

## Study on Extraction and Application of Natural Dye from Neem Bark (*Melia azadirachta* L.)

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### Abstract

The present research work focuses on the extraction of natural dye from the stem bark of neem (*Melia azadirachta* L.) from Amarapura Township, Mandalay Region. The chemical compounds present in neem bark were investigated by phytochemical tests. Natural dye was extracted from the bark powder with pure water at 90°C for 60 min. 1.5 % w/v of dye solution was used for the dyeing of cotton fabrics. In this research, three dyeing methods such as pre-mordanting, post-mordanting, one pot dyeing and mordanting methods and also three different mordants such as alum, copper (II) sulphate and lime were used. The effects of dyeing methods and mordants on properties of dyed cotton fabrics were also investigated. Moreover, washing, rubbing and light fastness tests on dyed cotton fabrics were assessed by using standard Grey Scale. Washing and rubbing fastness of cotton fabrics dyed by post-mordanting method with alum gave the best result in comparing with other dyeing methods and mordants. Good light fastness was obtained by dyeing with alum and copper (II) sulphate mordants.

**Keywords:** Natural dye, mordant, cotton fabric, fastness test

### Introduction

Dyes can be defined as organic chemical compounds which have property of producing the phenomenon of colour by light absorption. These are intensely coloured materials that retained in substances by physical absorption, mechanical retention and by formation of covalent chemical bonds or complexes with salts or metals. Dyes are the colourant that has substantivity for a substrate, either inherent or induced by reactants (Anita Pal. 2017). Dyes are used for imparting colour to an infinite variety of materials like textiles, paper, wood, varnishes, leather, ink, fur, foodstuff, cosmetics, medicine, etc. Dyes are broadly categorized into two types namely; synthetic dyes obtained from chemical substance or derived through chemical process and natural dyes obtained from natural sources. The term Natural dyes covers all the dyes derived from natural sources such as plants, insects and minerals (Gupta, 1990).

Most natural dyes are of adjective types which need the use of chemicals called mordants to ascertain and promote the absorption, fixing and also to prevent bleaching and fading of the colours. The mordants are used to form chemical bonds between the dye molecules and the protein molecules in the fibers or in the textile. They can change the colour produced by the dye. But, indigo and a few other dyes do not penetrate the fibres and the dye molecules gradually become detached from the outer surface. Since ancient time, there has been many natural substances which can be used as mordants such as crab apple juice, tannins, urine, and wood ash. Chemicals such as salts of aluminium, chromium, copper, iron, and tin are also used as mordants which can be applied prior to dyeing, or after dyeing. Some are utilized simultaneously during dyeing. (Hancock,1997) 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 processing. They impart colour when applied to substrate. This phenomenon is known as dyeing. (Marjo Moeye, 1993).

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Nowadays, the use of synthetic dyes in textile industry is increasing rapidly. Since many of these products are resistant to biological degradation, this causes water pollution when released as effluent into the water sources. Natural dyes are biodegradable, unlike the synthetic dyes. They do not pose a problem of pollution for the waste. After the dyeing process, they are easily absorbed by the soil. *Azadirachta indica* (Neem) is a tree in the mahogany family Meliaceae. It is to be found throughout the dry zone of Myanmar. It is widely recognized as a forest crop with multiple uses including fuel wood timber production, oil tannins, pesticides, organic manures and medicinal products (Charles MC CANN). The objectives of this research work are to produce natural dyes from neem bark, to study the effect of dyeing methods and mordants on the properties of dyed cotton fabrics and to evaluate the fastness properties of dyed cotton fabrics.

## Materials and Methods

### Materials

Stem bark of neem (*Melia azadirachta L.*) samples were collected from Amarapura Township, Mandalay Region. Alum, copper (II) sulphate and lime were used in the dyeing process.

### Methods

#### Preparation of Raw Materials

Before extraction of natural dyestuff, the bark samples were rinsed with water and dried at room temperature. After cleaning and drying, the samples were powdered by grinding machine. These powder samples were screened by passing through mesh no-42 screen and stored for further works.



