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Investigating *Morinda citrifolia* wood as a sustainable dye source: a return to natural colours

Manasseh Moses E [1], S Lakshmana Prabu[2,a] & I Jaisankar[1]

[1]Natural Resource Management Division, ICAR–Central Island Agricultural Research Institute, Port Blair 744 101, India

[2]Department of Pharmaceutical Technology, University College of Engineering (BIT Campus), Anna University Tiruchirappalli 620 024, India

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This study aimed to examine the dye-fixing properties of cotton fabrics dyed with a dye extracted from *Morinda citrifolia* L. wood under various physicochemical conditions, including temperature changes, heating durations, and mordant treatment techniques, to achieve noticeable and stable dye fixation. For pre-, post-, and simultaneous mordanting procedures, iron salts such as alum, ferric chloride, stannous chloride, and ferrous sulphate were utilized. Dyeing with ferrous sulfate resulted in a higher dye uptake on cotton fibres (34.43%), according to the results. The colour fastness of noni wood dye is best achieved by using alum with a pre-mordanting technique.

Keywords: *Morinda citrifolia,* Mordants, Natural dyes, Noni, Wood dye

1 Introduction

Throughout history, people have used naturally occurring colours. Before synthetic dyes were invented, natural dyes were used to colour fabrics. People liked the colourants because they looked good, offered health benefits, and were environmentally friendly. They came from plants, minerals, and animals[1]. Synthetic dyes are very bad for your health and the environment because they are toxic and don't break down. Because of the problem, there is now a strong demand for natural, eco-friendly colours. Recent restrictions have made people more aware of environmental issues. It might be helpful for the modern textile industry to look into traditional dye plants[2]. Many people suggest that morinda is a type of plant that may produce natural dyes. The site's long history and extensive cultural heritage make it important. This genus of the Rubiaceae family has more than 200 different types[3]. The Plant List (2013) states that of the 131 plants recognized in 2013[4], 48 were placed in the same order. Indian mulberry, also known as Noni, is a plant that has been widely studied and used. This tough plant grows on the tropical islands of the southeast region and the Pacific. This plant does well in both tropical and coastal areas. People use it to colour textiles and make herbal medicine.

Ethnobotanical records show that *M. citrifolia* has been used in the past on the islands of Sumatra, Java, and Bali, as well as on other islands in Indonesia, because it can produce dyes. People have used the plant's roots and stems to make bright yellow and red colours, especially for cotton fabrics[5]. People really liked these natural colourants because they lasted a long time and could bind well with natural fibres. They also didn't need synthetic colouring agents or fixatives most of the time. Scientists today are more interested in the plant's medical uses than in its potential as a dye source, even though it has been used for a long time.

Interestingly, the wood of *M. citrifolia* hasn't been studied much for dye synthesis, even though the plant's fruits, leaves, and roots have been. This is a significant knowledge gap because many plant woods contain bioactive substances, such as tannins and flavonoids, that might help achieve stable, bright dyeing. The purpose of this research is to determine the dyeing potential of *M. citrifolia* wood, with a focus on cotton textiles.

2 Materials and Methods

2.1 Sample Collection

Noni (*M. citrifolia* L.) wood was collected in Mithakhari village, Ferrargunj Tehsil, South Andaman District, Andaman and Nicobar Islands. A twelve-year-old, fully grown tree was selected for this research. A height of 1.34 m was used to cut the tree

[a]Corresponding author.
E-mail: slaxmanvel@aubit.edu.in

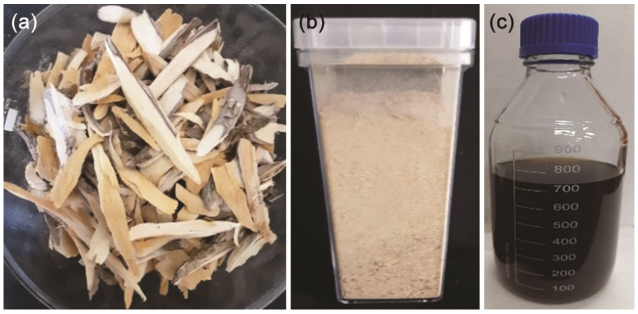


Fig. 1 — Different forms of Noni wood (a) chopped, (b) ground, and (c) dye extract

trunks. This study's specimen was collected in February 2024. Fresh wood was collected, chopped into small pieces, and then dried for 12 days at 45 °C in a hot-air oven. The dried wood sample was then ground into a fine powder for further studies.

