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Extraction of Natural Dye from Mangosteen Peel for Application on Dyeing of Cotton Fabric

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Abstract

The present research work focused on the extraction of natural dye from Mangosteen peel (*Garciniamangostana* Linn). The physico-chemical properties such as moisture content, ash content, total solids content and pH of Mangosteen peel were determined by Association of Official Analytical Chemists (AOAC) method. The chemical compounds present in Mangosteen peel were investigated by phytochemical tests. Natural dye was extracted from Mangosteen peel with pure water at 70°C for 60 minutes. The physico-chemical properties of extracted natural dye were also determined by AOAC method. An attempt was made on the dyeing of cotton fabrics using extracted natural dye with three different mordants such as zinc sulphate, potassium dichromate, potash alum and three different dyeing methods such as pre-mordanting, post-mordanting and simultaneous mordanting and dyeing method. The effect of dyeing time, different mordants and different dyeing methods on the properties of dyed cotton fabrics were also investigated. Moreover, washing and rubbing fastness properties, staining and colour change of dyed cotton fabrics were assessed by using standard Grey Scale.

Key words: Natural dye, mordant, cotton fabric, dyeing, Grey scale, fastness

Introduction

Natural dyes are derived from natural sources without chemical processing. 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. After the dyeing process, they are easily absorbed by the soil (Dange, 1996). In textile dyeing industry, most of the dyes employed are the synthetic azo dyes of which the azo bond cleavage brings about aromatic amines that may be harmful to human health and environment (Moeye, 1993). Mangosteen (*Garciniamangostana* Linn) is a

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tropical fruit in Guttiferae family. Mangosteen is dark purple to red-purple fruits. The edible fruit aril is white, soft, and juicy with a sweet, slightly acid taste and a pleasant aroma (www.elsevier.com/locate/foodchemtox). The major bioactive compounds found in Mangosteen are phenolic acid, prenylated xanthone derivatives, anthocyanins, and procyanidins. Ten phenolic acids were identified in Mangosteen fruit. The major anthocyanin in mangosteen was cyanidin-3-sophoroside (Chaovanalikit, *et al.*, 2012).

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 Mangosteen peel 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, Mangosteen peels were collected from Oak Tar Village, Taungoo Township, Bago Region. 95% ethanol, zinc sulphate, potassium dichromate, potash alum, ferric chloride, potassium ferrocyanide, 95% sulphuric acid, sodium chloride, sodium hydroxide and benedict's solution (commercial grade, British Drug House Co. Ltd., England) were used. Magnesium turnings and vinegar (Myanmar Vinegar) were purchased from Theingyi Market, Pabedan Township, Yangon Region.

Methods

Physico-chemical Characterization of Mangosteen Peels

Mangosteen peels were washed with water to remove the adhering dirt and impurities. They were dried at room temperature and weighed. The physico-chemical characteristics such as moisture content, ash content, total solids content and crude fibre content were determined by AOAC method. pH of Mangosteen peels was determined by pH meter.

Phytochemical Investigation of Mangosteen Peels

The main compounds which are commonly found in Mangosteen peels are polyphenol, flavonoids, glycosides, phenolic compound, sugar and tannins. Phytochemical investigation of Mangosteen peels 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 Mangosteen Peels

In the first extraction of natural dye, 100 g of Mangosteen peels and 1000 mL of water were placed in a stainless steel pot and heated at 70°C for 20 min. The Mangosteen peel extract was filtered and collected in a glass bottle. The second and third extractions of natural dye were conducted using the same Mangosteen peel residue with 1000 mL of water for each extraction using the above procedure. Then, the first, second and third Mangosteen peel extract were mixed and concentrated at 70°C for 70 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 at the wavelength of 420, 512, 520 and 700 nm by UV-Vis spectrophotometer (TRILP, TRSP-722). The colour density and anthocyanin content of extracted natural dye were calculated using the following equations.

$$\text{Colour density} = \{(A_{420\text{nm}} - A_{700\text{nm}}) + (A_{512\text{nm}} - A_{700\text{nm}})\} \times \text{dilution factor}$$

$$A = \text{Absorbance value}$$

$$\text{Anthocyanin content (mg/100g)} = \frac{\text{Absorbance} \times \text{Dilution factor} \times 100}{\text{Sample weight} \times 55.9}$$

(Chaovanalikit, *et al.*, 2012)

Determination of the Absorbance Value of Mordanted Natural Dye

(200) mL of extracted natural dye was mixed with (1) g of zinc sulphate and heated at 60°C for (5) min. Then, it was cooled and the absorbance value was measured at the wavelength of 520 nm by UV-Vis spectrophotometer. The above procedure was conducted using the different amounts (3) g and (5) g of zinc sulphate and different mordants such as potassium dichromate and potash alum 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 zinc sulphate with 100 ml of water. The prepared mordant solution and 13 g of cotton fabric (8"x 8") 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 potash alum 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 (200) mL of extracted natural dye were added to a stainless steel pot and simmered at 60°C for each of 10 min, 15 min and 20 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 simultaneous 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 simultaneous 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 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 simultaneous 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 Mangosteen peels were determined. The results in Table (1) indicate that 27.07 %w/w moisture content, 8.79 %w/w ash content 72.93 %w/w total solids content, 13.2134 %w/w crude fibre content and 5.15 pH in Mangosteen peels were observed. According to the results in Table (2), it was found that polyphenols, flavonoids, glycosides, phenolic compound, sugar and tannins were present in Mangosteen peels. The results in Table (3) show that 5.23 pH, 1.7 °Brix total soluble solids, 1.521 absorbance value, 41.8 colour density, 24.488 anthocyanin content were present in 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 simultaneous mordanting and dyeing method with potash alum give 13.44 % of dye uptake on cotton fabrics at 60°C for 15 minutes. Therefore, (15) 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 simultaneous mordanting and dyeing method with potash alum at 60°C for 15 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) Mangosteen Peels



