PREPARATION OF NATURAL DYE POWDER EXTRACT FROM MANGO (NETTE) BARK AND APPLICATION ON COTTON FABRIC

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Abstract

This research concerns with the preparation of natural dye powder extract from *Mangifera indica* L. (Mango) bark. Natural dye solution was extracted from mango bark by using water, alkaline, acidic and alcoholic medium. The resultant concentrated dye solutions were dried in an oven at 60°C. Alcoholic medium was selected based on the yield percent and absorbance value and the dye powder was prepared at the various ethanol-water ratio, solid to liquid ratio, extraction temperature and time. The chemical compounds present in dye powder were studied by phytochemical methods and the functional groups were identified by FT-IR spectroscopy. Furthermore, content of moisture, ash and heavy metals in natural dye powder were also determined. The dye powder was then applied on cotton fabrics by using mordant with pre-mordanting, post-mordanting and simultaneous mordanting and dyeing methods. Fastness tests especially for washing, rubbing and light on dyed cotton fabrics were also studied.

Keywords: mango bark, extraction, natural dye powder, dyeing, mordant, fastness

Introduction

Natural dyes are basically elements of natural resources, and these dyes are generally classified as plant, animal, mineral, and microbial dyes based on their source of origin, colour, application and chemical nature. Natural dyes can reduce environmental pollution and they are renewable and biodegradable. The experience with natural dyeing has given an understanding of plants to find in the neighborhood. Finding textiles colors in plants that grow easily and fast has lead into a new colors that give unusual and interesting shades. These natural colors have luxuriance and luster that synthetics can never achieve. It has become a common exception that natural dyes only produce beiges and browns and colour ranging from yellow to black. In reality, vibrant, fast natural colors can be produced, which are comparable with and often surpass the colors of synthetics(Padma Shree Vankar, 2017).

Natural dyes are either substantive, meaning that which do not require a mordant, or adjective, they require a mordant. Adjective dyes are mordanted with a metallic salt or with the addition tannin tannic acid to the dye bath. Examples of such dyes are logwood, madder, cochineal, cutch and lac. In their refined state, adjective dyes are generally only slightly coloured and produce poor shades when used alone.

Mordants (from Latin mordere, 'to bite', because the mordant consume away the surface of the fibre so that the dye can seep in) are chemicals in the form of metallic salts which are generally used to make an affinity between the fibre and the dye.

The main objective of the mordant when used with adjective dyes is to open up the pores so that the dye can penetrate the fibres, thereby supporting in the fixation of the dyestuffs on the substrate. However, mordants can also be used with dyes which may be applied directly to the fiber. In this case their function is to form an insoluble compound with the dyestuff within th fibre itself, thereby improving the fastness properties of the dyed material (Patel, 2011).

The bark of the mango tree contains tannin (16-20%) and may be used for tanning purposes; it contains resinous matter. The bark yields a colouring matter which produces beautiful, through light, yellow shades on cotton, silk and wool; in conjuction with tumeric and lime, the bark dyes

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cotton a bright rose-pink. Mangiferine has been isolated from the bark (The Wealth of India,1985).

Thus, the objectives of this study are to prepare natural dye powder extract from mango bark, to determine the optimum conditions for the extraction of natural dye powder, to prepare dye powder with low cost and high yield, to study the colour developed on cotton fabric dyed with extracted dye powder.

Materials and Methods

Materials

Mango bark (Nette) was collected from selected old trees in Amarapura Township, Mandalay Region. For solvent extraction, 95% ethanol was used to extract natural dye solution from mango bark.

Methods

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

Preparation of Natural Dye Powder Extract from Mango (Nette) Bark Using Different Media

Natural dye was extracted as liquid form from mango bark powder (-42 mesh) by using different media such as pure water , 1% acetic acid, 50% ethanol and 0.5% sodium carbonate solution (Excutive summary,2010).

Mango bark powder 10 g was placed in a 3-neck round-bottomed flask. 100 ml of pure water was added into the flask (solid to liquid ratio 1:10) and the flask was kept for 5 hours. The flask was heated by a water bath at 60˚C. The extraction time was kept for 1 hour. Then the extracted dye solution was concentrated and dried at 60˚C for 6 hours in an oven.

Similar experiments were carried out using different media as described above. The absorbance value of dye powder extract was studied by UV/VIS spectrometer. Among the different media, alcoholic medium was selected as the most suitable medium due to its yield percent and absorbance value. Yield percent of natural dye powder extract was calculated based on weight of mango bark powder.

