

Title	Study on the Extraction and Utilization of Natural Dye from Noni Root
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Abstract

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Introduction

Dyes and textile are an important part of our everyday life. In the production of clothes, dyeing is a very important process. Natural dyes are comprised of dyes and pigments that are obtained from animals or vegetable matter without chemical processing. They impart colour when applied to substrate. This phenomenon is known as dyeing. Main sources of natural dyes are vegetable, animals and minerals. They can be obtained from any part of plants viz. leaves, fruits, seeds, flower, barks and root (Moeye, 1993).

Natural dyes are pleasing to the eye and the mind because natural dyes are pigments derived from nature. Moreover, uses of natural dyes in textile industry can reduce environmental pollution. Nowadays the use of synthetic dyes in textile industry is increasing rapidly. Since many of these products are resistant to biological degradation, this cause water pollution when released as effluent into the water sources (<http://www.nopr.niscair.res.in/bisteream>). Natural dyes of different origin can be extracted using aqueous method i.e. by using water for the extraction with or without addition of salt or acid or alkali or alcohol. They can be biodegradable and do not pose a problem of pollution for the waste (Dange, 1996).

Noni is the Tahitian name for the fruit of *Morinda citrifolia*, it has, many different names. The bark of the *morinda* produces a reddish purple and brown dye used in making batik and the tree was widely grown for this purpose in Java. In Hawaii, a yellowish dye was also extracted from the roots and also used to dye cloth ([http:// www.lifsource 4 lif.com/noni.htm](http://www.lifsource4lif.com/noni.htm)).

The background theory of this research work is to know the chemistry of dyes and interaction between mordant dyes and fibers; to get maximum yield of colourant and reproducible shades; to have commercial availability of extracted natural dyes. The objective of this research work was to produce the natural dye from from Noni root and to utilize it in the dyeing of cotton fabrics. And then, to evaluate the colour fastness properties of dyed cotton fabrics using washing and rubbing fastness tests.

Materials and Methods

Materials

In this research work, Noni root was collected from Taungoo Township, Bago Region. 95% ethanol, potassium dichromate, copper II sulphate, ferric chloride, potassium ferrocyanide, sulphuric acid, sodium chloride, sodium hydroxide and benedict's solution (commercial grade, British Drug House Co. Ltd., England) were used. Magnesium turnings, potash alum and vinegar (Myanmar Vinegar) were purchased from Theingyi Market, Pabedan Township, Yangon Region.

Methods

Physico-chemical Characterization of Noni Root

Noni root was washed with water to remove the adhering dirt and impurities. It was dried at room temperature and cut into small pieces. The physico-chemical characteristics such as moisture content, ash content and total solids content of Noni root were determined by AOAC method. pH of Noni root was determined by pH meter.

Phytochemical Investigation of Noni Root

The main compounds which are commonly found in Noni root are polyphenols, flavonoids, glycosides, phenolics, sugar and tannins. Phytochemical investigation of Noni root was performed to determine the presence or absence of phytochemical compounds according to the methods and procedures expressed in the Phytochemical Bulletin of Botanical Society of America (Harbone, 1984).

Preparation of Natural Dye from Noni Root

In the first extraction of natural dye, 100 g of chopped Noni root and 1000 mL of water were placed in a stainless steel pot and heated at 70°C for 30 min. The Noni root dye extract was filtered and collected in a glass bottle. The second, third and fourth extractions of natural dye were conducted using the same Noni root residue with 1000 mL of water for each extraction using the above procedure. Then, the first, second, third and fourth Noni root dye extract were mixed and concentrated at 90°C for 60 min in a water bath to obtain the concentrated natural dye. Extracted natural dye was weighed and stored in a freezer at 4°C for dyeing of cotton fabrics.

Physico-chemical Characterization of Extracted Natural Dye

The physico-chemical characteristics such as pH and total soluble solids content of extracted natural dye were determined by AOAC method. The absorbance values of extracted natural dye were determined from the wavelength 425 nm to 445 nm by using UV-Vis spectrophotometer (TRILP, TRSP-722). The highest absorbance value of extracted natural dye was obtained at wavelength 435 nm.

Determination of the Absorbance Value of Mordanted Natural Dye

(100) mL of extracted natural dye was mixed with (1) g of potash alum and heated at 60°C for (5) min. Then, it was cooled and the absorbance value was measured at the wavelength of 435nm by UV-Vis spectrophotometer. The above procedure was conducted using the different amounts (3) g and (5) g of potash alum and different mordants such as potassium dichromate and copper II sulphate ranging from (1) g to (5) g. The respective absorbance values were recorded.