Figure (2) Extracted Natural Dye

Table (1) Physico-chemical Properties of Mangosteen Peels

Sr. No.	Properties	Mangosteen Peel	
		Experimental Value	Literature Value*
1	Moisture content, %w/w	27.07	24.5- 66
2	Ash content, %w/w	8.79	-
3	Total solids content, %w/w	72.93	75.5 – 34
4	Crude fibre content, %w/w	13.2134	24.9- 29.1
5	pH	5.15	5-6

*(<http://www.cabdirect.org/abstracts/20093224593.html>)

Table (2) Phytochemical Investigation of Mangosteen Peels

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	Blue-black	+
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 mangosteen peel = 100 g
 Volume of water = 1000 mL × 3times = 3000 mL
 Yield of natural dye = 1000 mL / 998g
 Extraction condition = 70°C, 20 min × 3 times

Sr. No.	Properties	Experimental Value
1	pH	5.23
2	Total soluble solids, °Brix	1.70
3	Absorbance value at 520nm	1.521
4	Colouredensity	41.8
5	Anthocyanin content, (mg/ 100g)	24.488

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 520 nm		
			Zinc Sulphate	Potassium dichromate	Potash Alum
1	200	1	1.804	1.909	1.694
2	200	3*	1.875	1.924	1.898
3	200	5	1.878	1.925	1.898

*Suitable amount of mordant

Table (5) Effect of Dyeing Time on the Amount of Dyed 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	200	60	10	6.613	Yellowish orange
2	200	60	15*	7.429	Yellowish orange
3	200	60	20	7.101	Yellowish orange

*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, (mg/g)		
		Temp. (°C)	Time (min)	Zinc Sulphate	Potassium Dichromate	Potash Alum
1	200	60	10	8.43	8.32	8.35
2	200	60	15*	8.80	8.89	8.95
3	200	60	20	8.77	8.89	8.90

*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, (mg/g)		
		Temp. (°C)	Time (min)	Zinc Sulphate	Potassium Dichromate	Potash Alum
1	200	60	10	8.43	8.32	8.35
2	200	60	15*	8.80	8.89	8.95
3	200	60	20	8.77	8.89	8.90

*Suitable dyeing time

Table (8) Effect of Dyeing Time on the Amount of Dye on Cotton Fabrics using Simultaneous 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, (mg/g)		
		Temp. (°C)	Time (min)	Zinc Sulphate	Potassium Dichromate	Potash Alum
1	200	60	10	8.43	8.32	8.35
2	200	60	15*	8.80	8.89	8.95
3	200	60	20	8.77	8.89	8.90

*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	Zinc sulphate	3	3-4	2	3-4
	Potassium dichromate	4	3-4	2-3	4
	Potash alum *	4	4	3	3-4
Post-mordanting method	Zinc sulphate	4	3-4	2-3	3-4
	Potassium dichromate	4	3-4	3	4
	Potash alum *	4	3-4	3-4	4
Simultaneous mordanting and dyeing method**	Zinc sulphate	4	3-4	3	4
	Potassium dichromate	4	3-4	3-4	4
	Potash alum *	4	4-5	4	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	Simultaneous Mordanting and Dyeing Method
10				
15				
20				

Figure (3) Colour Development on Dyed Cotton Fabrics using Zinc Sulphate Mordant

Dyeing Time (min)	Direct Dyeing Method	Pre-mordanting Method	Post-mordanting Method	Simultaneous Mordanting and Dyeing Method
10				
15				
20				

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

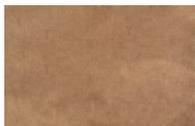
Dyeing Time (min)	Direct Dyeing Method	Pre-mordanting Method	Post-mordanting Method	Simultaneous Mordanting and Dyeing Method
10				
15				
20				

Figure (5) Colour Development on Dyed Cotton Fabrics using Potash Alum Mordant

Conclusions

By simple boiling of Mangosteen peels in water, the water soluble dye could be isolated and later used to dye the cotton fabrics. Natural dye extracted from Mangosteen peels are 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 were used as a fixing agent for dyeing of cotton fabrics and affected the shade and colour fastness properties of the dyes. On comparing with the unmordanted dyed cotton fabrics, the cotton fabrics dyed with simultaneous mordanting and dyeing method for natural dye exhibited attractive colours and acceptable fastness on cotton fabric especially with potash alum. 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 Mangosteen peels, the most suitable extraction conditions are Mangosteen peel to water ratio 1:30, extraction temperature and time 70°C for 3 times of 20 min and concentration temperature 70°C for 70 min.

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