)

A beaker containing 1l of clean well water was heated to 90 0C with the addition of 3.2g of copper sulphate, 16ml vinegar and 40g of desized and bleached cotton fabric. It was heated over an hour for mordanting. The fabric was kept steeped overnight in the solution then washed with cold water and dried under shade.

- Mordanting with ferrous sulphate (FeSO₄)

A beaker containing 1l of clean well water was heated to 75 0C with the addition of 0.8g of ferrous sulphate and 40g of desized and bleached cotton fabric. It was heated over 30 minutes for mordanting. The fabric was kept steeped overnight in the solution then washed with cold water and dried under shade.

- Mordanting with alum [KAl(SO₄)₂·12H₂O]

A beaker containing 1l of clean well water was heated to 90 0C with the addition of 6g of alum and 40g of desized and bleached cotton fabric. It was heated over 45 minutes for mordanting. The fabric was kept steeped during 24 hours in the solution then washed with cold water and dried under shade.

- Mordanting with Sepalika (*Nectanthes arbor-tristis*) flowers

A beaker containing 1l of clean well water was heated to 95 0C with the addition of 20g of Sepalika flowers and 40g of desized and bleached cotton fabric. It was heated over 30 minutes for mordanting. The fabric was kept steeped overnight in the solution then washed with cold water and dried under shade.

1.1.4 Dyeing process

Dyeing was carried out with both dyes following the same method. To compare the colours of the dye liquors extracted at different pH values, 1.10g of desized and

bleached cotton fabric samples were dyed in 500ml of dye liquors. The dye liquors were first heated to about 50°C and the fabric samples were entered to the dye liquors. They were kept at that temperature for 30 minutes. Then within about 5 minutes, the temperature was raised to 95°C. It was kept for another 25 minutes at that temperature. Then the temperature was allowed to reduce to the room temperature. Dyed samples were left steeped in the dye baths for 6 hours at the room temperature. Finally the samples were washed with cold water and dried under shade. The dyeing curve is as follows.

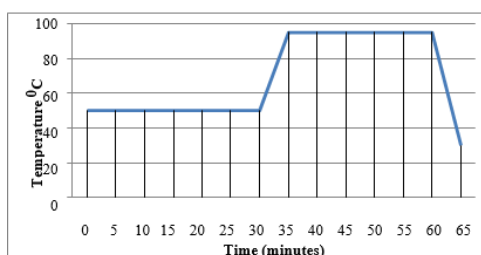


Figure 1.1 Dyeing curve

The same dyeing method was followed to compare the colours of the pre mordanted samples dyed in both dye liquors extracted in neutral, acidic (pH 4-5) and alkaline (pH 12-13) conditions. After dyeing the colour of the mordanted and none mordanted dyed samples was compared.

1.1.5 Experiments to evaluate the optimum dyeing time

To determine the optimum dyeing time, experiments were done using three different dyeing times while the other conditions were kept constant. 500ml of neutrally extracted dye liquor from both plants to dye 1.10g none mordanted fabric sample and 10g of common salt were used.

- Dyeing over 30 minutes

The temperatures of both dye liquors were raised to about 50°C and the fabric samples were entered to the dye liquors. They were kept at that temperature for 15

minutes. Then within about 5 minutes, the temperature was raised to about 95°C. It was kept for another 10 minutes at that temperature. Then the temperature was allowed to reduce to the room temperature. Dyed samples were left steeped in the dye baths for further 6 hours at the room temperature. Finally the samples were washed with cold water and dried under shade. The figure 1.2 shows the dyeing curve of this step.

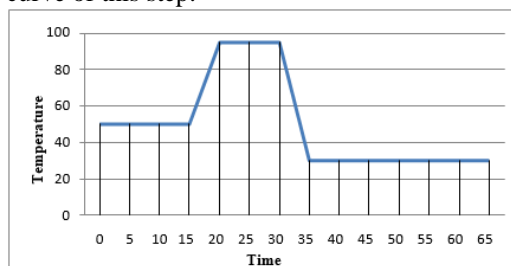


Figure 1.2 Dyeing over 30 minutes

- Dyeing over 45 minutes

The temperatures of both dye liquors were raised to about 50°C and the fabric samples were entered to the dye liquors. They were kept at that temperature for 15 minutes. Then within about 5 minutes, the temperature was raised to about 95°C. It was kept for another 25 minutes at that temperature. Then the temperature was allowed to reduce to the room temperature. Dyed samples were left steeped in the dye baths for further 6 hours at the room temperature. Finally the samples were washed with cold water and dried under shade. Figure 1.3 shows the dyeing curve of this step.

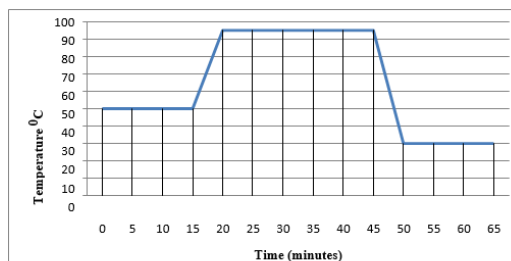


Figure 1.3 Dyeing over 45 minutes

- Dyeing over 60 minutes

The temperatures of both dye liquors were raised to about 50°C and the fabric samples were entered to the dye liquors. They were kept at that temperature for 15 minutes. Then within about 5 minutes, the temperature was raised to about 95°C. It was kept for another 40 minutes at that temperature. Then the temperature was allowed to reduce to the room temperature. Dyed samples were left steeped in the dye baths for further 6 hours at the room temperature. Finally the samples were washed with cold water and dried under shade. Figure 1.4 shows the dyeing curve of this step.