Mercerizing of Cotton Fabrics

Firstly, (2) g of caustic soda was dissolved in 100 mL of distilled water before adding to the cotton fabrics. Cotton fabrics were simmered in caustic soda solution at 90°C for 10 min. The simmered cotton fabrics were washed with water to remove excess caustic soda. Any remaining alkali was neutralized by treating the cotton fabrics with vinegar solution (10 mL vinegar to 100 mL of water) for 30 minutes. To prepare cleaned cotton fabrics, the cotton fabrics were rinsed with water and then dried at room temperature. After that, the cotton fabrics were tested with a few drops of iodine. The cotton fabrics were cleaned if purple black colour did not appear (<http://www.dharmatrading.com/info/mercerization/html>).

Mordanting of Cotton Fabrics

In this method, mordant solution was prepared by heating 3 g of potash alum with 100 mL of water. The prepared mordant solution and 13 g of cotton fabric (10"x 10") was heated at 90°C for 10 minutes. During mordanting, the cotton fabric was frequently stirred to obtain good penetration of mordant into the cotton fabric. After that, mordant solution was removed and the mordanted cotton fabric was rinsed with water and then dried at room temperature. The same procedure was carried out with different mordants such as 3 g of potassium dichromate and 3 g of copper II sulphate to obtain the mordanted cotton fabrics.

Dyeing of Cotton Fabrics

For direct dyeing of cleaned cotton fabrics, (13) g of cleaned cotton fabric (10"x 10") and (100) mL of extracted natural dye were added to a stainless steel pot and simmered at 70°C for each of 15 min, 30 min and 45 min respectively. After dyeing, the cotton fabric was rinsed with water to remove unfixed dye and subjected to air drying. The amount of dye uptake on cotton fabric was determined and colour development on dyed cotton fabric was observed. In this research, pre-mordanting method, post-mordanting method and one-pot mordanting and dyeing method were used. In pre-mordanting method, mordanting was done before dyeing. In post-mordanting method, mordanting was done after dyeing. In one-pot mordanting and dyeing method, mordant, dye and cotton fabric were added in a pot and treated together.

Effect of Dyeing Time on the Amount of Dye Uptake on Cotton Fabrics

The effect of dyeing time on the amount of dye uptake on cotton fabrics was determined for different dyed cotton fabrics using different dyeing processes such as direct dyeing, pre-mordanting, post-mordanting and one-pot mordanting and dyeing methods. The amounts of dye uptake on cotton fabrics were determined as follows.

$$(\%) \text{ Dye uptake} = (A_0 - A_t) / A_0 \times 100$$

where, A_0 = absorbance value of natural dye before dyeing

A_t = absorbance value of natural dye after dyeing at T

(Khanchaiyapoom and Prachayawarakorn, 2008)

Testing the Colour Fastness Properties of Dyed Cotton Fabrics

Washing Fastness Properties

In this experiment, Launder Meter washing machine (Model No.L-4), containing 4-rack test bottle was used and the operation was carried out with Test No.3 of ISO 105 to assess the colour fastness to washing. Dyed cotton fabric was cut into (10 cm × 10 cm) and was sandwiched between two adjacent white bleached cotton fabrics having the same dimension with the test specimen. Four sides of the composite specimen were sewn at each corner by using a white thread. The required soap solution was prepared with (5) g of soap per liter.

After that, the soap solution was added to the jar and preheated at 60°C. Then, the composite sample was placed in the jar and tested at 60°C for 30 minutes. After washing, the specimen was rinsed with water for (5) times and dried at room temperature. The change in colour of the specimen was assessed by comparing with the original dyed cotton fabric and the rating is defined by using Grey scale for colour change and for staining.

Rubbing Fastness Properties

The rubbing fastness of dyed cotton fabric was determined by Rubbing Tester machine (JIS STDL-0241). The test specimen of (25 cm x 25 cm) and a piece of white bleached cotton fabric about 5 cm were cut parallel to warp direction for rubbing fastness test. In rubbing fastness test, the test specimen was mounted on the white bleached cotton fabric and placed on the rubbing tester. The rubbing distance was 100 mm. The test specimen was rubbed 100 times with (500) g load at the speed of 30 circles per minute. The white bleached cotton fabric and the test specimen were rubbed together on the tester by the aid of motor. Six different materials were tested simultaneously on the machine. In this study, both of dry and wet rubbing fastness tests were carried out. In the wet rubbing fastness test, the rubbed cotton fabric was wetted with water and the amount of water was two times of the fabric weight. The fabric was finally dried at room temperature (<http://en.wikipedia.org/wiki/Colour-fastness>).

Results and Discussion

The physico-chemical characteristics and phytochemicals investigation of Noni root were determined. The results in Table (1) indicate that 11.08 %w/w moisture content, 24.01 %w/w ash content, 88.92 %w/w total solids content and 6.2 pH in Noni root were observed. According to the results in Table (2), it was found that polyphenols, flavonoids, glycosides, sugar and tannins were present in Noni root. The results in Table (3) show that 5.5 pH, 1.5 °Brix total soluble solids and 1.150 absorbance value of extracted natural dye.

