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The Pharma Innovation

**ISSN (E): 2277- 7695**
**ISSN (P): 2349-8242**
**NAAS Rating: 5.03**
TPI 2019; 8(3): 517-520

www.thepharmajournal.com
Received: 18-01-2019
Accepted: 22-02-2019

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# Silk dyeing with natural dye extracted from floral parts of African marigold (*Tagetes erecta* L.) and its fastness

**Amit Lohar and Jayoti Majumder**

**Abstract**
The present study demonstrated dyeing of silk fabric with the pigment extracted through various methods from marigold (*Tagetes erecta* L.). Marigold creates the colour range from yellow to orange. Dyeing of 100% silk along with mordanting techniques which included scouring, mordanting and pre mordanting was carried out. After scouring, mordanting and dyeing the fabrics were evaluated for colour measurements in HunterlabD25LT Chroma Meter for L* a* and b* values. The L* value were found to be superior when the fabrics were treated with alum. The highest L* value (76.11) was obtained in $T_3$ i.e., pre-treatment of Stannous chloride + 70% alcohol + shade dry in marigold. The increased a* value was found in $T_9$ with (7.22) Alum + 70% alcohol + shade dry in marigold. b* $T_9$ with (48.31) Alum + 70% alcohol + shade dry was found in marigold dyed silk fabric respectively. Good light fastness, wash fastness and rubbing fastness were observed in case of fabrics mordanted with copper sulphate. These findings reveal that *Tagetes erecta* L. can serve as a potential source of natural colorant which can be used in textile industry for dyeing purpose.

**Keywords:** Silk, dyeing, *Tagetes erecta* L. mordant, fastness

## Introduction
India has a rich biodiversity and it is not only one of the world's twelve mega diversity countries, but also one of the eight major centers of origin and diversification of domesticated taxa). Nature has gifted us more than 500 dye-yielding plant species (Dayal and Dobhal 2001, Siva 2007) [3, 10] in existence among them the edible ornamental crops like African marigold (*Tagetes erecta* L.) are acknowledged (Niizu & Rodriguez-Amaya, 2005) [9]. Marigold is one of the widely used flowers in many of the Indian festivals, rituals and traditions. African marigold is known for their large flower sizes with very bright orange colour. Most of the flowers discarded after use and hence natural dyeing is an alternative to use this wasted biomass. Marigold flowers are rich in bio active components especially the carotenoid pigment, lutein. Nowadays, Lutein is becoming an increasingly popular active ingredient used in the Food Industry and Textile coloration. This pigment has acquired greater significance because of its excellent colour value. In 1972, Quackenbush and Miller separated lutein and 16 common lutein fatty acid esters which, on the whole, represented over 95% of the pigments identified in the petals of the marigold. They are also valued for aesthetics and medicinal uses. The gigantic application of these synthetic dyes has escalated environment pollution and health hazards (Bechtold *et al*. 2003) [2]. Therefore, green minded consumers are back to natural and biodegradable alternatives. The sources of natural dye could be mainly three types like animals, plants and minerals (Jha *et al*. 2015) [4]. One such product from nature is the dye. Dyes are one of the most important uses of the plants, as they are related with cultural practices, rituals, arts and crafts, fabrics and to satisfy personal embodiment, however, dye yielding plants have not received significant attention. Although marigold flower extract has been used in veterinary feeds, the potential use of marigold as a natural textile colorant has not been exploited to its full extent. This is due to the lack of information on its safety, stability, and compatibility in textile coloration (Jothi, 2008) [5]. Natural dyes are mostly non-substantive and must be applied on textiles by the help of mordants, usually a metallic salt, having an affinity for both the colouring matter and the fibre. Therefore, the present study was performed with the explicit objective of extracting natural dye from the petals of african marigold flower using different solvents such as Ethanol, Hcl and Water, and to investigate its dyeing effects on silk fabrics samples. The effects of various mordants on the stability of the dye as well as on the color shade were also analyzed. The color shade differences, L*, a*, b*, ΔE values were estimated via CCM software analysis tool using GretagMacbath 7000 A º spectrophotometer.

Light fastness, Wash fastness and Rubbing fastness were also evaluated for fabrics sample.