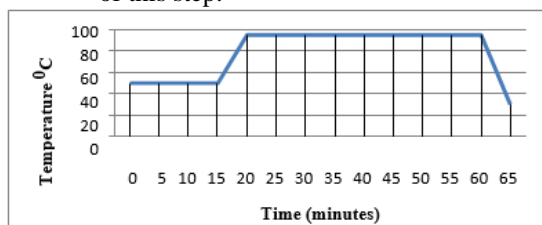


Figure 1.4 Dyeing over 60 minutes

- Dyeing over 75 minutes

The temperatures of both dye liquors were raised to about 50°C and the fabric samples were entered to the dye liquors. They were kept at that temperature for 15 minutes. Then within about 5 minutes, the temperature was raised to about 95°C. It was kept for another 55 minutes at that temperature. Then the temperature was allowed to reduce to the room temperature. Dyed samples were left steeped in the dye baths for further 6 hours at the room temperature. Figure 1.5 shows the dyeing curve of this step.

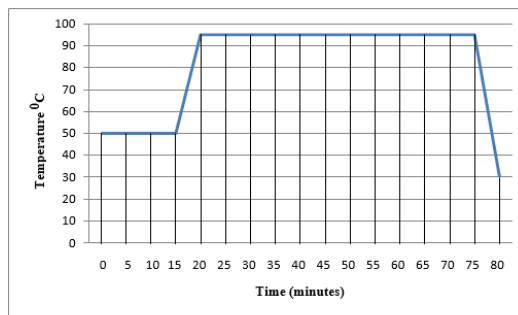


Figure 1.5 Dyeing over 75 minutes

Finally all the samples were washed with cold water and dried under shade. The differences in the hue of the dyed samples were visually evaluated under the colour assessment cabinet using the grey scale for assessing staining (ISO 105 A03: 1993).

1.1.6 Experiments to evaluate the optimum dye steeping time

To determine the optimum dye steeping time, experiments were done using three different steeping times while the other conditions kept constant. 500ml of neutrally extracted dye liquor from both plants were used to dye 1.10g of none mordanted fabric samples.

- Steeping during 30 minutes

The temperatures of both dye liquors were raised to about 50°C and then the fabric samples were entered to the dye liquors. They were kept at that temperature for 30 minutes. Then within about 5 minutes, the temperature was raised to about 95°C. It was kept for another 25 minutes at that temperature. Then the temperature was allowed to reduce to the room temperature. Dyed samples were left steeped in the dye baths for 30 minutes more at the room temperature. Finally the samples were washed with cold water and dried under shade.

- Steeping during 6 hours

The temperatures of both dye liquors were raised to about 50°C and then the fabric samples were entered to the dye liquors. They were kept at that temperature

for 30 minutes. Then within about 5 minutes, the temperature was raised to about 95°C. It was kept for another 25 minutes at that temperature. Then the temperature was allowed to reduce to the room temperature. Dyed samples were left steeped in the dye baths for 6 hours more at the room temperature. Finally the samples were washed with cold water and dried under shade.

- Steeping during over night

The temperatures of both dye liquors were raised to about 50°C and then the fabric samples were entered to the dye liquors. They were kept at that temperature for 30 minutes. Then within about 5 minutes, the temperature was raised to about 95°C. It was kept for another 25 minutes more at that temperature. Then the temperature was allowed to reduce to the room temperature. Dyed samples were left steeped in the dye baths over night at the room temperature.

Finally, all the samples were washed with cold water and dried under shade. The differences between the colours were visually assessed under the colour assessment cabinet using the grey scale for assessing staining (ISO 105 AO3: 1993).

1.1.7 Experiments to evaluate the dye affinity at different pH values

The dye affinity of both dyes under different pH values from acidic to alkali was evaluated. As the dye liquors, neutrally extracted dyes of both dyes were taken. Three types of fabrics were taken to dye. They were desized and bleached cotton, mercerized cotton and polyester fabrics. The temperature of both dye liquors was raised to about 50°C and the fabric samples were entered to the dye liquors. Then they were kept at that temperature for 30 minutes. Within about 5 minutes, the temperature was raised to about 95°C. To improve the alkalinity of the dye liquors, sodium hydroxide was added during dyeing and pH was adjusted in to 13. To improve the acidity of the dye liquors, lemon juice was added during

dyeing and the pH was adjusted in to 4. It was kept for another 25 minutes at that temperature. Then the temperature was allowed to reduce to the room temperature. Dyed samples were left steeped in the dye baths over night at the room temperature. Finally, all the samples were washed with cold water and dried under shade. The differences between the colours were visually assessed under the colour assessment cabinet using the grey scale for assessing staining (ISO 105 AO3: 1993).

1.1.8 Colour fastness evaluations of the dyes

Colour fastness tests were done for dyed samples of both the dyes to assess their fastness properties to light and washing. None mordanted and mordanted samples dyed at neutral, alkaline and acidic conditions were subjected to these fastness tests.

1.1.9 Wash fastness test

Wash fastness test was done using the beaker dyeing machine. The sample fabric size was 10cm x 4cm and the weight of a single sample was 1g. First, none dyed cotton fabric pieces of 5cm x 4cm were stitched to each dyed samples. 50ml of water was added to each beaker in the beaker dyeing machine. 5g/l common liquid detergent was added to each beaker. The temperature of the beaker dyeing machine was adjusted to 50°C. The dyed samples were attached to the hooks of beaker lid and washed for 45 minutes. After washing, all the samples were dried in drying oven at a temperature below 60°C. Changes in colour were visually assessed under the colour assessment cabinet using the grey scale for assessing change in colour (ISO 105 AO2: 1993).



Figure 1.6 Dyed fabric samples stitched to none dyed samples

1.1.10 Light fastness test

Light fastness test was done using the light fastness tester. The sample fabric size was 1cm x 5cm. All the samples were exposed to the UV light during time period of 53 hours until the standard sample was getting in to 3 according to the grey scale. Finally the changes in colour were visually assessed under the colour assessment cabinet using the grey scale for assessing change in colour (ISO 105 AO2: 1993).