The absorbance values of extracted natural dye with different mordants and the different amounts of mordants (1,3,5) g were measured by UV-Vis spectrophotometer. From the results in Table (4), it was noted that mordanted natural dye solutions containing (3) g of each of mordant enhancing the highest absorbance value than the other amounts. The effect of dyeing time on the dye uptake and colour developed on dyed cotton fabrics were determined. According to the results in Tables (5) to (8) and Figures (3) to (5), it was found that post- mordanting and dyeing method with potassium dichromate gave 26.65 % of dye uptake on cotton fabrics at 70°C for 30 minutes. Therefore, (30) minutes of dyeing time is a suitable dyeing time which gave comparatively the highest amount of dye uptake on cotton fabrics than the other dyeing times.

From the results in Table (9), it can be seen that the dyed cotton fabrics using post- mordanting and dyeing method with potassium dichromate at 70°C for 30 min had slightly changed in shade, good staining on cotton fabrics and good to excellent (fastness rating – 4 to 5) of dry and wet rubbing fastness. Rubbing fastness results of dyed cotton fabrics indicated that all dyed cotton fabrics samples were found to be good in dry state and fair to good in wet state.



Figure (1) Noni Root



Figure (2) Extracted Natural Dye

Table (1) Physico-chemical Properties of Noni Root

Sr. No.	Properties	Experimental Value
1	Moisture content, %w/w	11.08
2	Ash content, %w/w	24.01
3	Total solids content, %w/w	88.92
4	pH	6.2

Table (2) Phytochemical Investigation of Noni Root

Sr. No.	Tests	Solvent	Reagents	Observation (colour)	Inference
1	Polyphenols	EtOH	1%FeCl ₃ + 1%K ₃ {Fe(CN) ₆ }	Greenish blue	+
2	Flavonoids	EtOH	H ₂ SO ₄ (conc:) + Mg	Pink	+
3	Glycosides	H ₂ O	10% FeCl ₃	Purple	+
4	Phenolics	H ₂ O	10% FeCl ₃	-	-
5	Sugar	H ₂ O	Benedict's Sol ⁿ	Red ppt	+
6	Tannins	H ₂ O	2% NaCl+1%FeCl ₃	Deep blue ppt	+

Note; (+) = Present, (-) = Absent

Table (3) Physico-chemical Properties of Extracted Natural Dye

Weight of chopped Noni root = 100 g
 Volume of water = 1000 mL × 4 times = 4000 mL
 Yield of natural dye = 1000 mL
 Extraction condition = 70°C, 30 min × 4 times

Sr. No.	Properties	Experimental Value
1	pH	5.5
2	Total soluble solids, °Brix	1.5
3	Absorbance value at 435nm	1.150

Table (4) Absorbance Values of Extracted Natural Dye with Different Mordants

Sr. No.	Volume of Natural Dye (mL)	Weight of Mordant (g)	Absorbance Values at 435 nm		
			Potash Alum	Potassium dichromate	Copper II Sulphate
1	100	1	1.497	1.783	1.152
2	100	3*	1.589	1.789	1.211
3	100	5	1.592	1.789	1.213

*Suitable amount of mordant

Table (5) Effect of Dyeing Time on the Amount of Dye on Cotton Fabric using Direct Dyeing Method

Size of mercerized cotton fabrics = 10"length x 10" wide

Sample No	Natural Dye (mL)	Dyeing Temp. (°C)	Dyeing Time (min)	Cotton Fabrics	
				Dye Uptake (%)	Colour
1	100	70	15	15.60	Pale Yellow
2	100	70	30*	16.12	Yellow
3	100	70	45	16.18	Yellow

*Suitable dyeing time

Table (6) Effect of Dyeing Time on the Amount of Dye on Cotton Fabrics using Pre-mordanting Method

Amount of mordant = 3 g

Size of mercerized cotton fabrics = 10"length x 10" wide

Sr. No.	Natural Dye (mL)	Dyeing		Dye on Cotton Fabrics, (%)		
		Temp. (°C)	Time (min)	Potash Alum	Potassium Dichromate	Copper II Sulphate
1	100	70	15	18.56	15.46	20.42
2	100	70	30*	20.25	19.63	21.68
3	100	70	45	20.31	19.68	21.71

*Suitable dyeing time

Table (7) Effect of Dyeing Time on the Amount of Dye on Cotton Fabrics using Post-mordanting Method