**Materials and Methods**

The experiment was conducted at Department of Floriculture and Landscaping, Faculty of Horticulture, Bidhan Chandra Krishi Viswavidyalaya, Mohanpur, Nadia, West Bengal in 2016 to 2017. Fully bloomed marigold flowers of seracole variety freshly harvested in the early morning when they were fully turgid were procured from the experimental plots of the Department of Horticulture. The fabric of silk was bought from Nalli®, India. All chemicals used were of analytical grade, Ethanol (EtOH), Hydrochloric acid (Hcl), carbonate ($Na_2CO_3$) were procured from Merck (Darmstadt, Germany). The mordants (stannous chloride, copper sulphate and alum) were purchased from HiMedia (Mumbai). The dyeing method was carried out stepwise i.e., pre-treatment of flower petals, extraction of dye, mordanting and dyeing of fabric and colour fastness test.

**Dye Extraction**

The 5 gm of flower petals were crushed in a blender and further homogenised. The crushed samples were transferred into conical flasks for different pre-treatments viz. 50% EtOH (Ethanol-Water mixture of 50:30 v/v), 70% conc. Hcl (Hydrochloric acid – Water mixture of 70:30 v/v) and 0.5g/l $Na_2Co_3$ in a ratio of 1:20 (w/v) was carried out by boiling at 85 $^0$C for 2 hour with regular stirring. The extract was filtered using muslin cloth and the biomass was re-heated for next half hour and kept in hot air oven at 60 $^0$C overnight and extract was collected using muslin cloth.

**Scouring of cotton and silk cloth**

Scouring of 10cm × 6cm silk cloth 100% was done by washing it in a solution containing 2 g/L non-ionic detergent solution at 50 °C for 25 minutes. The scoured material was thoroughly washed with tap water and dried at room temperature. The scoured material was soaked in clean water for 30 mins prior to dyeing or mordanting. (Jothi, 2008; Kulkrani. S.S *et.al*, 2011) [5, 7]

**Mordanting**

Silk cloth were treated with Alum, Stannous chloride and copper sulfate individually (2% mordant was made in distilled water). The cloth were brought to boil at 80°C for 30min and left for another 30min in the mordant solution. This mordanted material was then rinsed, squeezed and dried at room temperature. Mordanted cloth was immediately used for dyeing since the mordants are light sensitive (D. Jothi, 2008) [5].

**Dyeing Procedure**

The cotton and silk samples were dyed with dye extract. Dyeing was carried out at 80 °C for 1hour in water bath. After the completion of dyeing; the samples were washed off with cold water and dried at the room temperature. The dyed samples were then dipped in a brine solution for dye fixing.

**Colour measurement**

The color strength of the colored samples was analyzed using CCM software (Gretag Macbath 7000 A °spectrophotometer). L*, a*, b* values were obtained. The maximum value for L* is 100 which represents a perfect reflecting diffuser. The minimum value for L* is 0 which represents black. Positive 'a' is red; negative 'a' is green. Positive 'b' is yellow and negative 'b' is blue. The color axis are based on the fact that colors cannot be both red and green, or both blue and yellow, because these colors oppose each other. The average color values for the samples were recorded and the total color difference ΔE* was calculated, which was single value that takes into account the differences between L*, a*, b* of the sample and the standard. The total colour difference ΔE* was calculated through the average colour values (Jha *et al*. 2016) of the samples.

$$\Delta E = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2}$$

**Fabric fastness test**

The dyed material was tested viz. silk while dyeing with marigold has been done in this study. The fastness tests which were followed were i) light fastness and ii) wash and rubbing fastness. For light fastness, the fabrics were exposed to direct sunlight for 1 day. Then the colour fastness was evaluated comparing the colour changed due to sunlight exposure with unexposed or control fabrics. The colour fastness to light was evaluated by comparison of colour change of the exposed portion to the unexposed original material (Samanta and Agarwal 2009) [11].

For washing and rubbing fastness, the dyed samples were sandwiched in between two undyed fabric sample and then they were placed with preheated washing solution (Surf, at 60 °C) in a ratio of 1:50 (washing solution: water). 1.0 g of sandwiched fabric were put inside it for 30 minutes and rubbed manually. They were rinsed in cold water and dried in room temperature. The wash and rubbing fastness was measured by the presence of colour in un-dyed sample (Samanta and Agarwal 2009, Kanchana *et al*. 2013) [11, 13].

Scores for wash fastness and rubbing fastness: 5 - Excellent, 4-Good, 3- Fair, 2-Poor, 1- Very Poor

Scores for light fastness: 1- Very Poor, 2-Poor, 3- Moderate, 4- Fairly good, 5-Good, 6- Very good, 7- Excellent, 8-Outstanding.

**Statistical analysis** L*, a*, b*, ΔE values and Fastness Properties of each dyeing condition was evaluated by analysis of variance and each treatment was replicated three times the standard error of difference SED(±) was calculated for each mordant.

