Extraction and Application of Natural Dyestuff from Bark of Siris Tree

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Abstract: *The main aim of this research is to reduce the environmental pollution problems related to the use of synthetic dyes. The objectives of this research are to extract the natural dyestuff from Siris bark in powder form, to study the effect of colours produced on dyed cotton materials with various types of mordant and to study the fastness properties on dyed cotton materials.*

In this study, the required raw Siris bark samples are collected and the preliminary phytochemical tests are performed to investigate the types of chemical compounds in the Siris bark. The best extraction method is determined by using four types of extraction mediums. Then, the optimum dye extraction conditions is determined by varying the solvent ratio, extraction time and material to liquor ratio, and natural dyestuff is extracted as in powder form by using this selected medium. And then, natural dye powder extract is applied on cotton materials by using seven different types of mordant. After that, the effect of mordant on colour fastness is determined by doing washing, rubbing and light fastness tests on dyed cotton materials.

In this research, alcoholic medium is chosen as optimum extraction medium because this medium gives soft, attractive and subtle colour on cotton materials. And, the optimum condition for extraction is occurred in solvent ratio of 30% alcohol, five hour extraction time and material to liquor ratio of 1:10. From the fastness test results, all types of mordant give acceptable fastness properties on dyed cotton materials.

Keywords: *Extraction; Natural dyestuff; Mordant; Colour Fastness*

1. INTRODUCTION:

Dyes are generally used in textile, paper, cosmetic, food, pharmaceutical and leather industries. Dyes and textiles are an important part of our everyday life. In the production of clothes, dyeing is a very important process. The art of dyeing was an old as human civilization. The dyeing process used takes a lot of time and energy but in recent years there has been an interest manifested towards natural dyes because of the ecological movement, bio-degradability and higher compatibility of natural dyes with environment [1].

The use of non-allergic, non-toxic and ecofriendly natural dyes on textiles has become a matter of significant importance due to the increased environmental awareness in order to avoid some hazardous synthetic dyes [2]. Nowadays, a number of commercial dyers and small textile houses started looking at the possibilities of using natural dyes for regular basis dyeing and printing of textiles to overcome environmental pollution caused by the synthetic dyes. Hence, there is a world's movement to return to the natural dyes [3].

Natural dyes offer much more advantages including renewable sources, minimal health hazard, mild reaction conditions, no disposal problem and harmonization with nature [2]. So, the use of natural dyes in textile dyeing are very welcoming and the clothes dyed with natural dyestuff produce attractive colours. For these reasons, the research has steadily increased to extract natural colourants and use them for dyeing textiles [1]. Our interest in the extraction of the dye from plant sources has shown that a species, namely *AlbiziaLebbeck* belonging to the family *Fabaceae,* and found in Myanmar, contains a colour component which can be exploited in dyeing cotton materials. The use of the bark of *A. labbeck* as a potential source of dye is interesting and hence, the present work was undertaken to extract the colouring component of the bark and study the application on cotton materials.

A. AlbiziaLebbeck (Siris Tree)

The genus *Albizia* comprises approximately 150 species, mostly trees and shrubs native to tropical and subtropical region of Asia and Africa. *AlbiziaLebbeck* is native to deciduous and semidecidous forests in Asia from eastern Pakistan through India and Siri Lanka to Myanmar [4].

AlbiziaLebbeck is a fast-growing, medium-sized deciduous tree with a spreading umbrella-shaped crown of thin foliage and smoothish, finely fissured, grayish-brown bark. Depending on site conditions, annual height growth ranges from 0.5 to 2.0 metre; on good sites, individual trees attain an average maximum height of 18 to 25 metre. Flowers usually appear with new leaves over an extended period beginning at the end of the dry season. The fruits, flattened pods are produced in large numbers and each contains several seeds. Immature pods are green, turning straw-coloured on maturity. Seeds are small, oblong and broad, compressed and light brown in colour. Leaves, flowers and pods fall to the ground gradually during the dry season [4].

B.Nomenclature

Family Name - Fabaceae
Botical Name - AlbiziaLebbeck (L.)
Common Name - East Indian walnut, Siris-tree, Kokko, Women's tongue, Rattle pod [5].

2. MATERIALS AND METHOD:

2.1 Raw Materials

Natural dyes are obtained from vegetables, fruits, roots, stems, leaves, flowers, dried bodies of certain insects and minerals. In this study, bark of Siris tree is used as raw material for extraction of natural dyestuff because Siris tree can be found abundantly occurred in Myanmar and the bark can easily be cut from the trunk of Siris tree in any season.