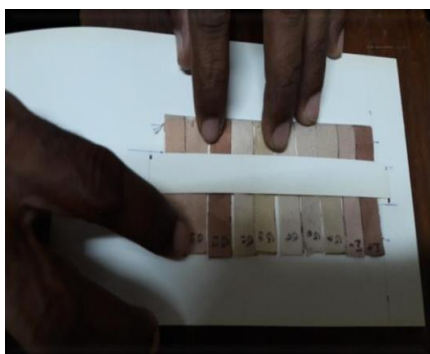


Figure 1.8 Sample preparations for light fastness test

1.1.11 Medicinal values of Thespesia populnea and Bauhinia racemosa

According to the Ayurveda, *Thespesia populnea* and *Bauhinia racemosa* plants are considered as herbal plants which are used to cure certain diseases. In the case of *Thespesia populnea*, the leaves and bark are used as a blood purifier, for piles and to prepare oils which are applied on fractures. The leaves are employed for dressing ulcers and bark on boils. Gruel prepared from the juice of the leaves and bark with grains of *Setaria italica* is given for injuries due to falls, flatulence and as a purgative. The decoction of the bark is given as an astringent, tonic and alterative. (Jayaweera, 1982). According to another record, *Thespesia populnea* plant has an

astrigent property and germ killing property. The leaves and bark of this plant are used to clean the wounds. It is often used for fractures, treatments for boils and ulcers (Compendium of Medicinal Plants, 2001). According to the other local records, the leaves, fruits and bark of the *Thespesia populnea* plant can be used to cure scabies, hives and skin infections. As the uses of *Bauhinia racemosa*, a decoction of the leaves is used to allay headaches due to malaria fever. The dried flowers and bark of the stem are used as decoction for dysentery, diarrhoea, internal haemorrhages, bleeding and threatened abortion and bleeding from haemorrhoids. It is often employed as a substitute for *Woodfordia fruticosa* (Jayaweera, 1982). These evidences have been found from several Ayurveda books and the copies of the relevant pages of these books are attached in the appendices.

Among both of them, *Thespesia populnea* can be considered as an ideal treatment for skin infectious people who have a sensitive skin and allergies. These characteristics can be important when it is used as a textile dye. An experiment was carried out to observe the curing ability of *Thespesia populnea* when applied to a textile based element.

Before carrying out the experiment, several recommendations for research ethics given by American Psychological Association were followed. First, advices on the treatment using *Thespesia populnea* have been taken from an experienced Ayurveda doctor. He was advised to boil the washed small pieces of barks in the clean portable water without adding any chemicals. All the information about the herbal value of *Thespesia populnea* and the advices given by the Ayurveda doctor was clearly informed to the selected participant. She was instructed with the purpose of the research, experimental nature of the treatment, adverse effects or potential risks, benefits etc. Information was given to the participant about how her

data will be used and what will be done with photos. The patient was voluntary participated with full knowledge about the experiment. Then the experiment was carried out under the permission of the Ayurveda doctor and the willingness of the patient. The patient was a middle aged woman suffering eczema because of rheumatics. Earlier, she was having diabetics slightly and at that moment she was healed with the regular use of western medicine. Additionally, she is already having cholesterol slightly and does not take any medicines but controls the dietary. During the whole treatment of *Thespesia populnea*, she did not take any medicine (either Western medicine or Ayurveda medicine) even for any other illnesses. Also she has avoided from taking torrid foods which may have a potential to improve the eczema during the treatment.

First, the bark of *Thespesia populnea* was dried and cut in to small pieces. They were washed well with water to remove impurities. Pure well water was boiled first in a fresh and clean pot which was used to boil the barks to remove germs. Then *Thespesia populnea* bark pieces were put in to the pot containing pure well water and simmered during an hour as I have done in the case of neutral extraction. After the extraction, the bark pieces were filtered from the pot and a gauze bandage was put to the extraction pot. It was simmered for an hour. The dyed gauze bandage was kept in the pot for 6 hours at the room temperature. Then the bandage was put on the patient. This practice was done continuously over a week and the results were observed to determine the germ curing ability of *Thespesia populnea* extract used on the bandage. The figure 1.10 shows the gauze bandage after dyed and the figure 1.11 shows the nature of the wound and the bandage wrapped around the eczema.



Figure 1.10 Dyed gauze bandage



Figure 1.11 The dyed bandage wrapped around an eczema

RESULTS AND DATA ANALYSIS

2.1 Dye extraction at different pH values

The dyes extracted under different pH values were kept separately. They were used to dye cotton fabric samples under some conditions to compare the strength of the dye liquor. The figures 2.1 to 2.5 show dye liquors and 5 dyed samples of *Thespesia populnea* at 5 different pH values.

Figure 2.1 Thespesia populnea extracted dye at pH 3 (a) with dyed sample (b)



Figure 2.2 Thespesia populnea extracted dye at pH 5 (a) with dyed sample (b)

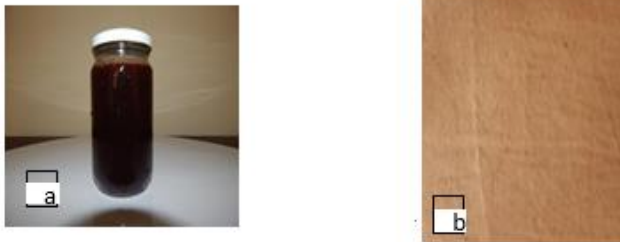


Figure 2.3 Thespesia populnea extracted dye at pH 6-7 (a) with dyed sample (b)



Figure 2.4 Thespesia populnea extracted dye at pH 8-9 (a) with dyed sample (b)

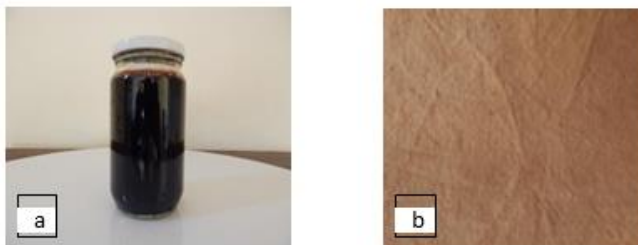


Figure 2.5 Thespesia populnea extracted dye at pH 13-14 (a) with dyed sample (b)

The figures 2.6 to 2.10 depict dye liquors and 5 dyed samples of *Bauhinia racemosa* at 5 different pH values.