Figure1 *Melia Azadirachta L.* Bark (Neem Bark)

#### Phytochemical Investigation of Neem Bark

The preliminary phytochemical tests were carried out to study the main compounds which are present or absent in bark. Tests for alkaloids, polyphenol, flavonoids, glycosides, phenolics, saponins, lipophilics, and tannin were performed according to the methods and procedures prescribed in the Text Book of "A Guide to Modern Techniques of Plant Analysis" (Harbone, 1984).

## **Extraction of Natural Dye Solution**

About 3 g stem bark powder (- 42 mesh) and 400 ml water were boiled in a stainless steel vessel at 90°C for 1hr to obtain the dye solution of 200ml. This dye solution was cooled and then filtered with filter cloth. It was used for dyeing of cotton fabrics.

## **Dyeing Process on Cotton Fabrics**

### **Mercerization of Cotton Fabrics**

Firstly, 5 g of caustic soda were dissolved in 2 L of warm water before adding to the cotton fabrics. To prepare mercerized cotton fabrics, 1 yard of cotton fabrics was simmered in caustic soda solution for 60min and rinsed with water. After completion of this step, the cotton fabrics were put into vinegar solution (4ml acetic acid to 1L of water) for 30 min to neutralize it. To prepare clean 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. Purple black colour did not appear and the fabrics had been mercerized.

### **Pre- mordanting Method**

In this method, mordant solution was prepared by heating 1.25g alum with 200 ml water to obtain a volume of material to liquor ratio of 10:200. This solution was heated to a temperature of 80°C and 10g cleaned fabric was simmered in mordant solution for about 45 minutes. During mordanting , the fabric was frequently stirred to obtain good penetration of mordants into the materials. After that, the fabric was rinsed with water.

After mordanting, the dye liquid prepared was added to a beaker to obtain material to liquor ratio 10:200 and heated at 80°C. Then the mordanted fabric was simmered in this solution for about 30 minutes. Then the fabric was rinsed with water and allowed to air dry. The same experiment was carried out with different mordants such as copper (II) sulphate and lime.

### **Post - mordanting Method**

After dyeing, mordanting bath was prepared by heating 1.25 g alum with 200ml water to obtain material to liquor ratio of 10: 200. This solution was heated to 80°C and 10g cleaned fabric was simmered in it for 45 minutes. After that, similar experiments were carried out as pre-mordanting method. Different mordants such as copper (II) sulphate and lime were used for dyeing process.

### **One pot Dyeing and Mordanting Method**

The dye liquid was added to a beaker containing 1.25 g alum to make a volume of material to liquor ratio 10:200. Then, the dye bath was heated at 80°C and 10g cleaned fabric was simmered in that solution for about 30 minutes. After that, the fabric was rinsed with water and allowed to air drying. The same experiment was carried out with different mordants such as copper (II) sulphate and lime.

### **Testing the Colour Fastness of Dyed Fabrics**

After dyeing, testing for colour fastness of dyed fabrics was carried out. In the dyeing process, fastness of textile substrates to the effects such as light, washing, and rubbing were determined.

Launder Meter, Model L-4 (4-rack testing bottle) washing machine was used and Test No.3 of ISO 105 was used to assess the colour fastness to washing. 2g/litre of soap solution was added to the jar a material to liquor ratio of 1:50 and the soap solution was preheated at 60°C. Then, the composite sample was placed in the jar and treated at 60°C for 30 minutes.

After washing, the specimen was rinsed with water for (3) times and dried at room temperature.

For the test of the colour fastness to rubbing, a Crock Meter, JIS.L 0823 / 0849 with a rubbing finger, comprising a cylinder of 1.6 cm diameter moving to and fro along a straight line of 10 cm track on the specimen with a load of 900 g was used.

An outdoor exposure cabinet was used for light fastness test. The cabinet consists of a glass-covered enclosure of wood to protect the specimens from rain and weather. The dyed fabrics measuring (2 in × 1.5 in) of the material to be tested were cut out. These specimens were placed on the rack and the cabinet was covered. Then the specimens were exposed on sunny days between the hours of 9 a.m. and 3 p.m. for 7 days. The change in colour is assessed by comparing with the original dyed fabrics.

