Purification of the Natural Dyestuff Extracted from Mango Bark for the Application on Protein Fibres

Zin Mar Win , Moe Moe Swe

***Abstract*—**An increased interest for natural dyes, started several years ago, on the part of the consumers, is just beginning to be felt. This paper concerns with the development of process for the purification of natural dyestuff extracted from an abundantly occurring plant namely Mangifera Indica Linn (Mango Tree), available almost everywhere in Myanmar. The objectives of this work are to produce the purified natural dyestuff extracted from mango bark, to study the colour of the dyed protein fibres (wool and silk yarns) by using various dyeing methods, and to study the fastness properties of the dyed sample materials. The compounds present in the mango bark and dyestuff are determined by Phytochemical tests. Optimization of dye purification condition is done by varying solvent types, solid to liquid ratio, time and temperature. Purified natural dye powder extract is then applied on protein fibres (wool and silk yarn) by using mordant with various dyeing conditions. Fastness tests on dyed samples for light, rubbing and washing fastness are carried out. The range of colour developed on dyed materials are evaluated in terms of (L*a*b*) CIELAB Coordinates and the dye absorption concentration on the textile materials is studied by using K/S values.

***Keywords*—**Purification, Natural Dyestuff, Mangifera Indica Linn, Protein Fibres

I. Introduction

A dye is a coloured substance which can be made to adhere to fabrics such as cotton, silk or linen. Natural dyes are obtained from flowers, trees, shrubs, berries, leaves, insects and minerals. These dyes have been used for centuries to produce colors for fabrics, yarns, leather, foods, etc. Natural dyes can give subtle and soft colours through to the brightest colour to the yarns and fabrics. Primitive dyeing techniques included sticking plants to fabric or rubbing crushed pigments into cloth. The methods became more sophisticated with time and techniques using natural dyes from crushed fruits, berries and other plants, which were boiled into the fabric, were developed. The natural dye extraction methods and dyeing methods described by traditional dyers are incomplete. Therefore, there is an absolute necessity to develop and standardize newer methods for the extraction and purification of natural dyes. The process of extraction, purification and drying of colours has been evolved to obtain the maximum colouring compounds in the final dye powder.

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II. Material and Methods

The bark of mango tree (Mangifera Indica Linn) is selected to extract the purified natural dyestuff. Mango trees are grown abundantly everywhere in Myanmar. Acetone, 95% alcohol and petroleum ether are used as the solvent to extract the colouring matter from mango bark. Various commonly used solvents and laboratory chemicals, laboratory equipments are used. Protein fibres are dyed with purified dyestuff and crude dyestuff to compare the colour of the dyed textile substrates. Chrome is used as the mordant. Launder-OMeter, Moderl L-4 (4-rack testing bottle) washing machine, JIS STDL-0241, GAKUSHIN type rubbing fastness tester and SDL 237A Light Fastness Tester are used for colour fastness tests of dyed materials. Spectrophotometer (Gretag Macbeth Colour-Eye 3100A) is used to measure the range of colour of the dyed textile materials by using L*, a*, b*, h*, C* and dye absorption concentration on the surface of Textile materials by using K/S values.

III. Preparation of Raw Materials

The raw materials of mango bark are cut from the trunk of mango tree and they are cleaned with water by brushing to remove the impurities. They are then dried at room temperature and ground into powder form in a grinder. Before commencing the extraction and purification of the natural dyestuff, the presence of major types of chemical compound in the mango bark is determined by using preliminary phytochemical tests.

IV. Optimization of the Conditions of Dye Purification

One hundred grams of mango bark powder are placed in a round bottomed flask and 500 ml of ethanol water (40:60) are added into it. The flask is heated in water-bath at 60°C for one hour. After extracting the dye, the extracted solution is filtered off and ethanol is recovered by using distillation method. After distillation, the concentrated dye solution is dried in the oven at 60°C. In this way, crude dyestuff is extracted as the powder form. Table I shows the conditions of crude dye extraction.

Since the purified dye yield depends on several parameters such as solvent types, solid to liquid ratio, time and temperature, the optimum conditions for purification of natural dyes are determined. Firstly, one hundred grams of mango bark powder reflux with the same procedure as the extraction of crude dyestuff. And then, this extracted solution is purified with different conditions. The optimum dye purification conditions are shown in Table II. After extracting the dye solution, it is filtered and the volume of the solution is reduced by distillation to get the ⅓ of the dye solution. And then this solution is kept overnight at room temperature for precipitation. The precipitate in the ethanol water solution is obtained with decantation. These active particles, purified dyes, are dried in the oven at 60°C.