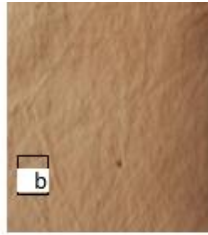


Figure 2.6 Bauhinia racemosa extracted dye at pH 3 (a) with dyed sample (b)



Figure 2.7 Bauhinia racemosa extracted dye at pH 5 (a) with dyed sample (b)



Figure 2.8 Bauhinia racemosa extracted dye at pH 6-7 (a) with dyed sample (b)

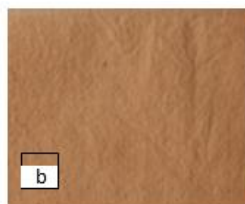
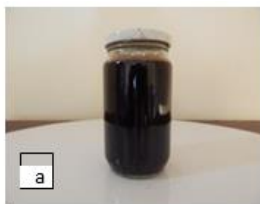


Figure 2.9 Bauhinia racemosa extracted dye at pH 8-9 (a) with dyed sample (b)



Figure 2.10 Bauhinia racemosa extracted dye at pH 13-14 (a) with dyed sample (b)

According to the results, the colours of the extracted liquors from both plants appeared similar. It can be seen that a considerable degree of colour components were discharged to the water during the extraction, even at the neutral state. But when the samples that were dyed separately using these liquors are observed, there was a difference between the hue of samples. It was observed that shades can be obtained when extraction was done using alkalis for both the plants. Tints can be obtained when the extraction was done using acids. The highest depth of shade of both dyes was observed in the samples dyed with dye liquors extracted with sodium hydroxide with the pH value of 13-14. Similar results were obtained in other experiments. According to a previous work, when the extraction of colour component from jackfruit wood was done under various pH conditions; the optimum condition for extraction was at pH-11.0 (Samanta et al., 2007). In contrary to what was observed in this work, another research work done by Samantha et al. in 2006 has proved that when extraction of red sandal wood is done under various pH conditions, the optimum condition for extraction of the colour component was at pH 4, giving absorbance of colour component at $2.63\lambda 603.0\text{nm}$ (As cited in Samanta et al., 2009).

2.2 Dye extraction at different temperatures and time

To investigate the differences between different extraction times and temperatures, some experiments were done. The figure 2.11 to 2.13 show dye liquors and dyed samples of *Thespesia populnea* extracted at 50°C for 3 different dyeing times such as 30 minutes, 45 minutes and 60 minutes respectively. Both extraction temperature and time have an effect on the amount of dye extracted by aqueous medium. This can be

observed from the colour of the extracted dye liquor. The differences were also observed in the dyed samples.

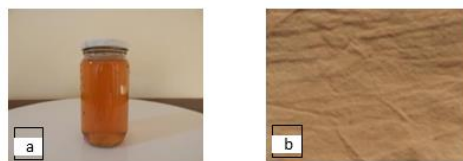


Figure 2.11 *Thespesia populnea* extracted dye at 50°C over 30 mins (a) with dyed sample (b)

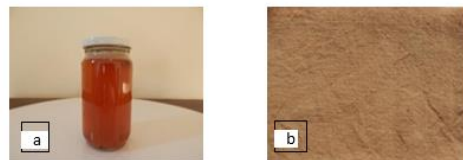


Figure 2.12 *Thespesia populnea* extracted dye at 50°C over 45 mins (a) with dyed sample (b)



Figure 2.13 *Thespesia populnea* extracted dye at 50°C over 60 mins (a) with dyed sample (b)

The figures 2.14 to 2.16 show dye liquors and dyed samples of *Thespesia populnea* extracted at 100°C for 3 different dyeing times such as 30 minutes, 45 minutes and 60 minutes respectively.



Figure 2.14 *Thespesia populnea* extracted dye at 100°C over 30 mins (a) with dyed sample (b)

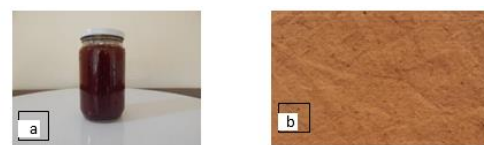


Figure 2.15 *Thespesia populnea* extracted dye at 100°C over 45 mins (a) with dyed sample (b)

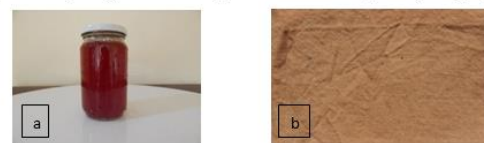


Figure 2.16 *Thespesia populnea* extracted dye at 100°C over 60 mins (a) with dyed sample (b)

The figures 2.17 to 2.19 show dye liquors and dyed samples of Bauhinia racemosa extracted at 50°C for 3 different dyeing times such as 30 minutes, 45 minutes and 60 minutes respectively.