<b>Fastness Rating</b>	<b>Properties</b>	<b>Degree of Alteration in Shade and Strength</b>
5	Excellent	Negligible or no change
4	Good	Slightly change
3	Fair	Noticeable change
2	Poor	Considerably change
1	Very poor	Much change

Source: Lyle, Siegert (1977)

### **Results and Discussion**

In the extraction of natural dyes from neem bark, the phytochemical investigation of neem bark powder is tested and the results are shown in Table (1). According to the results, alkaloids, polyphenol, flavonoid, glycosides, phenolics, saponins, lipophilics, and tannins are present in neem bark. From the results of Table (2), cotton fabrics dyeing by post-mordanting method using alum are most suitable because it has a change of shade 4-5 (Good-Excellent).

Table (3) indicates the colour fastness results of dyed cotton fabrics mordanted with copper (II) sulphate. From the results, post-mordanting method is suitable because it has a change of shade 4 (Good) and staining on cotton fabrics of 4-5(Good-Excellent). In Table (4), colour fastness of dyed cotton fabrics dyeing by post-mordanting method with lime gives better result. This is due to the observation of change of shade mark in grade 4 (Good) and wet rubbing grade of 4 (Good).

According to the fastness results of dyed cotton fabrics, dyeing by post-mordanting method with alum is found to be most suitable due to change of shade mark in washing fastness. From the study of light fastness in Tables (2) to (4), the results show that using post-mordanting with alum and copper (II) sulphate is more suitable than lime. The result is confirmed by the fact that post-mordanting with a metal salt can result in an improvement in the light fastness of some natural dyes (Gulrajani,1992).

Table 1. Results of Phytochemical Investigation of Neem Bark

Sr. No.	Test	Reagent used	Observation	Inference
1	Alkaloids	(i) Dragendroff's reagent	(i) Orange ppt	+
		(ii) Mayer's reagent	(ii) Cream ppt	+
2	Polyphenols	1% FeCl <sub>3</sub> +1% K <sub>3</sub> [Fe(CN) <sub>6</sub> ]	Green-blue colour	+
3	Flavonoids	HCl (conc:) + Mg turnings	Pink Colour	+
4	Glycosides	10% FeCl <sub>3</sub>	Purple colour	+
5	Phenolics	10% FeCl <sub>3</sub>	Purple colour	+
6	Saponins	NaHCO <sub>3</sub>	Froth	+
7	Lipophilics	0.5 N KOH	Deep colour	+
8	Tannins	2% NaCl, 1% FeCl <sub>3</sub>	Deep blue ppt	+

Notes: + = Present

Table 2. Colour fastness of Dyed Cotton fabrics Mordanted with Alum

Cotton Fabric = 10 g (10" – 10") Dyeing temperature and time = 80°C,30min

Mordanting temperature and time = 80°C ,45 min.

Volume of mordant solution = 200 ml

Volume of 1.5 % w/v of dye solution = 200 ml

Sr. No.	Method	Alum %(w/v)	Washing Test (60°C , 30 min)		Rubbing Test 900g , 10 times		Light Test ( 7 days)
			Change of shade	Staining on Cotton	Dry	Wet	
1	Pre-mordanting	0.625	3-4	4	4-5	4	4
2	Post-mordanting*	0.625	4-5	4-5	4-5	3-4	4
3	One-pot dyeing and mordanting	0.625	3-4	4-5	4-5	3-4	4

Notes: \* Suitable method

Fastness rating 1 = Very poor, 2=Poor, 3=Fair, 4=Good, 5=Excellent (Lyle, 1977)

Fastness tests were conducted at Paleik Spinning, Weaving and Finishing Factory, Paleik Township, Mandalay Region.

Table 3. Colour fastness of Dyed Cotton fabrics Mordanted with Cu(II)SO<sub>4</sub>  
 Cotton Fabric = 10 g (10" – 10") Dyeing temperature and time = 80°C,30min  
 Mordanting temperature and time = 80°C ,45 min.  
 Volume of mordant solution = 200 ml  
 Volume of 1.5 % w/v of dye solution = 200 ml

Sr. No	Method	Cu(II)SO <sub>4</sub> % (w/v)	Washing Test (60°C , 30 min)		Rubbing Test 900g , 10 times		Light Test ( 7 days)
			Change of shade	Staining on Cotton	Dry	Wet	
1	Pre-mordanting	0.05	3-4	4-5	4-5	3-4	4
2	Post-mordanting*	0.05	4	4-5	4-5	3-4	4
3	One-pot dyeing and mordanting	0.05	4	4	4-5	3-4	4

Notes: \* Suitable method

Fastness rating 1 = Very poor, 2=Poor, 3=Fair, 4=Good, 5=Excellent (Lyle, 1977)

Fastness tests were conducted at Paleik Spinning, Weaving and Finishing Factory, Paleik Township, Mandalay Region.

