Utilization of Extracted Natural Dye from Marigold Flowers in Cotton dyeing

Khin Thet Thet Htwe[1], Kalyar Min Min Htaik[2], Thin Thin Hlaing[3]

ABSTRACT

This research work focused on the extraction of natural dye from marigold flowers with water at 70 °C. The yield percent of extracted natural dye from marigold flower is 28.8 %. The some properties of natural dye were characterized by UV-Vis Spectrophotometer and pH meter. The colour intensity of extracted natural dye was investigated by Tintometer. The extracted natural dye was dyed on cotton fabrics by using various mordants (alum, $CuSO_4$, $FeSO_4$ and $K_2Cr_2O_7$) and different dyeing methods such as pre-mordanting, post-mordanting and simultaneous-mordanting. The effect of dyeing time on dye uptake was determined for direct dyeing method. According this result, (27 %) of dye uptake on cotton fabric for 60 min is suitable than the others. These dyeing methods were used at dyeing temperature (70 °C) and dyeing time 60 min. The colour fastness properties, such as, light fastness, rubbing fastness and washing fastness of all dyed cotton fabrics were investigated by using standard grey scale.

Keywords: Marigold flower, Natural Dye, Dyeing method, Colour Fastness Properties

Introduction

Marigolds have dark green leaves that are deeply divided and fragrant. Their flowers are in the yellow, gold, bronze and orange hue palate, with sizes that vary according to their category (Samuels, 2017). Marigold flowers contain various chemical constituents such as thiophenes, flavonoids, carotenoids and triterpenoids. Carotenoids are one of the major constituents and the main pigment of marigold flower (Gauri and Siddhraj, 2015).

Natural dyes

Natural dyes are known for their use in colouring of food substrate, leather as well as natural protein fibers like wool, silk and cotton as major areas of application since pre-historic times. The use of non-allergic, non-toxic and eco-friendly natural dyes on textiles has become a matter of significant importance due to the increased environmental awareness in order to avoid some hazardous synthetic dyes (Ashis and Priti, 2009).

Natural dyes, obtained from different sources such as plants, animals, and minerals, are renewable and sustainable bio-resource products with minimum environmental impact and known since antiquity for their use, not only in colouration of textiles but also as food ingredients and cosmetics (Chandan *et al.*, 2015).

Mordant

A mordant or dye fixative is a substance used to set (i.e. bind) dyes on fabrics by forming a coordination complex with the dye, which then attaches to the fabric (or tissue). It may be used for dyeing fabrics or for intensifying stains in cell or tissue preparations. As applied to textiles, mordants are mainly of historical interest because the use of mordant dyes was largely displaced by directs. The natural dyes having limited substantively for the fiber, require use of the mordant which enhances the fixation of the natural colourant on the fiber by the formation of the complex with the dye. Although these metal mordants contribute to developing wide gamut of hues after complexing with the natural colouring compounds, most of these metals are

toxic in nature and only in trace quantity their presence is found to be safe for the wearer.

Methods of mordanting

Mordants and dyes may be applied in three ways. They are as follows:

• Pre-mordanting, where the mordant is applied first, followed by dyeing.

• Post- mordanting, where the dyeing is done first and then mordanting is carried out.

• Simultaneous mordanting, where mordant and dye are mixed together and applied.

Colour Fastness Properties

The term colour fastness describes the resistance of dyed or printed textiles to various agencies to which they may be exposed during use or manufactures. Ideally the dyestuffs on a printed fabric or on a dyeing should last as long as the fabric itself. When fabrics have been dyed they must be tested to ensure the colour is fast to washing, lighting and rubbing. Dress fabrics, while not requiring such high light fastness, must have good washing and perspiration fastness. Cotton articles are usually required to stand up to much severe washing, so the washing fastness of dyes printed or dyed on cotton is usually important (Clarke,1977).