2.2 Extraction Procedure

The wood extract was prepared by the aqueous method, and the different forms are shown in Fig. 1. For that, 40 g of wood sample was immersed in 1 L of distilled water and boiled at 60°C for 90 min in a heating mantle. Subsequently, the solution was filtered through the Whatman filter paper grade no 1. Direct heating of the post-dye solution for 3 h results in irreversible deterioration of the dye extract [6].The methanol extract was prepared by pulverizing the wood material and extracting it with methanol using the Soxhlet apparatus under reflux for 24 h at 60°C. The brown-coloured semisolid mass obtained was stored in a desiccator until ready for phytochemical analysis[7].

2.3 Phytochemical Analysis

The aqueous extract and methanol extract were used for testing for various phytochemicals, such as flavonoids (using ferric chloride and lead acetate tests), alkaloids (using Mayers and Hager's tests), tannins (using gelatin test), saponins (using Froth test), phenols (using ferric chloride test), glycosides (using Killer Killiani test), protein (using Biuret's test), amino acids (using Ninhydrin test), and Phyto steroids (using Salkowski's test)[8].

2.4 Optimization of Dyeing Conditions

To establish the optimal dyeing parameters for cotton using *M. citrifolia* L. wood dye, optimization studies were carried out with 20 g, 30 g, and 40 g at temperatures ranging from 40 °C to 60 °C. Likewise, different mordant concentrations ranging from 2 % to 4 % were considered. The UV-Vis spectrophotometer was used to measure the absorbance of the extracted natural dye over a wavelength range of 425-445 nm[9].

2.5 Selection and Pretreatment of Fabric

White Cotton fabric was chosen for the pretreatment. Cotton fabric was bought from a local textile shop in Bathubasthi village, South Andaman district, in Andaman & Nicobar Islands. About 10 × 10 cm^2 pieces were dipped in 1000 mLof hot water for 2 h with 10 mL of liquid soap to remove starch, dirt, and other chemical compounds from the fabric. Drained the soap solution and washed with cold water for 5 min. Then the cotton fabric was squeezed to remove the water and air-dried.

2.6 Tannic Acid Treatment

Subsequently, each piece of clean and air-dried cotton fabric was treated with 4 % on the Weight of Fabric (o.w.f) solution of tannic acid.

2.7 Mordant Techniques

Metallic mordants, viz., Alum, Ferric chloride, stannous chloride, and ferrous sulphate were used. Three different mordanting techniques, namely, pre-mordanting, simultaneous mordanting, and post-mordanting, were carried out[10, 11], in which the same set of cotton fabric is used for each technique.

2.7.1 Pre-Mordanting

Pre-mordanting was carried out on tannic acid-treated cotton fabric using 4% mordants of ferrous sulphate, alum, stannous chloride, and ferric chloride, individually. About 20g of respective mordants were dissolved in 500mL of distilled water. The tannic acid-pre-treated cotton fabric was immersed in a mordant solution, heated for 60 min at 60 °C, and then allowed to dry. The dried mordant-treated fabric is then mixed with 500 mL of *M. Citrifolia* wood dye extract for 1 h at 60°C. Then the same procedure was followed for different iron mordants on tannic acid-treated fabrics, and the fabrics were finally treated with noni wood dye. The fabrics were dried without washing[12,13].

2.7.2 Simultaneous-Mordanting

For each selected metallic mordant, 500 mL of *M. citrifolia* solution was used to dissolve 20 g of the mordant. The tannic acid-pretreated cotton dry fabric was then immersed in a mixture of dye and mordant for 60 min at 60 °C and allowed to dry[13].

2.7.3 Post-Mordanting

The Tannic acid-treated Cotton fabrics were immersed in 500mLof *M. citrifolia* wood extract, then heated to 60 °C for 60 min, and allowed to dry. The dried fabric was then treated with 4% mordant solution and heated at 60°C for 60 min. Finally, the fabrics were removed and allowed to dry[13].

2.7.4 No Mordant

The tannic acid-treated cotton fabric was immersed in 500ml of *M. citrifolia* wood extract without any mordants, heated up to 60 °C for 1 h, and made to dry without washing.