The bark of Siris tree contains colouring matters. The colouring matter in the bark produces beautiful, light brown shades on cotton materials. And the collection of raw materials for dye extraction is not difficult. By these reasons, the bark of Siris tree is selected to extract the natural dyestuff in this study.

Siris bark samples are collected from Yangon Technological University Campus, Insein Township, Yangon, Myanmar. First, they are cleaned with water by brushing to remove the impurities. Then they are dried under the sun for about two to three weeks and cut into small pieces. These pieces are ground into powder form by using motor and pestle. And then, these powder samples are passed through by a sieve and they are stored in bottle for future use.

2.2 Preliminary Phytochemical Tests on Bark of Siris

In this study, preliminary phytochemical tests are performed to study the major types of chemical compounds in the bark of Siris tree.

The main compound which are commonly found in plants are alkaloid, glycoside, flavonoid, reducing sugar, saponin, steroid, phenolic compound, amino acid, cyanogenic glycoside, carbohydrate, tannin and organic acid. These tests are carried out in the Pharmaceutical Research Department of Myanma Scientific and Technological Research Department.

2.3 Extraction of Natural Dye Solution from Siris Bark

Before extracting the dye powder form, natural dye solution is extracted with four types of extraction medium such as aqueous, alkaline, acidic and alcoholic medium. The conditions for extraction of dye solution in each medium are shown in Table 2.1.

After the dye solution is extracted from each medium, these solutions are used for dyeing with cotton materials to study the effect of extraction medium. In this study, the proper medium for extraction of natural dye powder is selected based on the colour produced on dyed samples. The colour obtained on dyed cotton materials are assessed by visual observation.

Table 2.1 Condition for Dye Extraction from Siris Bark

Sr. No.	Sample Code	Extraction Medium	Solvent Ratio (%)	Temp. (˚C)	Time (hr.)	Solid to Liquid Ratio
1	Method-1	Aqueous (distilled water)	100	Boiling	1	1:10
2	Method-2	Alkaline (Na_2CO_3 : H_2O)	1:99	Boiling	1	1:10
3	Method-3	Acidic (CH_3COOH : H_2O)	1:99	Boiling	1	1:10
4	Method-4	Alcoholic (C_2H_5OH : H_2O)	50:50	Boiling	1	1:10

2.4 Extraction of Natural Dye Powder from Siris Bark

After doing the selecting of proper extraction medium, natural dyestuff is extracted as in powder form by using this selected medium. In dye powder extraction, soxhlet extraction and distillation process are used. The dye extraction is done until the raw material cannot produce the colour.

The optimum condition of dye extraction is determined by varying the solvent ratio, extraction time and material to liquor ratio.

The optimum solvent ratio is determined by using 70%, 50%, and 30% alcohol. In the determination of optimum extraction time, it is varied from three hour to nine hour at two hours time interval. Generally, the yield of dye

increases with increase in material to liquor ratio up to a certain extent. So, the optimum material to liquor ratio is studied by increasing this ratio from 1:6 to 1:12. In each experiment, the other variables are fixed except the variable which is determined in its optimum condition.

In the determination of optimum dye extraction condition, the natural dyestuff is extract by refluxing the raw material in the soxhlet chamber with the selected solvent. After that, the extracted dye solution is concentrated by distillation and the concentrated solution is decanted into a petri dish. And then, it is dried in the oven at 60˚C. In this way, natural dyestuff is extracted as in powder form and the optimum dye extraction condition is determined by the amount of dye extract.

2.5 Mordanting and Dyeing the Cotton Materials with Selected Mordant

After extracting the dye powder, dyeing process is carried out on cotton materials by using various mordants to study the colour and the effect of fastness properties on dyed samples.

Most of the natural dyes require the use of mordant prior to dyeing of textile materials. Mordants are substances which are used to fix a dye to the fibre. They also improve the take-up quality of the fabric and help to improve the colour obtained and light-fastness properties. The process of mordanting prepares the textile materials to receive the dyestuff. They should thoroughly penetrate the textile materials for successful dyeing operation. If the mordants are presented in superficial stages, then dyeing and shade development will be very uneven. So, mordant is used as an assistant for dyeing operation.

In this study, common salt, lime, alum, acetic acid, ash from banana leaf, ferrous sulphate and copper sulphate are used as mordants because they can purchased easily and also suitable for natural dyes.