TABLE I
CONDITIONS FOR EXTRACTION OF CRUDE DYESTUFF

Solvent		Solid to Liquid ratio	Temp. (°C)	Time (hr)	Yield %
Alcohol	Water				
40%	60%	1:5	60	1	29.4205

TABLE II
OPTIMUM CONDITIONS FOR PURIFICATION OF NATURAL DYESTUFF

Solvent	Temp. (°C)	Time	Solvent	Temp. (°C)	Time	Yield %
Ethanol water	60°C	1hr.	Ethanol water	Room Temp.	Over-night	18.33

V. Mordanting the Textile Materials

Mordanting is a process to fix the dyestuff to the textile materials. Before mordanting and dyeing, the scoured wool yarns are soaked in water for one hour and the silk yarns are firstly degummed. Mordanting conditions with chrome mordant are shown in Table III.

TABLE III
CONDITIONS FOR MORDANTING

Sr. no.	Type of Textile Substrate	Type of Mordant	Mordanting Conditions			
			Composition of Mordanting Bath	Temp. (°C)	Time (min)	M:L
1	Wool Yarn	Chrome	3% o.w.g	80	60	1:25
2	Silk Yarn	Chrome	3% o.w.g	80	30	1:25

VI. Dyeing the Textile Materials

In this study, the three dyeing methods namely, pre-mordanting, post-mordanting and simultaneous mordanting and dyeing are carried out. In pre-mordanting method, Method I, mordanting process is done before dyeing operation. In simultaneous mordanting and dyeing method, Method II, mordant, dye and fabric are added in a pot and treated together. In post-mordanting method, Method III, mordanting process is done after dyeing operation. The conditions for each dyeing method are shown in Table IV.

TABLE IV
CONDITIONS FOR DYEING

Type of Textile Substrate	Type of Mordant	Type of Dye	Dyeing Conditions				Sample Code.
			Dye Conc. (%)	Temp. (°C)	Time (min)	M:L	
Wool	Chrome	Pure	3	80	60	1:25	Wp_{11}
			7				Wp_{12}
			10				Wp_{13}
		Crude	3				Wc_{11}
			7				Wc_{12}
			10				Wc_{13}
Silk	Chrome	Pure	3	80	45	1:25	Sp_{11}
			7				Sp_{12}
			10				Sp_{13}
		Crude	3				Sc_{11}
			7				Sc_{12}
			10				Sc_{13}

VII. Measurement of Colour and Dye Absorption

Spectrophotometer (Gretag Macbeth Colour-Eye 3100A) is used to measure the range of colour obtained on textile materials by using L*, a*, b*, h*, C* and dye absorption concentration on the surface of Textile materials by using K/S values. They are examined under D65/10 light respectively. The results are shown in Table V and VI.

TABLE V
COLOUR DEVELPOED AND DYE ABSORPTION ON WOOL YARNS BY USIND CRUDE DYE AND PURIFIED DYE

Sample Code	Dye Conc. %	CIEL*a*b* System					K/S	Dyeing Method
		L*	a*	b*	C*	h*		
Wp_{11}	3	76.8	1.6	15.8	15.9	84.3	3.3	Method I
Wc_{11}		81.1	2.1	7.1	7.4	73.4	2.0	
Wp_{12}	7	82.1	1.8	6.7	6.9	75.2	2.0	
Wc_{12}		79.6	3.0	6.5	7.2	65.5	1.9	
Wp_{13}	10	81.7	1.9	6.5	6.8	73.8	2.0	
Wc_{13}		81.9	2.5	5.4	5.9	65.4	1.9	
Wp_{21}	3	81.0	0.7	16.6	16.6	87.5	2.5	Method II
Wc_{21}		80.0	2.4	5.6	6.1	67.2	1.8	
Wp_{22}	7	78.0	1.4	19.8	19.8	85.5	3.0	
Wc_{22}		73.8	2.1	14.6	14.8	81.7	3.2	
Wp_{23}	10	72.2	3.2	22.5	22.7	82.0	4.0	
Wc_{23}		81.1	0.8	10.6	10.6	85.7	2.0	
Wp_{31}	3	71.8	2.9	18.1	18.3	80.8	3.8	Method III
Wc_{31}		74.6	2.9	10.1	10.5	74.1	2.9	
Wp_{32}	7	73.5	2.6	12.5	12.8	78.3	2.9	
Wc_{32}		70.9	3.3	10.3	10.8	72.1	3.1	
Wp_{33}	10	73.6	2.8	12.6	13.0	77.3	3.1	
Wc_{33}		72.3	3.4	11.0	11.5	73.1	2.9	