2.8 Dye Fixing

The mordant-treated fabrics were dipped in a 0.5 % sodium chloride solution for 60 min to fix the dye, then washed and dried in the shade. Sodium chloride can help to reduce this resistance and act as a Dyeing booster. It's also important to note that reactive dyes themselves carry a negative charge in both water and cellulose[14].

2.9 Dye Uptake

The percentage of dye uptake in cotton fabric was determined by measuring the difference in dye bath concentration before and after dyeing by using a UV-vis spectrophotometer. Various dyeing procedures, including direct Dyeing, pre-mordanting, simultaneous mordanting, and post-mordanting dyeing, were performed, and the dye uptake was calculated as the proportion of light reflected (R)[15].

$$\text{Dye uptake (\%)} = (A0 - AT) / A0 \times 100 \quad \ldots (1)$$

where A0 is the absorbance value of natural dye before dyeing; and AT, absorbance value of natural dye

2.10 Colour Strength Assessment

The Kubelka-Munk theory is a well-known method that can be used to determine the concentration of dye in wash-offs. Mordanting of the cotton fabric was performed using the Pre-mordanting, Simultaneous, and Post-mordanting methods. After dyeing, the cotton samples were left for 10 days before being washed. Each coloured sample was washed three times with a detergent solution containing 100 mL of water and 2 g of regular Surf Excel detergent powder. The wash-off from each sample was analyzed using a UV-visible spectrophotometer to determine the dye fastness level and the amount of dye released from the fabric. The conventional approach was used to calculate the proportion of light reflected (R) by the wash-off sample, as a function of the percentages absorbed (A) and transmitted (T), as shown below.

$$R = 100 - (A+T)$$

The following defines the relationship between the sample's spectral reflectance (R), light absorption (K), and scattering characteristics (S)[13].

$$\frac{K}{S} = \frac{(1-0.01R)^2}{0.02R} \quad \ldots (2)$$

2.11 Colour Variation

The shade observations were made using the Royal Horticultural Society (RHS) colour chart, which serves as the reference for recording plant colours. The sixth edition (2015; 2019 reprint) published by RHS media was used as a reference to identify different colours and variations between the dyeing treatments.

3 Results and Discussion

3.1 Phytochemical Analysis

Numerous beneficial chemicals were identified during the preliminary phytochemical screening of extracts from *M. Citrifolia* wood. The final yield of aqueous extract from Noni wood was 550 mL from the initial volume of 1 L. The phytochemical investigation of the methanol extract revealed the presence of diverse compounds, including steroids, saponins, glycosides, carbohydrates, flavonoids, and anthraquinones. Steroids, anthraquinones, saponins, and proteins were absent from the water dye extract. The phytochemical constituents were similar to those of the Noni root and fruit extracts[16,17], as shown in Table 1.

3.2 Optimization of Dyeing Conditions

To optimize the dyeing of cotton fabric with *M. citrifolia*, a dye-optimization study was conducted under different dyeing conditions. The study results demonstrated that 40 g/1000mL of the extract at 60°C

Table 1 — Phytochemical analysis of *M. citrifolia* wood dye

Phytochemical test	Aqueous	Methanol
Flavonoid	+	+
Alkaloids	+	+
Tannins	+	+
Phenols	-	+
Glycosides	+	+
Amino acids	+	+
Steroids	-	+
Carbohydrates	+	+
Saponins	-	+
Anthraquinones	-	+
Proteins	-	-

exhibited a maximum absorbance at 435 nm. The experimental conditions for the optimal dyeing by the extract are shown in Table 2.

3.3 Dye Uptake

The application of tannic acid can effectively enhance a cotton cloth's ability to absorb crucial mordants, resulting in clear, distinguishable colours[18]. The dye-uptake study was performed using different metallic mordants. The results demonstrated that pre-mordanting and dyeing with ferrous sulphate resulted in a higher (34.43%) dye uptake on cotton fibres. Cotton fabric treated with iron mordant produces the darkest shade, as the combination of dye and salt has a powerful effect on the fabric's colour. This observed result is supported by an analysis of the colour and fastness characteristics of wool fibres after application of the *Terminalia chebula* natural dye[19]. The order of dye uptake by different mordants was Ferrous sulphate > Ferrous chloride> Alum > Stannous chloride. The results of the dye uptake study are shown in Table 3. The Taguchi L9 orthogonal array was used for experimental optimization, and the results showed that neem wood extract effectively selected the appropriate dyeing conditions[20].