In dyeing operation, simultaneous dyeing and mordanting method is used in this work. The required amount of mordant and dye used are based on the weight of goods. The conditions for simultaneous dyeing and mordanting are shown in Table 2.2.

After that, the effect of mordants on colour fastness is determined by doing washing, rubbing and light fastness tests on dyed cotton materials. Among them, the best type of mordant is selected which produces good fastness properties.

Table 2.2 Dyeing Conditions for Simultaneous Dyeing and Mordanting Method

Sr. No.	Sample Code	Types of Mordant	Composition of Mordant	Dyeing Temp. (˚C)	Dyeing Time (min)	Material to Liquor Ratio	Dye Conc. (%)
1	A1	Common Salt	Common salt - 20% o.w.g	80	60	1:30	5
	A2						10
2	B1	Alum	Alum - 10% o.w.g	80	60	1:30	5
	B2		NaOH - 5% o.w.g				10
3	C1	Lime	Lime - 15% o.w.g	80	60	1:30	5
	C2						10
4	D1	Acetic Acid	pH4	80	60	1:30	5
	D2						10
5	E1	Ash from	Ash - 5% o.w.g	80	60	1:30	5
	E2	Banana Leaf					10
6	F1	Ferrous	Ferrous Sulphate -4% o.w.g	80	60	1:30	5
	F2	Sulphate					10
7	G1	Copper	Copper Sulphate -6% o.w.g	80	60	1:30	5
	G2	Sulphate					10

2.6 Testing the Colour Fastness on Dyed Materials

The colour fastness is defined as its resistance to change in colour and to transfer at colourant from it to adjacent material during exposure to particular conditions of processing or of use.

After dyeing operation, testing of colour fastness on dyed materials are necessary to determine the fastness properties. There are many test methods for evaluating the colour fastness of dyed materials. They are fastness to light, fastness to rubbing, fastness to washing, fastness to sea water, fastness to perspiration, fastness to gas fume and fastness to storage. Among them, fastness to light, fastness to washing and fastness to rubbing are carried out in this study because they are the most important properties for consumer.

To evaluate the colour fastness of the dyed materials, the changes of colour due to the conditions of test are compared with the colour of original dyed material. And then, the colour transfer and staining on cotton fabrics are determined by using Grey Scales.

3. RESULTS AND DISCUSSIONS:

3.1 Preliminary Phytochemical Tests on Bark of Siris

Preliminary phytochemical tests is carried out on the Siris bark to investigate the presence or absence of glycoside, flavonoid, reducing sugar, alkaloid, saponin glycoside, cyanogenic glycoside, steroid, phenolic compound, amino acid, carbohydrate, tannin and organic acid. Table 3.1 shows the results of phytochemical investigation on the bark of Siris tree. According to the results, Siris bark contains alkaloid, glycoside, reducing sugar, saponin glycoside, steroid, phenolic compound, amino acid, carbohydrate and tannin. Glycoside, tannin and phenolic compound are the substances which can give the colour.

Table 3.1 Results of Phytochemical Test on the Bark of Siris Tree

Sr. No.	Type of Compound	Reagent	Observation	Result
1	Alkaloid	Mayer's reagent	White ppt.	Present
		Dragendorff's reagent	Reddish brown ppt.	
2	Glycoside	10% lead acetate solution	White ppt.	Present
3	Reducing sugar	Dilute sodium hydroxide	Red ppt.	Present
		Solution and Benedict's Solution		
4	Saponin glycoside	Shakes with water	Frothing	Present
5	Cyanogenic glycoside	Conc. sulphuric acid	No brick-red colour	Absent
6	Steroid	Acetic anhydride and	Blue colour	Present
		conc. sulphuric acid		
7	Phenolic compound	10% ferric chloride solution	Black colour	Present
8	Amino acid	Ninhydrin reagent	Violet colour spot	Present
9	Carbohydrate	10% α - napthol and	Red ring	Present
		conc. sulphuric acid		
10	Organic acid	Bromocresol green	No colour change	Neutral
11	Tannin	10% ferric chloride solution	Bluish-black colour	Present
12	Flavonoid	Conc. sulphuric acid	No pink colour	Absent
		magnesium turning		

3.2 Determination of Optimum Dye Extraction Medium from Siris Bark

Before extracting the natural dye powder, the preliminary study on extraction of dye solution is carried out. The natural dye solution is extracted from Siris bark by using four types of extraction medium such as aqueous, alkaline, acidic and alcoholic. And then, these dye solutions are used for dyeing with cotton materials. After that, the proper medium for extraction of natural dye powder is selected based on the colour produced on dyed cotton materials. These dyed samples are shown in Table 3.2.The coloured obtained on dyed cotton materials are assessed by visual observation, where the colour obtaining from Method-4 gives clear, soft, attractive and subtle colour than the other dyed samples. So, the extraction medium used in Method-4 is chosen as optimum extraction medium in this study.