Determination of Optimum Conditions for Preparation of Natural Dye Powder Extract from Mango Bark

The optimum conditions for the preparation of natural dye powder extract was studied by varying the ethanol and water ratio in the range of (50:50, 40:60, 30:70, 20:80) and solid to liquid ratio (10:100 to 10:250) while the other variables like weight of bark, extraction temperature and extraction time as described were fixed.

The effect of temperature on the dye extraction was studied by varying the extraction temperature in the range of (60, 70, 80, 90˚C) and the effect of extraction time in the range of (1, 2 , 3 and 4) hours were also studied.

Phytochemical Investigation of Natural Dye Powder Extract

Phytochemical tests were carried out to study the main compounds which are present or absent in samples. Test for alkaloids, polyphenol, flavonoids, glycosides, phenolics, sugars, saponins, lipophilics, proteins and tannin were performed according to the procedures prescribed in the Text Book of "A Guide to Modern Techniques of Plant Analysis''.

Physico-chemical Characterization of Natural Dye Powder Extract from Mango Bark

The physico-chemical properties of natural dye powder extract from mango bark such as content of moisture, ash and heavy metals in natural dye powder were also determined. The heavy metal contents (As, Pb and Cd) were determined by using Atomic Absorption Spectrophotometer (AAS, Model No. AA-6200).

Dyeing Process of Cotton Fabrics

Mercerization

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 fabric was simmered in caustic soda solution for 60 min and rinsed with water. After the finishing of this step, cotton fabric was put into vinegar solution (4ml acetic acid in 1L of water) for 30 min to neutralize it. To prepare clean cotton fabric, neutralized fabric was rinsed with water and then dried at room temperature. After that, it was tested with a few drops of iodine. If purple black colour did not appear, the fabrics had been mercerized.

Mordanting

In this method, mordant solution was prepared by heating 0.05 g copper (II) sulphate with 200 ml water to obtain a volume of material to liquor ratio of 10:200. This mordant solution was heated at 80˚C and cleaned fabric (10" × 10") was simmered in this solution for about 45 minutes. During mordanting , the fabric was frequently stirred to obtain good penetration of mordants into the fabric. After that, the mordanted fabric was rinsed with water.

Dyeing

For dyeing of mercerized cotton fabric, 3 g of extracted natural dye powder and 200 ml of water was heated at 80°C and mercerized cotton fabric was simmered in this solution for 30 minutes. Then the fabric was rinsed with water and allowed to air drying. In this research, pre-mordanting method, post-mordanting method and simultaneous mordanting and dyeing method were used. In pre-mordanting method, mordanting was done before dyeing and in post-mordanting method, mordanting was done after dyeing. In simultaneous mordanting and dyeing method, mordant, dye powder and cotton fabric were added in a pot and treated together.

Testing the Colour Fastness of Dyed Fabrics

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

Washing Fastness

Launder Meter washing machine, Model L-4 (containing 4-rack testing bottle) washing machine was used and Test No.3 of ISO 105 was used to assess the colour fastness to

washing. 2 g/litre of soap solution was added to the jar (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.

Rubbing Fastness

The rubbing fastness of dyed fabric was determined by 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.

Light Fastness

The cabinet consists of a glass-covered enclosure of wood was used for light fastness test 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 9 a.m. to 3 p.m. for 7 days. The change in colour is assessed by comparing with the original dyed fabrics.

Results and Discussion

The effect of extraction medium on yield percent of natural dye powder extract was studied by using different polarity solvent (water, acetic, ethanol) and nonpolar solution (sodium carbonate). The results are shown in Table 1. Ethanol was chosen because alcohol group has high polarity than most non-polar but lower polar than water [9]. From the research, it was observed that alcoholic medium was suitable because it gave the maximum yield percent of dye powder and the results are shown in Table(1).

Table 1 Effect of Extraction Medium on the Yield Percent of Natural Dye Powder Extract using Different Media

Weight of mango bark powder = 10 g

Sr. No.	Extraction Medium*	Solid-Liquid Ratio	Extraction Condition		NDPE (g)	Yield (%)
			Temp (˚C)	Time (hr.)		
1	Water	1:10	60	1	1.4	14
2	1% Acetic acid	1:10	60	1	1.4	14
3	50% Ethanol**	1:10	60	1	2.0	20
4	0.5% Sodium carbonate solution	1:10	60	1	1.7	17

Note: NDPE = Natural dye powder extract, * (Excutive summary,2010) ** Suitable condition

According to the Beer-lambert law, absorbance value is directly proportational to concentration of dye solution and the alcoholic medium was selected because of the maximum absorbance value of 1.3. The results are shown in Table (2). Based on these results, acetic acid showed the lowest absorbance in most of stated concentration. The second lowest absorbance of overall result is water extract which has the slightest increment of absorbance compare with acetic acid. Ethanol is a polar solvent which can attract all kinds of active substances such as antioxidant, as well as good absorbance and low reactive toxicity [9].