3.4 Assessment of Colour Strength (K/S) by Wash-offs

By accurately assessing colour strength, we can optimize the use of dyes and pigments, reducing waste and minimizing production costs. Efficient colour control ensures that only the necessary amount of colourant is used to achieve the desired colour intensity. The pre-mordanting with alum provides the highest colour fastness when applying noni wood dye. Mordant $FeCl_2$ has the worst colour fastness to washing. Also, it was observed that other metallic mordants, such as $FeSO_4$ and $SnCl_2$, performed better than ferric chloride. Simultaneous mordanting with alum produced the most durable colour with noni-wood dye. On the other hand, the colour fastness of ferric chloride was the weakest. Compared to ferric chloride, other metallic mordants, such as ferrous sulfate and stannous chloride, also perform better. Post-mordanting with stannous chloride produced the longest-lasting colour with noni-wood dye. Conversely, once again, ferric chloride exhibited the lowest colour fastness when subjected to washing. Other mordants, such as ferrous sulfate and stannous chloride, produced better results than ferric chlorideas shown in Fig. 2.

Evaluation of different mordants was conducted separately to compare their performance across various mordanting processes. The results demonstrated that the alum in the pre-mordanting technique has the lowest wash-off colour strength value and is considered the best among all the mordants. Conversely, using ferric chloride in the simultaneous mordanting process produced the poorest results among the mordants, with the highest colour wash-off strength, indicating that the

Table 2— Optimal proportion for dyeing of cotton fabric

Optimal proportions for Noni wood Dyeing of cotton fabric	Selected proportions
Dye material concentrations	40 g / 1000 ml
Dye extraction time	90 min
Dye heating point	60 °C
Post-filtrate heating temperature	60 °C
Post-filtrate heating duration	180 min
Dyeing time	60 min
Mordanting time	60 min
Mordanting heating time	60 min

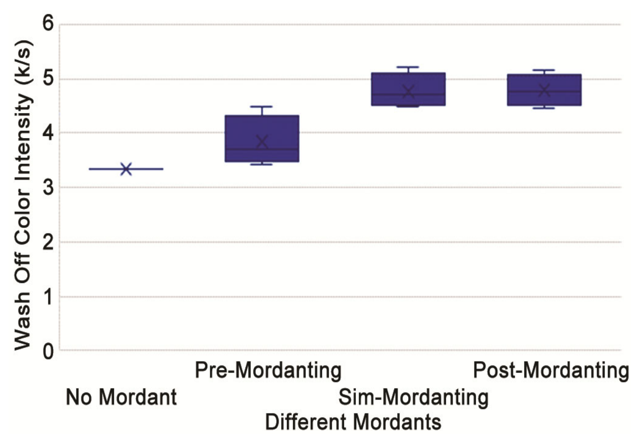


Fig. 2 — Comparison of different mordanting colour strengths by wash-offs

Table 3 — Dye uptake onto cotton by different mordanting methods

	Dye on Cotton Fabric %			
Methods	Ferrous sulphate	Alum	Ferric chloride	Stannous Chloride
Pre-mordant	34.43	22.87	33.92	16.02
Simultaneous Mordant	26.72	22.03	29.25	27.94
Post-Mordant	22.43	20.13	15.46	18.66
No Mordant	26.31			

M. citrifolia dye has the least colour fastness. On average, the pre-mordanting technique delivers the best results. The colour strength by different mordanting methods is shown in Table 4.

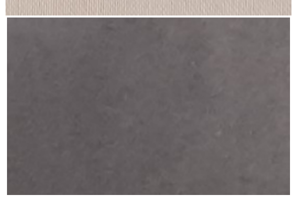
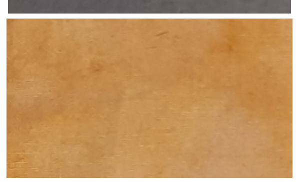
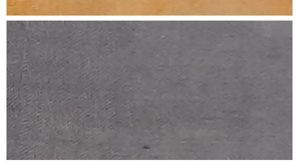
3.5 Colour Variations

Different colour variations were observed using pre-mordant, simultaneous, mordant and post-mordant methods. Significant changes of colour were observed in all the mordant methods with respect to different metallic mordants, except with alum, which possesses almost the lightest shade of colour in all the mordant methods. Alum-mordanted samples typically exhibited dark and light hues, as they prefer to form strong bonds with the dye rather than with the fibre due to weak coordination complexes; as a result, they block the dye and reduce its interaction with the fibre**.** On the other hand, the remaining iron mordants showed moderate to dark shades due to their strong coordination tendency and the formation of strong fibre–metal–dye complexes, and exhibited moderate and dark hues[21]. The observed different colour shades during the study are shown in Table 5.