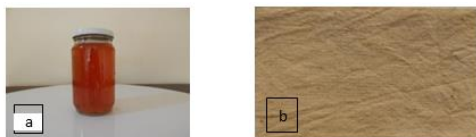


Figure 2.17 Bauhinia racemosa extracted dye at 50°C over 30 mins (a) with dyed sample (b)

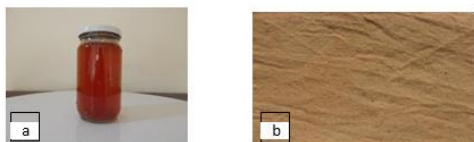


Figure 2.18 Bauhinia racemosa extracted dye at 50°C over 45 mins (a) with dyed sample (b)



Figure 2.19 Bauhinia racemosa extracted dye at 50°C over 60 mins (a) with dyed sample (b)

The figures 2.20 to 2.22 show dye liquors and dyed samples of Bauhinia racemosa extracted at 100°C for 3 different dyeing times such as 30 minutes, 45 minutes and 60 minutes respectively.

Figure 2.20 Bauhinia racemosa extracted dye at 100°C over 30 mins (a) with dyed sample (b)

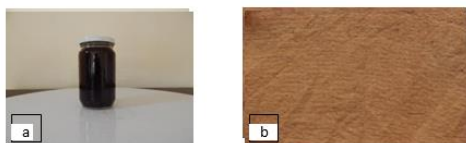


Figure 2.21 Bauhinia racemosa extracted dye at 100°C over 45 mins (a) with dyed sample (b)

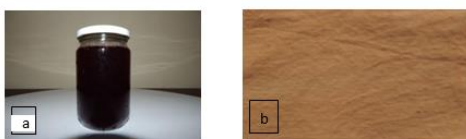


Figure 2.22 Bauhinia racemosa extracted dye at 100°C over 60 mins (a) with dyed sample (b)

The results show that with the increase of time and temperature, depth of shade can be improved. The depth of shade was significantly low when the extraction was done at temperatures around 50°C. According to the results, high depth of shade in colour yield was observed in both plant extractions, when the extraction was done close to 100°C over a duration of 60 minutes. However, when the extraction is continued at 100°C further, it is noticed a decrease in volume of liquor due to vaporization. Therefore, the temperature was kept about 90°C and slowly raised to 100°C when the duration was reaching to 60 minutes. According to the earlier experiments that have been done on improving the colour fastness of the selected natural dyes on cotton, optimum extraction time and temperature were determined. After boiling annatto seeds for 60 minutes, it was found that a reasonably good amount of dye can be extracted. Around 45 minutes dyeing time, it was noticed to produce good colour depth on cotton (Prabhavathi et al., 2014).

2.3 Effects of mordants

Pre mordanting was done for all fabric samples. Several samples were not mordanted but dyed in all dye liquors. The obtained results are discussed below. The figures 2.23 and 2.24 show the pre mordanted fabric samples with 4 different mordants before dyeing

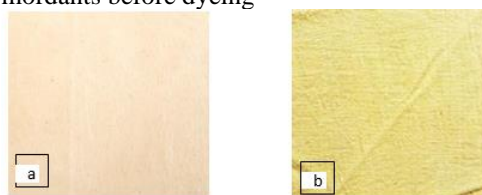


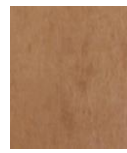


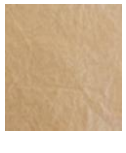
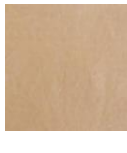

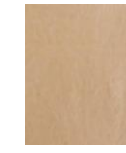

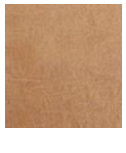


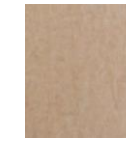



Figure 2.23 Samples pre mordanted with alum (a) and Sepalika flowers (b)



Figure 2.24 samples pre mordanted with ferrous sulphate (a) and copper sulphate (b)


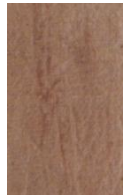
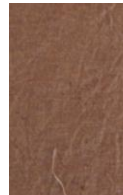


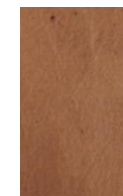
Table 2.1 Mordanted and none mordanted samples dyed with *Thespesia populnea* dye liquors of different pH

Nature of dye liquor	Sample without a mordant	Sample mordanted with alum	Sample mordanted with FeSO ₄	Sample mordanted with CuSO ₄	Sample mordanted with Sepalika
Neutrally extracted dye liquor					
Alkaline extracted dye liquor					
Acidic extracted dye liquor					

The table 2.1 shows the mordanted and none mordanted fabric samples dyed with *Thespesia populnea* solution extracted at different pH values. The differences between dyed samples are very significant. Different colour depths can be obtained by using different mordants and different dye liquors. In *Thespesia populnea* dyed samples, the sample

mordanted with ferrous sulphate and dyed in neutrally extracted dye liquor and the sample mordanted with ferrous sulphate dyed in acidic extracted dye liquor have produced deep shades. Comparatively light hues are observed in the samples mordanted with alum and Sepalika flowers for all three dye liquors. None mordanted samples have exhibited similar hues compared to the mordanted samples.

Table 2.2 Mordanted and none mordanted samples dyed with *Bauhinia racemosa* dye liquors of different pH

Nature of dye liquor	Sample without a mordant	Sample mordanted with alum	Sample mordanted with FeSO ₄	Sample mordanted with CuSO ₄	Sample mordanted with Sepalika
Neutral extracted dye liquor					
Alkaline extracted dye liquor					
Acidic extracted dye liquor					

The table 2.2 shows the mordanted and not mordanted fabric samples dyed with *Bauhinia racemosa* solution extracted at different pH values. The differences between dyed samples are very significant. Different colour depths can be obtained by using different mordants and different dye liquors. When considering the samples dyed in *Bauhinia racemosa* dye liquors, almost all the samples have deep shades. However it can be seen differences between the hue of the samples dyed with different mordants and different dye liquors. The sample mordanted with copper sulphate and dyed in neutrally

extracted dye liquor and the sample mordanted with copper sulphate dyed in alkaline extracted dye liquor have the deepest shades among all.