TABLE VI
COLOUR DEVELPOED AND DYE ABSORPTION ON SILK YARNS BY USIND CRUDE DYE AND PURIFIED DYE

Sample Code	Dye Conc. %	CIEL*a*b* System					K/S	Dyeing Method
		L*	a*	b*	C*	h*		
Sp_{11}	3	78.8	-0.6	32.3	32.3	91.0	4.5	Method I
Sc_{11}		75.3	3.8	15.6	16.1	76.2	3.0	
Sp_{12}	7	82.8	-1.4	21.6	21.6	93.6	3.8	
Sc_{12}		74.0	2.3	25.7	25.8	85.0	4.0	
Sp_{13}	10	82.4	-0.4	18.6	18.6	91.1	2.3	
Sc_{13}		77.3	1.8	19.8	19.8	84.9	3.3	
Sp_{21}	3	81.5	-1.1	24.4	24.4	92.6	2.5	Method II
Sc_{21}		82.1	-0.8	17.7	17.7	92.6	1.9	
Sp_{22}	7	85.7	-2.4	19.8	19.9	97.0	2.3	
Sc_{22}		81.9	-1.0	20.9	20.9	92.7	2.5	
Sp_{23}	10	83.4	-2.4	23.9	24.0	95.7	3.1	
Sc_{23}		83.9	-1.5	16.8	16.8	94.9	1.8	
Sp_{31}	3	75.0	1.0	27.1	27.1	88.0	5.3	Method III
Sc_{31}		75.9	2.9	16.2	16.4	79.8	3.1	
Sp_{32}	7	83.6	0.6	12.6	12.6	87.1	1.5	
Sc_{32}		78.6	2.6	12.3	12.6	77.8	1.2	
Sp_{33}	10	79.8	1.8	13.8	14.0	82.6	2.1	
Sc_{33}		78.0	2.8	12.1	12.4	76.8	1.5	

VIII. Testing the Colour Fastness of Dyed Materials

The fastness to light, washing and rubbing are carried out to study the fastness properties of natural dyestuff on wool and silk materials. The results are shown in Table VII.

TABLE VII
COLOUR FASTNESS RESULTS OF DYED SAMPLES

Sr. No.	Sample Code	Rubbing Fastness		Washing Fastness			Light Fastness
		Dry	Wet	Change of Shade	Staining on		
					Cotton	Multi-fibre	
1	Wp	4-5	3-4	5	4-5	4-5	4
2	Wc	4	3	5	4-5	4-5	4
3	Sp	4-5	4-5	5	4-5	4-5	4
4	Sc	4-5	4	5	4-5	4-5	4

IX. Discussion

Phytochemical tests confirm the presence or absence of the following compounds in the crude dyestuff and purified dyestuff. They are glycoside, saponin, tannin, flavonoid, phenolic compound, α-amino acid, carbohydrate, alkaloid, steroid, reducing sugar and cyanogenetic glycoside. According to Phytochcemical tests, the crude dyestuff contains glycoside, saponin, tannin, flavonoid, phenolic compound, α-amino acid, carbohydrate, alkaloid, steroid and reducing sugar. Among these compounds, glycoside, tannin, flavonoid and phenolic compound are the substances which can give the colour. Tannins are the most important ingredients which are necessary for dyeing with natural dyes. Moreover, most of the colouring matters are also found in flavonoid and phenolic compounds. So, the compounds that do not give colour are removed to obtain purified dyestuff. When the crude dyestuff has been purified, it contains glycoside, tannin, flavonoid and phenolic compound. In this study, the crude and purified natural dyestuffs are applied on the wool and silk yarns to compare the colour and their fastness properties.

A. The Effect of Purified Dye and Crude Dye on the Colour Produced and Dye Absorption on Textile Materials

The colour developed on dyed samples are determined by using CIEL*a*b* system coordinates L*, a*, b*, C* and h* values. L* value refers to the lightness of colours ranging from zero for black, 50 for grey and 100 for white. The positive and negative values of a* refer to red and green colour components while the positive and negative values of b* refer to yellow and blue colour components respectively.

The highest values of a* and b* for very brilliant colour are approximately +80 or -80. For neutral point, the values of a* and b* are zero.

The values of chroma C* and the angle of hue h* are used to show the colour saturation and the range of colour, the angle of hue, h*, being measured starting from red to yellow, ie., hue angle 90 refers to yellow shade, hue angle 180 refers to green shade, hue angle 270 refers to blue shade, etc. When the value of chroma is increased, the colour will be more saturated.

B. Colour Developed and Dyed Absorption on Wool Yarn

Colour developed and dye absorption on dyed samples is evaluated in terms of their CIEL*a*b* coordinates and K/S values. Table V shows the values of L*, a*, b*, C*, h* and K/S.

The colour developed on wool using purified and crude dye lies in the Yellow-Red zone. But the values of b* (yellowness) are more than those of a* (redness) in the samples, indicating that the samples are more yellowish in the shade than red.