Table 4 — Colour strength by different mordanting methods

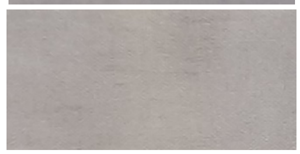
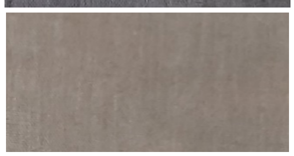
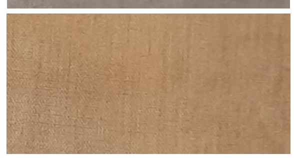
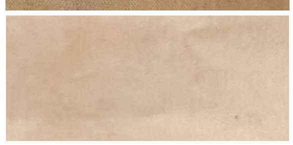
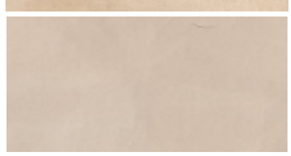
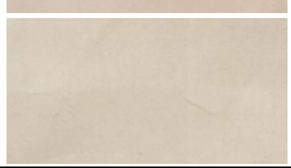
Mordant	Mordanting method	K/S
	No mordant	3.34
Ferrous sulfate	Pre- mordanting	3.62
	Sim- mordanting	4.65
	Post- mordanting	4.74
Alum	Pre- mordanting	3.42
	Sim- mordanting	4.48
	Post- mordanting	4.8
Ferric chloride	Pre- mordanting	4.48
	Sim- mordanting	5.2
	Post- mordanting	5.17
Stannous chloride	Pre- mordanting	3.81
	Sim- mordanting	4.77
	Post- mordanting	4.45

Table 5 — Colour variations as per RHS colour chart

Mordant	Mordanting method	Colour shades	Shade observation
	No mordant		Greyed-White Group 156 Yellowish grey (A)
Ferrous sulfate	Pre- mordanting		Greyed-Purple Group N186 Dark purplish grey (A)
	Sim- mordanting		Greyed-Orange Group 164 Moderate orange yellow (B)
	Post- mordanting		Black Group 202 Medium grey (B)

(*Contd.*)

Table 5 — Colour variations as per RHS colour chart (*Contd.*)

Mordant	Mordanting method	Colour shades	Shade observation
Alum	Pre- mordanting		Greyed-White Group 156 Yellowish grey (A)
	Sim- mordanting		Brown Group N200 Light brownish grey (C)
	Post- mordanting		Brown Group N200 Light brownish grey (C)
Ferric chloride	Pre- mordanting		Black Group 202 Medium grey (B)
	Sim- mordanting		Greyed-Green Group 197 Light olive grey (A)
	Post- mordanting		Greyed-Orange Group 164 Moderate orange yellow (B)
Stannous chloride	Pre- mordanting		Greyed-White Group 156 Yellowish grey (B)
	Sim- mordanting		Greyed-White Group 156 Yellowish grey (B)
	Post- mordanting		White Group 155 Pale yellow green (A)

4 Conclusion

Phytochemical analysis of the methanol extract of *M. citrifolia* wood reveals several bioactive compounds that could be useful in various pharmaceutical industries, particularly in coating and encapsulation processes for microparticles. Further, our investigation as a natural dye study results outlined that metallic mordants used in the pre-mordanting, simultaneous-mordanting, and post-mordanting techniques exhibited good Dyeing ability in cotton fabrics, and also showed satisfactory colour fastness to the fabric. The study found that pre-mordanting with alum produced the best colour fastness when using noni-wood dye on fabric.

Moreover, all the mordants performed better in pre-mordanting procedures than in simultaneous or post-mordanting methods. The effectiveness of blending different mordants, as demonstrated by some tested in this study, has been excellent across different scenarios. From this study, it is concluded that aqueous extract of *M. citrifolia* wood can be used as a natural dye for cotton fabric. Also, a wide range of dyes can be obtained by employing various mordants and mordanting processes. Further research with various mordants and combinations of these mordants is needed to improve their ability to bond with the dye, enhancing their overall mordanting capabilities.

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