Table 3.2 Coloured Developed on Dyed Cotton Materials by using Extracted Dye from Siris Bark without Mordant

Sr. No.	Sample Code	Extraction Medium	Dyed Sample
1	Method-1	Aqueous	
2	Method-2	Alkaline	
3	Method-3	Acidic	
4	Method-4	Alcoholic	

3.3 Determination of Optimum Condition for Extraction of Natural Dyestuff from Siris Bark

After being selected the best extraction medium, the optimum conditions of dye extraction is determined by varying several parameters such as solvent ratio, extraction time and material to liquor ratio.

3.3.1 Effect of Solvent Ratio

The effect of solvent ratio on dye yield is studied by varying the percentage ratio of alcohol and water. So, the study is carried out by using 70%, 50% and 30% alcohol. It is found that, 30% alcohol gives the maximum dye extract than the others. The conditions for the effect of solvent ratio on dye extraction are shown in Table 3.3.

Table 3.3 Effect of Solvent Ratio on Dye Extraction

Sr.	Solvent Ratio (%)		Time	Material to	Temp.	Yield
No.	Alcohol	Water	(hr.)	Liquor Ratio	(˚C)	(%)
1	70	30	5	1:10	80	4.83
2	50	50	5	1:10	80	4.96
3	30	70	5	1:10	80	8.31

3.3.2 Effect of Time

In the study of the effect of extraction time, natural dye powder is extracted by varying the extraction time for 3 hrs, 5 hrs, 7 hrs and 9 hrs, respectively. The maximum yield percent of dye extract is observed with 5 hrs extraction time, but after that decrease in yield percent of dye is observed. The results are shown in Table 3.4.

Table 3.4 Effect of Time on Dye Extraction

Sr.	Time	Solvent Ratio (%)		Material to	Temp.	Yield
No.	(hr.)	Alcohol	Water	Liquor Ratio	(˚C)	(%)
1	3	30	70	1:10	80	5.47
2	5	30	70	1:10	80	8.31
3	7	30	70	1:10	80	8.26
4	9	30	70	1:10	80	6.69

3.3.3 Effect of Material to Liquor Ratio

The maximum dye extraction is studied by varying the material to liquor ratio from 1:6 to 1:12. Yield percent of dye increases with the increase in material to liquor ratio 1:10. After that there is not much increase in yield percent as shown in Table 3.5.

Table 3.5 Effect of Material to Liquor Ration on Dye Extraction

Sr.	Material to	Solvent Ratio (%)		Time	Temp.	Yield
No.	Liquor Ratio	Alcohol	Water	(hr.)	(˚C)	(%)
1	1:6	30	70	5	80	4.98
2	1:8	30	70	5	80	5.57
3	1:10	30	70	5	80	8.31
4	1:12	30	70	5	80	8.00

3.3.4 Optimum Conditions for Extraction of Natural Dye

From these studies, the conditions for extraction can be optimized in this way and the required amount of dyestuff for the whole study is extracted by using these conditions, as shown in Table 3.6.

In this study, extraction processes are done at 80˚C because it is learnt that extraction temperature beyond 80˚C the characteristics of the dyes may change and the solubility of extracted dyes decreases.

Table 3.6 Optimum Conditions for Extraction of Natural Dye

Solvent Ratio (%)		Time	Material to	Temp.	Yield
Alcohol	Water	(hr.)	Liquor Ratio	(˚C)	(%)
30	70	5	1:10	80	8.31

3.4 Selection of the Best Type of Mordant for Dyeing Operation

The mordants used in this study are common salt, alum, lime, acetic acid, ash from banana leaf, ferrous sulphate and copper sulphate. Among them, the best type of mordant is selected which produces good fastness properties.

Simultaneous dyeing and mordanting method is used in this work. It can be seen that different colour tone is obtained by using different mordants as shown in Table 3.7.