Materials and Methods

Extraction of Dyes

100 mL of distilled water and 4 g of dried marigold flowers samples were placed in conical flask and heated at 70 °C for 1 hr with 130 rpm. The extracted solution was filtered and collected in beaker. The second and third extractions of natural dyes were conducted using the marigold flowers residue with above procedure. Then, all extracted dye solutions were mixed and stored in a freezer at 4 °C for dyeing of cotton fabrics.

Characterization of Extracted Natural Dyes

(a)Determination of colour intensity

The colour intensity of extracted natural dyes was determined by Tintometer at Livestock and Irrigation Small Scale Industry Department, Ministry of Agriculture. The result is shown in Table 1.

(b)Determination of pH

The pH of the extracted natural dyes was determined by pH meter. The result is shown in Table 2.

(c)Determination of maximum wavelength

The absorbance of the extracted natural dye was measured at the wavelength range 400 – 680 nm using UV- Vis Spectrophotometer. The maximum wavelength was obtained when the absorbance reached the maximum value. The result is shown in Table 2.

Dyeing on the Cotton Fabrics

(a) Mercerizing of Cotton Fabrics

20 g of sodium hydroxide was dissolved in 1000 mL of distilled water. The cotton fabrics were dipped in this sodium hydroxide solution at 100 °C for 10

min. The dipped cotton fabrics were washed with water to remove excess sodium hydroxide. The remaining alkali was neutralized by treating with vinegar solution. These cotton fabrics were washed with water again and then dried. After that, the cotton fabrics were tested with a few drops of iodine solution. The cotton fabrics were cleaned if purple black colour did not appear.

(b) Direct dyeing method

The mercerized cotton fabrics were dipped in extracted natural dye solution, material to liquid ratio is (1:20), at 70 °C for 30 min, 60 min and 90 min respectively. These cotton fabrics were rinsed with water to remove unfixed dye and air dried.

(c) Pre-mordanting dyeing method

The mercerized cotton fabrics were simmered in various mordant solution (from section 2.5 A, B, C and D), material to liquid ratio is (1:20), at 70 °C for 1 hr. The mordanted cotton fabrics were dipped in extracted natural dye solution material to liquid ratio is (1:20), at 70 °C for 30 min, 60 min and 90 min respectively. These cotton fabrics were rinsed with water to remove unfixed dye and air dried.

(d) Post-mordanting dyeing method

The mercerized cotton fabrics were dipped in extracted natural dye solution, material to liquid ratio is (1:20), at 70 °C for 30 min, 60 min and 90 min respectively. The dyed cotton fabrics were simmered in various mordant solution (from section 2.5 A, B, C and D), material to liquid ratio is (1:20), at 70 °C for 1 hr. These cotton fabrics were rinsed with water to remove unfixed dye and air dried.

(e) Simultaneous- mordanting dyeing method

1.8 g of various mordants, such as alum, copper II sulfate, ferrous sulfate and potassium dichromate, were added to 180 mL of extracted natural dye solution and heated at 70 °C. The mercerized cotton fabrics were dipped in dye- mordant solution, material to liquid ratio is (1:20), at 70 °C for 30 min, 60 min and 90 min respectively. These cotton fabrics were rinsed with water to remove unfixed dye and air dried.

Determination of Dye Uptake (%) on Cotton Fabrics

The absorbance of both extracted natural dye solution before dyeing and after dyeing was determined at 430 nm by using UV-Vis Spectrophotometer. The amount of dye uptake on the cotton fabric was calculated as following equation. The result is shown in Table 3.

$$(\%) \text{ Dye uptake} = [(A_0 - A_t) / A_0] \times 100$$

Where, A_0 = absorbance value of natural dye before dyeing

A_t = absorbance value of natural dye after dyeing

Investigation for Fastness Properties of Dyeing Cotton Fabrics

The colour fastness properties of fabric show the resistance of dyes and printed on the fabric. So, the quality of dyeing cotton was investigated by light fastness, washing fastness and rubbing fastness. These tests were investigated at Development Centre for Textile Technology (DCTT) laboratory. The results are shown in Table 4.