Table 2 Absorbance Values of Natural Dye Powder Extract using Different Media

Sr.No.	Extraction Medium	Pigment*	Wavelength λ_{max} (nm)		Absorbance Values
			NDPE	Literature**	
1	Water	Mangiferin	260	258	0.6
2	1% Acetic acid	Mangiferin	259	258	0.5
3	50% Ethanol	Mangiferin	260	258	1.3
4	0.5% Sodium carbonate solution	Mangiferin	263	258	0.9

Note: NDPE = Natural dye powder extract

* and * * = Harbone, A Guide to Modern Technique of Plant Analysis.

The effect of ethanol- water ratio and solid to liquid ratio on the yield of dye powder are shown in Tables (3) and (4). According to the results in Table (3), it was clearly seen that ethanol:water ratio of 30:70 gave highest yield percent of dye powder.

Table 3 Effect of Ethanol-Water Ratio on the Yield Percent of Natural Dye Powder Extract

Weight of mango bark powder = 10 g
Soaking time = 5 hr.

Sr. No.	Solvent Ratio (ml)		Solid-Liquid Ratio	Extraction Condition		NDPE (g)	Yield (%w/w)
	Ethanol	Water		Temp. (˚C)	Time (hr.)		
1	50	50	1:10	60	1	2	20
2	40	60	1:10	60	1	1.7	17
3*	30	70	1:10	60	1	2.1	21
4	20	80	1:10	60	1	1.8	18

Note: NDPE = Natural dye powder extract * Suitable condition

The data in Table (4) indicates that yield percent of dye powder increases with increase in solid to liquid ratio from 10:100 to 10:200 but decreases at 10:250. From the results, solid to liquid ratio of 10:200 gives the maximum yield percent of dye powder.

Table 4 Effect of Solid to Liquid Ratio on the Yield Percent of Natural Dye Powder Extract

Weight of mango bark powder = 10 g , Soaking time = 5 hr.
Ethanol : Water = 30 : 70

Sr. No.	Solid to Liquid Ratio	Soaking Time (hr)	Extraction Condition		NDPE (g)	Yield (%w/w)
	MBP : Liquid (g) : (ml)		Temp. (˚C)	Time (hr.)		
1	10:100	5	60	1	2.1	21
2	10:150	5	60	1	2.5	25
3	10:200*	5	60	1	3.0	30
4	10:250	5	60	1	1.9	19

Note: MBP = Mango bark powder NDPE = Natural dye powder extract * Suitable condition

Table (5) tabulates the results of the effect of extraction temperature on the yield of dye powder. It was found that extraction temperature 80ºC was found to be the most favourable condition with respect to yield percent.

Table 5 Effect of Temperature on the Yield Percent of Natural Dye Powder Extract

Weight of mango bark powder = 10 g ,
Solid to liquid ratio = 10:200 , Ethanol and water ratio = 30:70

Sr. No.	Extraction Temp. (˚C)	Soaking Time (hr.)	Extraction Time (hr.)	NDPE (g)	Yield (%w/w)
1	60	5	1	2.2	30
2	70	5	1	2	29
3	80*	5	1	2.5	32
4	90	5	1	2.1	31

Note: NDPE = Natural dye powder extract *Suitable condition

According to the results of Table (6), maximum yield percent of dye powder is observed with 1 hour extraction time but after that decrease in yield of dye powder is observed.

Table 6 Effect of Extraction Time on the Yield Percent of Dye Powder Extract

Weight of mango bark powder = 10 g ,
Solid to liquid ratio = 10:200 , Ethanol and water ratio = 30:70

Sr. No.	**Extraction Time (hr.)**	**Soaking Time (hr.)**	**Extraction Temp. (˚C)**	**NDPE (g)**	**Yield (%w/w)**
1	1*	5	80	2.5	32
2	2	5	80	2	30
3	3	5	80	2.3	23
4	4	5	80	2.2	22

Note: NDPE = Natural dye powder extract *Suitable condition

From the results of Tables (3) to (6), the maximum yield percent of dye powder was obtained by using ethanol:water ratio of 30:70, solid to liquid ratio of 10:200 and extraction temperature and time were 80˚C and 1 hour.

From the results of phytochemical investigations shown in Table (7), it can be seen that extracted dye powder contains alkaloids, polyphenols, flavonoids, glycosides, phenolics, saponins, lipophilics and tannins.