Table 3.7 Colour Developed on Dyed Cotton Materials with Various Types of Mordant

Sr. No.	Sample Code	Types of	Dyed Sample

		Mordant	Dye Conc. (5%)	Dye Conc. (10%)
1	A1, A2	Common Salt		
2	B1, B2	Alum		
3	C1, C2	Lime		
4	D1, D2	Acetic Acid		
5	E1, E2	Ash from Banana Leaf		
6	F1, F2	Ferrous Sulphate		
7	G1, G2	Copper SulphateSulphate		

3.5Fastness Properties on Dyed Cotton Materials

After dyeing operation is carried out, the colour fastness properties of dyed cotton materials are studied by using washing, rubbing and light fastness.

The colour fastness results of dyed cotton materials for each group of mordant are shown in Table 3.8, where fairly good light fastness and good dry and wet rubbing fastness can be observed in all cotton materials with natural dyestuff extracted from Siris bark.

In washing fastness, the change of shade ratings in all samples show fairly good in fastness and staining on cotton exhibit excellent fastness grade respectively.

From the fastness test results on dyed cotton materials, all types of mordant give good fastness properties and the colour developed on cotton materials by using seven different types of mordant give attractive colour.

So, all types of mordant used in this study give acceptable fastness properties on dyed cotton materials. Therefore, suitable mordant can be selected to use in dyeing operation depending on the colour desired.

Table 3.8 Colour Fastness Results of Dyed Cotton Materials by using Different Mordants

Sr.	Types of	Sample	Washing Fastness		Rubbing Fastness		Light Fastness
No.	Mordants	Code	Change	Staining	Dry	Wet	Change of
			of Shade	on Cotton			Shade
1	Common Salt	A1, A2	3	5	4	4	4
2	Alum	B1, B2	3	5	4	4	4
3	Lime	C1, C2	3	5	4	4	4
4	Acetic Acid	D1, D2	3	5	4	4	4
5	Ash from Banana Leaf	E1, E2	3	5	4	4	4
6	Ferrous Sulphate	F1, F2	3	5	4	4	4
7	Copper Sulphate	G1, G2	3	5	4	4	4

Change of shade 1=poor, 2=fair, 3=fairly good, 4=good, 5=very good
Staining on cotton 1=much, 2=considerable, 3=distinct, 4=slight, 5=virtually none
Light fastness grade 1=very poor, 2=poor, 3=fair, 4=fairly good, 5=good, 6=very good, 7=excellent, 8=outstanding

4. CONCLUSIONS:

From the study of "Extraction and Application of Natural Dyestuff from Bark of Siris Tree", the following conclusions can be drawn.

- According to phytochemical test, the bark of Siris tree contains alkaloid, glycoside, reducing sugar, saponin, steroid, phenolic compound, amino acid, carbohydrate and tannin. Among these compounds, glycoside, tannin and phenolic compound are regarded as colouring matters because they provide the colour in natural dye application.
- In this research, the natural dye powder is extracted from Siris bark with alcoholic medium which gives the proper shade of colour on cotton materials.

- Optimum dye extraction condition is occurred in solvent ratio of 30% alcohol, five hour extraction time and the material to liquor ratio of 1:10.
- Colour fastness properties of cotton materials dyed with natural dyestuff from Siris bark are fairly good in light fastness and in washing fastness and good in rubbing fastness. By using natural dyestuff extracted from Siris bark, there are no stains on adjacent fabrics in the washing fastness test.
- From the fastness test results, all types of mordant give acceptable fastness properties and the colour developed on cotton materials by using different types of mordant give pale and attractive colour.
- Natural dyestuff extracted from Siris bark is a type of polygenetic dye since variable shades can be obtained by changing the mordant.
- It is also a type of adjective dye or mordant dye because it has affinity for mordanted materials.
- Natural dye powder extracted from Siris bark is simple to use in dyeing operation and it produces more attractive and beautiful colour on cotton materials compared to synthetic dyes.
- Since natural dyestuff is extracted as powder form, with regard to the environmental and health problems related to the use of synthetic dyes, it is sustainable and desirable for dye house to cut down the amount of toxic effluent resulting from synthetic dye process.
- The findings of present research work indicate that the bark of Siris *(AlbiziaLabbeck)* can be a good source of natural dye for textile industries.

5. RECOMMENDATIONS:

As for future study, an attempt should be carried out in order to evaluate the colour value, colour strength and reflectance of Siris-dyed cotton fabrics. It is suggested that the dyestuff extracted from Siris bark should be purified and characterized in order to obtain reproducible and standardized dye in the application process on textile materials.

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