Results and Discussion

The colour intensity was determined by Tintometer. From this result, the extracted dye given was 2.0 yellow, 2.2 red and 0.1 blue. The results are shown in Table 1.

The highest total solid content was obtained at the 70 °C. The observed colour, pH, and total solid content are shown in Table 2.

The maximum wavelength of extracted natural dye was determined by UV – Vis spectrophotometer. The absorbance and transmittance value of extracted natural dye of different wavelengths are shown in Table 3. The maximum absorbance (3.00) was obtained at 430 nm.

Table 1 Colour Intensity of Extracted Natural Dyes

Sr.No	Experiment	Present Colour Results
1	Colour	2.0 yellow 2.2 Red 0.1 blue

Table 2 Physical Properties of Extracted Natural Dyes

Sr. No	Properties	Observed Value
1	pH	6.05
2	Total Solid Content	28.8 %
3	Maximum wavelength	430 nm
4	Colour	Yellowish brown

Table 3 Absorbance and Transmittance Value of Extracted Natural Dyes with Different Wavelengths

Sr. No	Wavelength (nm)	Absorbance	Transmittance (%)
1	280	1.07	8.5
2	300	1.48	3.5
3	330	2.1	0.6
4	380	2.54	0.3
5	400	2.82	0.1
6	430	3.00	0
7	480	1.92	1.2
8	500	0.72	18.9
9	520	0.42	37.7

10	540	0.29	51.2
11	630	0.19	63.4
12	680	0.17	66.8
13	700	0.12	70.1

Dye Uptake on Cotton Fabrics

The effect of dyeing time on dye uptake was determined for direct dyeing method. The results of dye uptake for dyed cotton fabrics with various dyeing time are shown in Table 4. According to this result, (27 %) of dye uptake on cotton fabrics for 60 min is suitable than the others.

Table 4 Dye Uptake (%) of Dyed Cotton Fabrics using Direct Dyeing Method

Sr. No	Dyeing time (min)	Absorbance at 430 nm		Dye Uptake (%)
		Before Dyeing	After Dyeing	
1	30	3.00	2.35	22
2	60	3.00	2.18	27
3	90	3.00	2.2	27

Size of cotton fabric = 12" x 10" Material to liquid ratio = 1: 20

Dyeing temperature = 70 ºC Maximum wavelength = 430 nm

Table 5 Photograph of Dyed Cotton with different mordant at dyeing time 60 min

Method / Mordant	1% alum	1% $CuSO_4$	1% $K_2Cr_2O_7$	1%$FeSO_4$
pre-mordanting				
post-mordanting				
simultaneous-mordanting				

Table 6 Colour Fastness Grade of AllDyed Samples

Dyed Samples		Washing			Lighting	Rubbing	
Dyeing Method	Mordants	C/S	C	P		Dry Mark	Wet Mark
Direct Dyeing	-	3	4	4	4	3-4	4
Pre – Mordanting	Alum	3	4	4	4	3-4	3
	Copper II Sulfate	3	4	4	3	3-4	3
	Ferrous Sulfate	1	4	4	3	3	2-3
	Potassium Dichromate	3	4	4	1	4	3
Post-Mordanting	Alum	3	4	4	3	4	3
	Copper II Sulfate	3	4	4	4	4	3
	Ferrous Sulfate	2	4	4	2	3	2
	Potassium Dichromate	3	4	4	1	4	3
Simultaneous-Mordanting	Alum	2	4	4	4	4	3
	Copper II Sulfate	2	4	4	4	3-4	3
	Ferrous Sulfate	1	4	4	4	3	2
	Potassium Dichromate	2	4	4	1	4	3

Score for fastness test

Grade 1 – Very poor Grade 2 – Poor Grade 3 – Fair

Grade 4 – Good Grade 5 – Excellent

C/S = Change of Shade : C = Staining on Cotton : P = Staining on Polyester

The light fastness grade of dyed samples using pre-mordanting method for 60 min was fair grade (3-4), post mordanting method was fair grade (3-4) and simultaneous-mordanting was good grade (4) (except used of potassium dichromate mordant). The results are presented in Table 6. According to light fastness results, direct dyeing method (without mordant) and simultaneous-mordanting method are good grade. However, Potassium dichromate is not suitable mordant for extracted dye from marigold flower.