According to a previous research on dye extraction from *Shorea robusta* bark, CuSO₄ gave the maximum darkness among all types of mordants (Sahoo T. et al., 2014). Another experiment has proved that, in pre mordanting, ferrous sulphate had the best colour strength followed by iron water and alum had the lowest colour strength. According to the results that I have obtained, we can agree with the statements that ferrous sulphate and

copper sulphate allow shades, while alum allows tints. In addition, Sepalika flowers also contribute to provide tints.

2.4 Dyeing behaviour of the extracted dyes

As already mentioned experimental dyeing was carried out to investigate the dyeing behaviour of the extracted dyes. In the following section results of the experiments are discussed.

2.4.1 Results of the experiments done to evaluate the optimum dyeing time

How dyeing time was varied had been already described above. After dyeing the samples under four dyeing times, the hue of the samples were observed. Results were obtained as numerical values by visually observing the hue of the samples under the colour assessment cabinet using the grey scale for assessing staining (ISO 105 AO3: 1993). These results are given in the tables 2.3 and 2.4.

Table 2.3 Results of the experiment carried out to determine the optimum dyeing time for *Thespesia populnea*









Duration of dyeing	Samples	Staining values
30 minutes		2
45 minutes		2
60 minutes		2
75 minutes		1-2

Table 2.4 Results of the experiment carried out to determine the optimum dyeing time for *Bauhinia racemosa*

Duration of dyeing	Samples	Staining values
30 minutes		2
45 minutes		1-2
60 minutes		1-2
75 minutes		1-2

According to the results, there is no significant difference between samples dyed under different dyeing times for both the dyes, however when the dyeing is done for 75minutes, a small influence of dyeing time can be seen for *Thespesia populnea* dye. An earlier experiment done to improve the colour fastness of a natural dye extracted from annatto seeds on cotton proved that around 45 minutes, dyeing time was noticed to produce a good colour depth on cotton (Prabhavathi et al., 2014).

2.4.2 Results of the experiments done to evaluate the optimum dye steeping time As explained in previous section, dyeing was carried out under different durations of steeping. Three different time periods were used. The hue of the samples dyed with three steeping times was compared. Results were obtained in numerical values by visual observation under the colour assessment cabinet using the grey scale for assessing staining (ISO 105 AO3: 1993). The tables 2.5 and 2.6 illustrate the outcomes for the two different dyes.

Table 2.5 Results of the experiment carried out to determine the optimum dye steeping time for *Thespesia populnea*

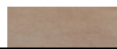





Steeping time	Samples	Staining value
30 minutes		3
6 hours		2-3
Over night		2

Table 2.6 Results of the experiment carried out to determine the optimum dye steeping time for *Bauhinia racemosa*

Steeping time	Samples	Staining value
30 minutes		1-2
6 hours		1
Over night		1

According to the results of *Thespesia populnea* dyeing, deep shades were obtained for steeping times of 6 hours and over. A shade change can be observed over the steeping time and the staining was best when the sample was steeped overnight. When concerning the results of *Bauhinia racemosa* dyeing, deep shades were obtained in steeping when the samples were left 6 hours and over as in the case of *Thespesia populnea* dyeing. A significant shade change is also noticed

over the steeping time. The best staining was obtained by steeping over 6 hours.

2.4.3 Results of the experiments done to evaluate the dye affinity at different pH values

Three different fabrics were dyed in dye liquors with different pH values. The hue of the dyed samples was observed under the colour assessment cabinet using the grey scale for assessing staining (ISO 105 AO3: 1993). The tables 2.7 and 2.8 show the effect of pH on dye affinity of different fabrics.

Table 2.7 Effect of pH on dye affinity of different fabrics for *Thespesia populnea*



Fabric type	Fabric dyed at pH 13-14	Staining value	Fabric dyed at pH 4-5	Staining value
Desized and bleached cotton		2-3		2
Mercerized cotton		2		2
polyester		4		3-4

Table 2.7 Effect of pH on dye affinity of different fabrics for *Thespesia populnea*





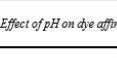
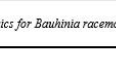





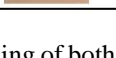
Fabric type	Fabric dyed at pH 13-14	Staining value	Fabric dyed at pH 4-5	Staining value
Desized and bleached cotton		2-3		2
Mercerized cotton		2		2
polyester		4		3-4

Table 2.8 Effect of pH on dye affinity of different fabrics for *Bauhinia racemosa*

Fabric type	Fabric dyed at pH 13-14	Staining value	Fabric dyed at pH 4-5	Staining value
Desized and bleached cotton		2-3		2-3
Mercerized cotton		2		2
polyester		3-4		3

The results show a good staining of both dyes when dyed using desized and bleached cotton and mercerized cotton. But polyester was having a poor dye

affinity to both dyes. There can be seen a difference in hues between the samples dyed at acid and alkali added dye liquors. However, the dye affinity was better when an alkali was added to the dye liquor to dye cotton. In the case of polyester dyeing, dye affinity was comparatively improved by the addition of acids.

2.5 Fastness properties of extracted dyes

As described above, both wash fastness and fastness to light were tested. The results of these tests are discussed below.

2.5.1 Results of the wash fastness test

After the washing was over, a considerable degree of staining on none dyed samples was not observed, however there could be seen certain changes in colour after washing. Changes in colour were visually assessed under the colour assessment cabinet using the grey scale for assessing change in colour (ISO 105 AO2: 1993). The results are given in the tables 2.9 to 2.12 for *Thespesia populnea* and *Bauhinia racemosa* respectively.