Table 7 Phytochemical Characteristics of Natural Dye Powder Extract from Mango Bark

Sr. No.	Test	Extract	Reagents	Observation	Inference
1	Alkaloids	1% HCl	(i) Dragendroff's reagent (ii) Mayer's reagent	(i)Orange ppt (ii)Cream ppt	+ +
2	Polyphenols	EtOH	1%$FeCl_3$ + 1%$K_3[Fe(CN)_6]$	Green-blue colour	+
3	Flavonoids	EtOH Benzene	HCl (conc:) + Mg turnings 10% $FeCl_3$	Pink colour Greenish blue colour	+ +
4	Glycosides	H_2O	10% $FeCl_3$	Purple colour	+
5	Phenolics	H_2O	10% $FeCl_3$	Purple colour	+
6	Sugars	H_2O	Benedict's solution	Red ppt	_
7	Saponins	H_2O EtOH	$NaHCO_3$ H_2SO_4 (conc:)	Froth Red colour	+ +
8	Lipophilics	H_2O	0.5 N KOH	Deep colour	+
9	Proteins	H_2O	10% NaOH + 3% $CaSO_4$	Red	_
10	Tannin	H_2O	2% NaCl, 1% $FeCl_3$	Deep blue ppt	+

Note : + = Present - = Absent

Heavy metals are found everywhere in the environment and enters through human activities, mining, power generation, leaded gasoline. Humans risk to exposure from environmental concentrations that occur naturally or human activities. [10] The results in Table (8) indicate that 8.51% w/w moisture content, 7.99 % w/w ash content and heavy metals (0.09 ppm of As, 0.27 ppm of Pb, 0.13 ppm of Cd) were observed in natural dye powder extract. The standard permissible limits of heavy metals by WHO and FDA are 3 ppm of As, 1ppm of Pb and 1 ppm of Cd. From the results, the level of arsenic, lead and cadmium were found to be below the WHO and FDA maximum permissible limits.

Table 8 Physico-Chemical properties of Natural Dye Powder Extract from MangoBark

Sr. No.	Parameter	Contents	Literature*
1	Moisture (%w/w)	8.51	-
2	Ash (%w/w)	7.99	-
3	Arsenic (As) (ppm)	0.09	3
4	Lead (Pb) (ppm)	0.27	1
5	Cadmium (Cd) (ppm)	0.13	1

* (Lakshmi. T,2015)

Mango bark contain very high levels of phenolic compounds, mainly mangiferin and protocatechic acid, catechin, mangiferin, alanine, glycine, γ-amino-butyric acid, kinic acid, shikimic acid, etc were present. Figure (1) shows the FT-IR Spectra of natural dye powder extract. An occurrence of a strong band at 3342 cm^{-1} confirmed –OH stretching frequency of alcohol or phenol group. The C=C stretching vibrations at 1616 cm^{-1} confirmed the presence of aromatic or phenol groups. The band at 1452 cm^{-1} confirmed the C-H in-plane bending vibration. C-H in plane bending vibration of aromatic compounds were found at 1369 cm^{-1} and 1286 cm^{-1}. The band at 1200-1000 cm^{-1} was attributed to C-O stretching vibration. Another characteristics peak occurred at less than 1000 cm^{-1} were characteristics of C-H bending

vibrations. According to FT-IR analysis the colour compounds such as, polyphenols, flavonoids, phenolic, glycosides and tannin were present in dye powder.