In general, dry rubbing fastness of all dyed samples is better than wet rubbing fastness. The results are presented in Table 6. According to rubbing fastness, direct dyeing method (without mordant) is good grade. In pre, post and simultaneous – mordanting method, the rubbing fastness for dry and wet mark of all dyeing times are not too different. These are the heavy depths rating are good but ferrous sulfate is not suitable mordant for extracted dye from marigold flower.

The change of shade of dyed samples making pre and post-mordanting method was fair grade (3) and simultaneous-mordanting method was 2 (except used of ferrous sulfate mordant). The change of shade of dyed samples without mordant was 3. The results are presented in Table 6. According to washing fastness results, the

colour staining on cotton and polyester of all dyed samples was good grade. In pre and post– mordanting method, the change of shade of dyed samples were fair grade (except used of ferrous sulfate mordant). In simultaneous-mordanting method, the change of shade of dyed samples was poor grade. The change of shade of dyed samples without mordant was fair grade. Thus, ferrous sulfate is not suitable mordant for extracted dye from marigold flower

Conclusion

The extraction of natural dye from marigold flowers with water at 70 °C. The yield percent of extracted natural dye from marigold flower is 28.8 %. The pH of extracted dye was 6.05 and maximum wavelength was 430 nm. The observed colour of natural dye was yellowish brown. The colour intensity of extracted natural dye was investigated by Tintometer. From this result, the extracted dye giving 2.0 yellow, 2.2 red and 0.1 blue.

The effect of dyeing time on dye uptake was determined for direct dyeing method. According this result, (27 %) of dye uptake on cotton fabric for 60 min is suitable than the others.

The colour fastness properties of dyed samples of direct dyeing and pre, post and simultaneous-mordanting method are not too different (except potassium dichromate and ferrous sulfate mordant). Therefore, potassium dichromate and ferrous sulfate are not suitable mordant for extracted dye from marigold flower.

Thus, extracted dye from marigold flower was successfully applied on cotton dyeing and the use of direct dyeing method (without mordant) not only provide good grade of colour fastness properties but also reduce the environmental pollution with mordant chemicals.

Acknowledgements

I would like to express my special thanks to Dr Nu Nu Yee and Dr Nay Thwe Kyi, Pro-Rector, Dagon University for their kind permission to do this research.

I would like to express my sincere gratitude to Professor and Head Cho Cho Win, Department of Chemistry, Dagon University and Professor Dr Khin Than Win, Department of Chemistry, Dagon University for providing of all the departmental facilities, invaluable guidance and constant encouragement. Finally, my sincere thanks go to all my colleagues from the Department of Chemistry, Dagon University for their help in various ways.

References

Ashis, K. S and A. Priti. (2009). "Application of natural dyes on textiles". *Indian Journal of Fibre and Textile Research*, **34**, 384-399

Chandan, K.J., R. Kumar, V. S. Kumar and V, Devi Rajeswari. (2015). "Extraction of natural dye from marigold flower (*Tegetes erecta* L.) and dyeing of fabric and yarns: A focus on colourimetric analysis and fastness properties". *Der Pharmacia Lettre*, **7**(1), 185-195

Clarke, W. (1977). *An Introduction to Textile Printing*. London: Fourth Edition, Butterworth & Co (Publisher) Ltd., 56-72

Gauri Karwani and S. Siddhraj .(2015). "*Tegetes erecta* Plant: Review with significant pharmacological activities". *World Journal of Pharmaceutical Sciences*, **3**(6), 1180-1183

Samuels, T. M. (2017). "Information about *Tegetes* Flowers". https://www. gardenguides.com/85758-information-Tegetes-flowers.html, (Accessed, 6th January 2019)