Amount of mordant = 3 g

Size of mercerized cotton fabrics = 10"length x 10" wide

Sr. No.	Natural Dye (mL)	Dyeing		Dye on Cotton Fabrics, (%)		
		Temp. (°C)	Time (min)	Potash Alum	Potassium Dichromate	Copper II Sulphate
1	100	70	15	25.32	22.78	21.81
2	100	70	30*	26.54	26.65	23.65
3	100	70	45	26.61	26.56	23.68

*Suitable dyeing time

Table (8) Effect of Dyeing Time on the Amount of Dye on Cotton Fabrics using One-pot Mordanting and Dyeing Method

Amount of mordant = 3 g

Size of mercerized cotton fabrics = 10"length x 10" wide

Sr. No.	Natural Dye (mL)	Dyeing		Dye on Cotton Fabrics, (%)		
		Temp. (°C)	Time (min)	Potash Alum	Potassium Dichromate	Copper II Sulphate
1	100	70	15	19.65	21.65	22.34
2	100	70	30*	20.57	23.58	23.45
3	100	70	45	20.63	23.61	23.49

*Suitable dyeing time

Table (9) Colour Fastness Properties of Dyed Cotton Fabrics using Various Methods and Mordants

Methods	Mordants	Rubbing Fastness		Washing Fastness	
		Dry Mark Code (1-5)	Wet Mark Code (1-5)	Change of Shade	Staining on Cotton Fabric
Pre-mordanting method	Potash alum	4	3-4	2	4
	Potassium dichromate*	4	3-4	2	4
	Copper II sulphate	4	3-4	2	4
Post-mordanting method**	Potash alum	4	3-4	2	4
	Potassium dichromate*	4-5	4	3-4	4-5
	Copper II sulphate	4-5	4	2-3	4
One-pot mordanting and dyeing method	Potash alum	4	3-4	2	4
	Potassium dichromate*	4-5	4	2-3	4
	Copper II sulphate	4	3-4	2	4

* Suitable mordant , ** Suitable dyeing method
 Note: 5 = excellent, 4 = good, 3 = fair, 2 = poor, 1 = very poor

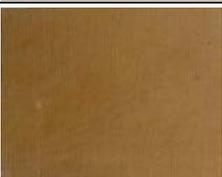
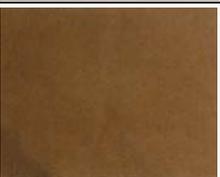
Dyeing Time (min)	Direct Dyeing Method	Pre-mordanting Method	Post-mordanting Method	One-pot Mordanting and Dyeing Method
15				
30				
45				

Figure (3) Colour Development on Dyed Cotton Fabrics using Potash Alum Mordant

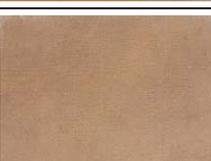
Dyeing Time (min)	Direct Dyeing Method	Pre-mordanting Method	Post-mordanting Method	One-pot Mordanting and Dyeing Method
15				
30				
45				

Figure (4) Colour Development on Dyed Cotton Fabrics using Potassium Dichromate Mordant

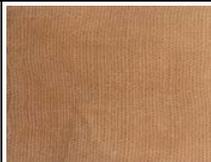
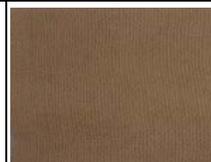
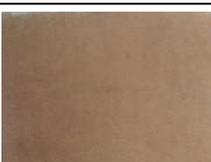
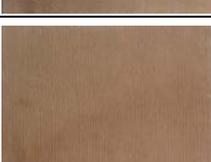
Dyeing Time (min)	Direct Dyeing Method	Pre-mordanting Method	Post-mordanting Method	One-pot Mordanting and Dyeing Method
15				
30				
45				

Figure (5) Colour Development on Dyed Cotton Fabrics using Copper II Sulphate Mordant

Conclusions

By simple boiling of chopped Noni root in water, the water soluble dye could be isolated and later used to dye the cotton fabrics. Natural dye extracted from chopped Noni root is a type of adjective dye or mordant dye because they have no direct affinity for unmordanted material and they have to be applied by the use of a mordant. Mordants or metal salts used as a fixing agent for dyeing affected the shade and colour fastness properties of the dyes. As the mordant is a metal salt compound, it can attract the dye molecules and form a complex structure with the dyes. As a result, the dyes are generally more strongly fixed on the cotton fabrics. Therefore, the dyed cotton fabrics using mordanting process have better fixation on the cotton fabrics. However, as the molecular structure of natural dye from Noni root is rather unstable, the washing process may cause a shade variation more or less depending on the types of mordant employed in the dyeing. The results generated in this research show a promising potential of the natural dye to be applied practically in the textile industry where the uniqueness of the products is exploited. For the extraction of natural dye from Noni root, the most suitable extraction conditions are chopped Noni root to water ratio 1:40, extraction temperature and time 70°C for 4 times of 30 min and concentration temperature 90°C for 60 min.

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