Table 2.9 Comparison of dyed samples with *Thespesia populnea* before and after washing

Nature of dye liquor	None mordant sample	Sample with alum	Sample with FeSO ₄	Sample with CuSO ₄	Sample with Sepalika
Neutral extraction Pre wash					
After wash					
Alkaline extraction Pre wash					
After wash					
Acidic extraction Pre wash					
After wash					

Table 2.10 Change in colour values due to washing of samples dyed with *Thespesia populnea*

Nature of the dye liquor	None mordant sample	Sample with alum	Sample with FeSO ₄	Sample with CuSO ₄	Sample with Sepalika
Neutrally extracted liquor	4-5	4-5	4-5	4-5	3-4
Alkaline extracted liquor	3	4-5	4-5	4-5	3-4
Acidic extracted liquor	3	3	3-4	3-4	3-4

Table 2.11 Comparison of dyed samples with *Bauhinia racemosa* before and after washing


Nature of dye liquor	None mordant sample	Sample with alum	Sample with FeSO ₄	Sample with CuSO ₄	Sample with Sepalika
Neutral extraction Pre wash					
After wash					
Alkaline extraction Pre wash					
After wash					
Acidic extraction Pre wash					
After wash					

Table 2.12 Change in colour values due to washing of samples dyed with *Bauhinia racemosa*

Nature of the dye liquor	None mordant sample	Sample with alum	Sample with FeSO ₄	Sample with CuSO ₄	Sample with Sepalika
Neutrally extracted liquor	4	4-5	4	4-5	4
Alkaline extracted liquor	4	4	4-5	4-5	4
Acidic extracted liquor	3-4	4-5	4-5	4-5	4-5

The results show that there are differences between pre wash and after wash samples. When comparing the values obtained from change in colour assessment for *Thespesia populnea* dye, a least change in colour was observed in the samples dyed in neutrally extracted and alkaline extracted dye liquors mordanted with alum, copper sulphate and ferrous sulphate. Sepalika mordanted samples also show a low change in colour after washing. In the case of *Bauhinia racemosa*, least change in colour was observed after washing in the samples dyed in neutrally extracted liquor mordanted with alum and copper sulphate, alkaline extracted liquor mordanted with ferrous sulphate, copper sulphate and acidic extracted liquor with all mordanted samples. Even a mordant is not used; both dyes have shown a moderately to good wash fastness. According to a previous work done by Mahale G Sakshi and Sunanda R.K. (as cited in Samanta A.

K. And Agarwal P. 2009), cotton yarns when treated with *Acalypha* dye after pre mordanting with potash alum, potassium dichromate, copper sulphate and ferrous sulphate showed excellent colour fastness properties. It can also be applicable to the dyes of the present study. Another publication reported that the cotton fabrics dyed with dyes extracted from the stem bark of *Albizia coriaria* without the use of mordants exhibited a very good wash fastness of 4-5 (Janani, 2014).

2.5.2 Results of the light fastness test

As described earlier, fastness to light was evaluated. When the standard sample was getting 3 according to the grey scale, changes in colour were visually assessed under the colour assessment cabinet using the grey scale for assessing change in colour (ISO 105 AO2: 1993). The results of these tests are given in the tables 2.13 to 2.16.

Table 2.13 Comparison of fabric samples dyed with *Thespesia populnea* before and after exposing to light

Nature of dye liquor	None mordant sample	Sample with alum	Sample with FeSO ₄	Sample with CuSO ₄	Sample with Sepalika
Neutral extraction	Before exposure				
	After exposure				
Alkaline extraction	Before exposure				
	After exposure				
Acidic extraction	Before exposure				
	After exposure				

Table 2.14 Change in colour values due to light of samples dyed with *Thespesia populnea*

Nature of the dye liquor	None mordant sample	Sample with alum	Sample with FeSO ₄	Sample with CuSO ₄	Sample with Sepalika
Neutrally extracted liquor	3	2-3	3	3	3
Alkaline extracted liquor	3-4	3-4	3-4	3-4	3-4
Acidic extracted liquor	3	2-3	3	3	3

Table 2.15 Comparison of fabric samples dyed with Bauhinia racemosa before and after exposing to light

Nature of dye liquor	None mordant sample	Sample with alum	Sample with FeSO ₄	Sample with CuSO ₄	Sample with Sepalika	
Neutral extraction	Before exposure					
	After exposure					
Alkaline extraction	Before exposure					
	After exposure					
Acidic extraction	Before exposure					
	After exposure					

Table 2.16 Change in colour values due to light of samples dyed with Bauhinia racemosa

Nature of the dye liquor	None mordant sample	Sample with alum	Sample with FeSO ₄	Sample with CuSO ₄	Sample with Sepalika
Neutrally extracted liquor	2-3	2-3	3	3	3
Alkaline extracted liquor	3	2-3	2-3	2-3	3
Acidic extracted liquor	3	3	3	3	3

According to the results, little change of colour was observed in samples dyed with *Thespesia populnea* liquor extracted in alkaline condition. However, samples mordanted with alum and dyed in neutrally extracted and acidic extracted dye liquors have shown changes in colour after exposing to the light. Other samples have shown moderate changes in colour. In the case of *Bauhinia racemosa*, not mordanted sample and alum mordanted sample dyed in neutrally extracted liquor,

alum mordanted, ferrous sulphate mordanted and copper sulphate mordanted samples and dyed in alkaline extracted liquors have shown a considerable change in colour for light. Others have shown moderate change in colour. Comparatively, light fastness of *Bauhinia racemosa* dyed samples is lower than that of the *Thespesia populnea* dyed samples. According to previous research, most of the dyes tested in the yellow natural dye study had only poor to moderate light fastness (Crews, 1982). Another report has mentioned that the cotton fabrics dyed with dyes extracted from the stem bark of *Albizia coriaria* without the use of mordants exhibited a moderate light fastness of 4 (Janani, 2014).