Moreover, the hue angles lie between 73.8 to 87.5 for purified dye and 65.4 to 85.7 for crude dye, so all of the samples are closer to yellow shade than red. The samples obtained from purified dye are more yellowish in comparison with the crude dye according to the value of b*.

The colour developed on wool yarns using purified dye lies in the range of 71.8 to 82.1 lightness, 6.5 to 22.5 yellow and 0.7 to 3.2 red whereas the colour of the dyed wool yarns using crude dye lies in the range of 70.9 to 81.9 lightness, 5.4 to 14.6 yellow and 0.8 to 3.4 red.

According to chroma values, the colour saturation by using purified dye is more in comparison with crude dye. The maximum colour saturation and dye absorption occur by using

simultaneous method with 10% purified dye concentration.

C. Colour Developed and Dyed Absorption on Silk Yarn

The results of colour value and dye absorption on dyed silk samples using purified dye and crude dye are shown in Table VI.

The colour developed on dyed sample obtained by using purified dye lies in the range of 75.0 to 85.7 lightness, 12.6 to 32.3 yellow with a little trace of 0.6 to 1.8 red and -0.4 to -2.4 green. The colour developed with purified dye by using post-mordanting method lie in the region between yellow and red while the colour produced by using pre-mordanting and simultaneous method appears in the region between yellow and green.

The colour developed on samples using crude dye lies in the Yellow, Red and Green region with 74.0 to 83.9 lightness, 12.1 to 25.7 yellow, 1.8 to 3.8 red and -0.8 to -1.45 green. All of the samples are closer to yellow because the values of b* are more than those of a*. The angles of hue also lie between 82.6 and 97.0 for purified dye and for crude dye, between 76.2 and 94.9.

The highest dye absorption in terms of K/S is obtained by using post-mordanting method with three percent purified dye concentration. There is a significant increase in dye absorption in case of purified dye. The colour saturation of the dyed samples using purified dye is also more in comparison with the crude dye. The maximum colour saturation occurs by using pre-mordanting method with three percent purified dye concentration.

D. Fastness Properties of Dyed Samples

Colour fastness results of dyed wool and silk yarns are shown in Table VI. As may be seen in the tables, good light fastness can be observed in all samples dyed with purified dye and crude dye extracted from mango bark, by using all dyeing methods at each dye concentration.

In case of dyed wool and silk yarns, it is evident that both purified dye and crude dye show exceptionally good fastness to washing with the rating of 5 in terms of change in colour. No staining on the adjacent undyed cotton and multifibre fabric is observed in washing, both in the case of dyed wool and silk yarns.

Good rubbing fastness can be observed in silk dyed with purified dye and crude dye. But, there is a slight improvement in wet rubbing test on dyeing with purified dye. In rubbing fastness for dyed wool samples, the wet rubbing fastness ratings show good in the case of purified dye whereas the wet rubbing fastness ratings show fair in the case of crude dye.

X. Conclusion

From the study of "Purification of the Natural Dyestuff Extracted from Mango Bark for the Application on Protein Fibres", the following conclusion can be drawn. According to the phytochemical test, the purified dyestuff extracted from mango bark contains colour compounds such as glycoside, tannin, flavonoid and phenolic compound.

In this study, yield percents of purified dyestuff is less than those of crude dyestuff since purified dye consists of only colour compounds. In the application of purified dyestuff and crude dyestuff extracted from mango bark, purified dyestuff gives better fastness results and more attractive colours than crude dyestuff. On the other hand, the cost of extracting purified dyestuff may be greater than that of crude dyestuff. In this study, the effects of dyeing properties on silk and wool fibres are emphasized rather than cost comparison between purified and crude dye.

A remarkable increase in colour value is occurred in the case of purified dyestuff. The colour yields of the purified dyestuff on all samples are better than those of the crude dyestuff. All dyed samples using purified dyestuff tend to get darker with the decrease in colour value of lightness. The purified dyestuff gives the higher colour value of dye absorption and more colour saturation than crude dyestuff.

Colour fastness properties of crude dyes extracted from mango bark are good in light fastness. The purified dyestuff gives better in light fastness than crude dyestuff because the colour of dyed yarns does not alter in the shade within 40 hours of testing time. In case of dyed wool and silk yarns, both purified dye and crude dye show exceptionally good fastness to washing. There are no stain on the adjacent undyed cotton and multifibre fabric in washing. By making reference to the results obtained, the purified dye improves the rubbing fastness remarkably.

Purified natural dyestuff extracted from mango bark gives subtle and soft colours to yarn. Since purified natural dyestuff is provided in powder form, it can be easily used on cottage-industry level and mass production level dyeing.

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