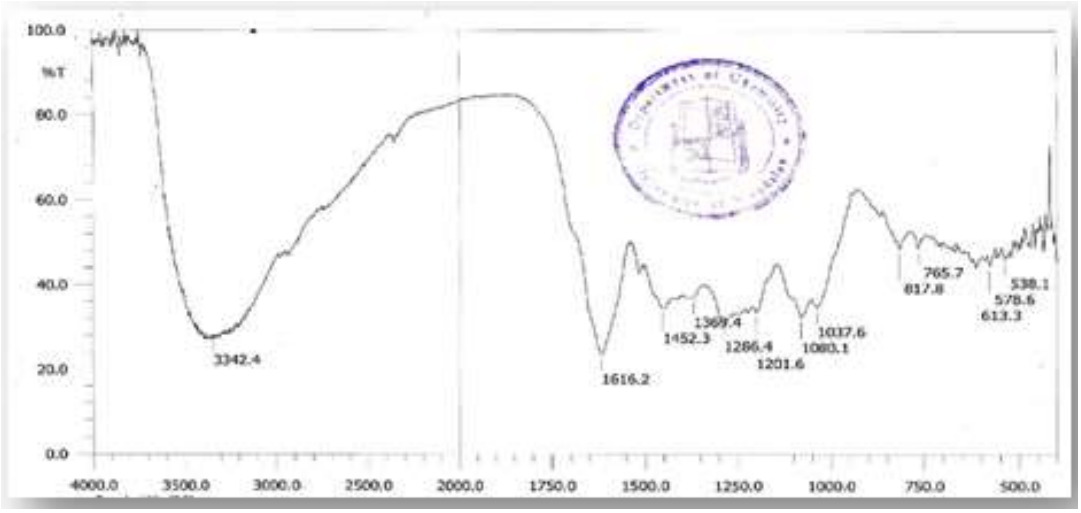

Figure 1 FT-IR Spectrum of Natural Dye Powder Extract

The data in Table (9) indicates that post-mordanting method with $CuSO_4$ mordant was found to be the most suitable condition due to its washing fastness grade of 4-5 (Good-Excellent). From the light fastness of dyed cotton fabrics, a slight change in colour was observed in different dyeing methods.

Table 9 Colour Fastness of Dyed Cotton fabrics Mordanted with $CuSO_4$

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	$CuSO_4$ % (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	2-3	4	4-5	3-4	4
2	Post-mordanting*	0.05	4-5	4-5	4-5	4	4
3	Simultaneous mordanting and dyeing	0.05	2-3	4	4-5	3-4	4

Notes : * Suitable method
Fastness rating 1 = Very poor, 2=Poor, 3=Fair, 4=Good, 5=Excellent (Lyle, 1977

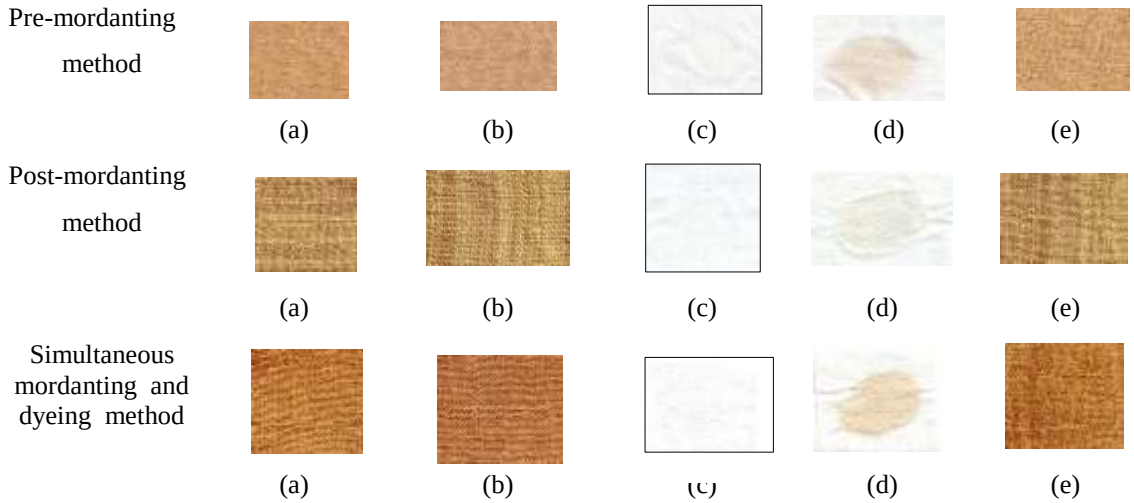

Figure 2 Color Fastness of Cotton Fabrics Dyeing with Natural Dye Powder Extract
(a) Original Sample (b)Washing Fastness (c) Dry Rubbing Test
(d) Wet Rubbing Test (e) Light Test

Conclusion

Natural dye powder was prepared from mango bark using water, alcoholic, acidic and alkaline method. Among the four medium alcoholic medium is the best because it gives the maximum yield percent and maximum absorbance value. For the preparation of natural dye powder from mango bark, the most suitable extraction conditions are mango bark powder to water ratio (1:20), ethanol-water ratio (3:7) and extraction temperature and time were 80˚C and 1 hour. According to the results obtained from phytochemical test and FT-IR analysis, it was found that the colour compounds such as alkaloids, polyphenols, flavonoids, glycosides, phenolics, saponins, lipophilics and tannins were present in dye powder. From the analysis of natural dye powder extract by AAS standard method, it can be seen that the presence of toxic heavy metals are below the WHO and FDA maximum permissible limits. It was evident that good fastness results were found in fabric dyed by post- mordanting method with copper (II) sulphate mordant due to its washing fastness grade of 4-5 (Good- Excellent). Moreover, variations in color shade were found when three mordanting methods were compared. As a result, good light fastness can be observed in all cotton fabrics dyed with all dyeing methods.

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