2.6 Results and discussion of the experiments done to evaluate the medicinal value of *Thespesia populnea*

As explained above, *Thespesia populnea* dye was neutrally extracted and a gauze bandage was dyed without adding any chemical to the dye bath. The dyed bandage was then put on a patient who was having eczema. This practice was done over a week to find out if any cure can be seen. During this treatment any other treatments or medications were avoided.

Figure 2.25 The appearance of the eczema at the beginning





Figure 2.26 The bandaged eczema with Thespesia populnea dyed bandage



Figure 2.27 Appearance of the eczema after 2 days, 4 days and 6 days during the treatment



Figure 2.28 Appearance of the Eczema after 11 days

The results have shown that *Thespesia populnea* extraction has a considerable healing ability against those wounds. It was apparent that the sorely effect of the wound was reduced little by little within several days and the wound was considerably cured. It has been already

mentioned in Ayurveda books. The leaves and bark are used as a blood purifier, for piles and to prepare oils which are applied on fractures. The leaves are employed for dressing ulcers and bark on boils (Jayaweera, 1982). According to another record, *Thespesia populnea* plant has an astringent property and germ killing property. The leaves and bark of this plant are used to clean the wounds. It is often used for fractures, treatments for boils and ulcers (Compendium of Medicinal Plants, 2001). In another Ayurveda book explains that the boiled leaves, fruits and bark of *Thespesia populnea* can be used to wash scratchy eczema, ulcers, scabies etc. Also *Thespesia populnea* is considered as a germ killer for the skin infections caused by rheumatoid (Weragoda, 1994). It is apparent that the wound was considerably healed with the continuous use of *Thespesia populnea* dyed bandage.

There can be found several examples for herbal dyes apart from those given in the present study. Previous research reports the dye ability and antimicrobial properties of cotton fabrics finished with *Punica granatum* extracts. The antimicrobial properties were tested by the disc diffusion method and parallel streak method (Rajendran, 2011). Another experiment done for the study on antibacterial activity of natural dye from the bark of *Araucaria columnaris* and its application in textile cotton fabrics has shown the antibacterial property using parallel streak method (Saranya et al., 2014). These methods have scientifically proved the antibacterial property of the extracts. As a future prospect, similar experiments can be tested for the plant extracts used in present study to prove the antibacterial properties scientifically.

CONCLUSION AND RECOMMENDATIONS

The present study has shown that it is possible to use the barks of *Thespesia populnea* and *Bauhinia racemosa* to extract natural dyes for colouring cotton textiles. The aqueous extraction method is chosen for both plants to extract dyes however it is possible to consider the alcoholic extraction in further experiments. The deepness of the hues of extracted liquors is difficult to evaluate visually but it can be evaluated when a fabric was dyed using these dye liquors. Thus the depth of shade in extraction can be improved by the addition of an alkali during extraction of both dyes. The results have shown low colour yield for both dyes when the extraction was done at the temperatures about 50°C. The colour yield was apparently improved when the extraction was done at the temperatures close to 100°C over 30 minutes.

In the case of dyeing, duration of dyeing did not affect heavily on staining. However, duration of steeping affected staining of both dyes. According to the results, deep shades can be obtained steeping the samples for 6 hours or more in the dye bath. Use of mordants has changed the hue of the dyed samples. Ferrous sulphate and copper sulphate mordants have contributed to improve the original hue while alum and Sepalika flowers were contributed to lighten the original hue. In this case, Sepalika flowers can be introduced to the natural mordants as a lightening element. In most of experiments done on natural dyes, it is mentioned that the natural dyes have poor affinity towards synthetic fibres. According to the results obtained by the present study, both *Thespesia populnea* and *Bauhinia racemosa* dyes have shown poor affinity towards polyester fabrics. When considering the effect of pH of the dye liquor during dyeing, increased pH has contributed to improve the dye affinity of cotton. Also the pH difference, whether the dyeing liquor is alkaline or acidic allows the colour changes in dyed

samples. In the case of fastness properties of these dyes, use of mordants has improved the wash fastness of both dyes. The change in colour was apparently least in wash fastness but the light fastness properties are not very satisfactory. The change in colour of *Thespesia populnea* dye is moderate even in mordanted samples. Samples dyed in alkaline extracted dye liquor have shown somewhat good fastness to light. When considering *Bauhinia racemosa* dye, change in colour for light is high comparing to the *Thespesia populnea*. In contrary to *Thespesia populnea*, it is observed a bigger change in colour of samples that were dyed in alkaline extracted liquor. None mordanted samples dyed in both plant extractions have shown moderate wash and light fastness.

The herbal value of the used plants was evaluated. Referring the Ayurveda books, neutrally extracted *Thespesia populnea* was used on a bandage and put on a patient having eczema. The effect was observed daily. It was apparent that the sorely effect of the wound was reduced little by little and the wound was considerably cured with the daily application of dyed bandages. In addition, there cannot be observed any adverse effect or allergy during or after the treatment. However, further scientific investigations have to be done to find out the antimicrobial properties of *Thespesia populnea* dye. Through them, it may be possible to determine the suitability of the dye for the end use.

During the study, any harmful synthetic chemicals were not added in extraction, dyeing or mordanting processes. The used chemicals do not include any heavy metals and do not cause hazardous effects to the environment. Most of the time, it was aimed to use natural things to develop the dyes. The used plants sources are also available in many areas of the country. Among them, *Thespesia populnea* is also considered as an invasive species therefore

it can be commercially used in an effective way. Furthermore, the present study is an effort to promote the use of natural plant sources to extract dyes suitable for textiles.

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