Table 4. Colour fastness of Dyed Cotton fabrics Mordanted with Lime  
 Cotton Fabric = 10 g (10" – 10") Dyeing temperature and time = 80°C,30min  
 Mordanting temperature and time = 80°C ,45 min.  
 Volume of mordant solution = 200 ml  
 Volume of 1.5 % w/v of dye solution = 200 ml

Sr. No	Method	Lime % (w/v)	Washing Test (60°C , 30 min)		Rubbing Test 900g , 10 times		Light Test ( 7 days)
			Change of shade	Staining on Cotton	Dry	Wet	
1	Pre-mordanting	0.1	3-4	4-5	4	3-4	3
2	Post-mordanting*	0.1	4	4-5	4-5	4	3
3	One-pot dyeing and mordanting	0.1	3-4	4-5	4-5	3-4	3

Notes: \* Suitable method

Fastness rating 1 = Very poor, 2=Poor, 3=Fair, 4=Good, 5=Excellent (Lyle, 1977)

Fastness tests were conducted at Paleik Spinning, Weaving and Finishing Factory, Paleik Township, Mandalay Region.

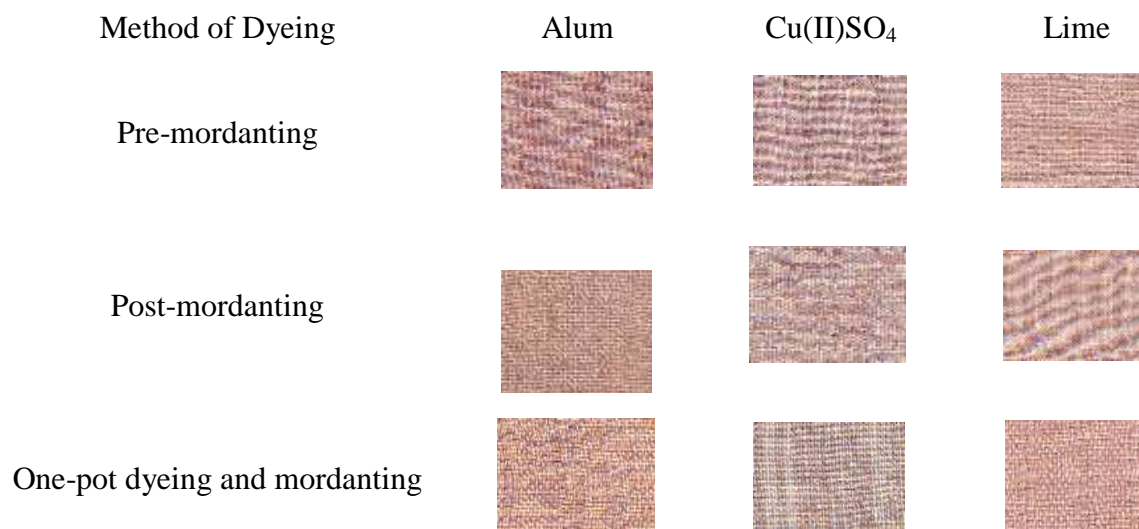


Figure 2 Colour Development of Cotton Dyed with Different Dyeing Methods and Mordants

### Conclusion

In the application of natural dye on cotton fabrics, it can be observed that mordanting with copper (II) sulphate in all dyeing methods showed attraction colours more than the other mordants. It can also be concluded that cotton fabrics dyeing by pre-mordanting method gave vibrant colour than the other dyeing methods. From the fastness results of dyed cotton fabrics, dyeing by post-mordanting method with alum was the most suitable condition for dyeing of cotton fabrics. This is because of the change of shade in washing fastness grade of 4-5 (Good-Excellent). Moreover, good light fastness was found in cotton fabrics dyed by using alum and copper (II) sulphate mordants.

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