**Results and Discussions**

**Colour measurements**

It can be observed in Table 1, the effect of stannous chloride over the dye extracted from marigold showed brighter colour effect than copper sulphate and alum. The hue (L*) value of marigold changed a little with alum. In case of silk fabric the L* value was superior (76.11) while the dye was extracted through the treatment combination of $M_1S_2$ viz. (Stannous chloride + 70% alcohol + shade dry) and minimum results in treatment $M_1S_1$ (61.77) (Stannous chloride + Deionized water + shade dry). The a* value (redness) was superior where the silk fabric was treated with alum. The highest a* value (7.22) was obtained in silk fabric from marigold dye with $M_3S_2$ treatment (alum + 70% alcohol + 15days shade drying), and lowest results was observed by $M_1S_3$ (0.38) (stannous chloride + (1N) 20%Dil.Hcl + shade dry). b* value (alum + 70% alcohol + 15days shade drying) results in the highest increase in treatment $M_3S_2$ (48.31), and minimum was observed in $M_1S_2$ (18.40) (Stannous chloride + 70% alcohol + shade dry).

According to Table 3, the dE* value is found to be the highest (85.36) in marigold dye with the $M_3S_2$ treatment in combinations of (70% alcohol + shade drying + alum). Jothi (2008) [5] showed that the effect of alum and stannous chloride mordant on marigold dye and found the alum gave superior L* and a* value over stannous chloride.

**Fastness properties of dyed samples**

**1. Light fastness of dyed samples**

Good to very good (4-5) fastness was observed in silk fabrics dyed with extract of marigold flower using different mordant are indicated in Table 1. $T_7$ (2% Copper sulphate + 1N 20% Dil. Hcl) recorded the highest grade of 4-5. The next best treatments was found to be $T_9$ (70% alcohol + 2% alum) and $T_6$ (70% alcohol + 2% Copper sulphate) by obtaining grade of 4 and they are on par with each other. The lowest grade (2) was recorded in $T_1$ Control. This may be due to formation of a complex with the metal salts which protects the chromatophore to minimum photolytic degradation (Ali *et al.*, 2009) [1]. For light fastness, the substitution pattern of dyes seems to play an important role for colour determination. The increase or decrease of electron density through the substituent may accelerate oxidation otherwise reduction reaction of molecules. Which can be seen that the marigold lutein has two hydroxyl groups gives good fastness (Ali *et al.*, 2009, Kanchana *et al.*, 2013) [1, 13].

**2. Washing Fastness**

Marigold dyed silk fabric when mordanted with $T_7$ (2% Copper sulphate + 1N 20% Dil. Hcl) recorded the highest grade of 4-5. The next best treatments was found to be $T_9$ (70% alcohol + 2% alum) registered the maximum grade of 4 for wash fastness property (Table 1). The lowest grade of 1 was registered in $T_1$ (Control-No mordant + Deionized water). That a change of some of the colors could be noticed in the silk dyed samples with different mordants. (Kumaresan *et al*., 2011) [7] after washing with soap. This is may be due to several factors, such as: - The dye itself decomposes, thus converting to colorless or a differentially colored compound. - The dye detaches from the substrate due to the wear dye-fiber bond between the natural dye and the fiber. - Ionization of the natural dye during alkaline washing.

**3. Rubbing fastness**

With regards to the rubbing fastness of cotton fabric dyed with marigold $T_7$ (1N 20% Dil. Hcl + 2% copper sulphate) recorded the maximum grade (4-5) under varying dyeing treatments (Table 1). This is followed by $T_5$ (Deionized water + 2% copper sulphate) $T_3$, $T_6$, $T_9$ and $T_{10}$ by obtaining grade of (4) and they are on par with each other. $T_1$ (Control) registered the lowest grade (2) for rubbing fastness. In general, rub fastness of all the mordants used with the natural dyes of marigold are found to be good to excellent and does not require any after treatment (Samanta and Agarwal, 2009) [11]. Kumaresan *et al.* (2011) [7] also evaluated the colour fastness to rubbing (dry and wet) of silk dyed with an extract of *cordial subestena* using a manually operated crock meter and grey scale as per 1S0-105 A03 (extent of staining) as excellent.

**Table 1:** Different pre-mordant, fastness properties, and L*, a*, b* values for dyed silk with Marigold flower at $l_{max}$ = 453nm

| Treatment | L* | a* | b* | dE* | Wash fastness | Light fastness | Rubbing fastness |
|---|---|---|---|---|---|---|---|
| $T_1$ | 70.53 | 3.43 | 25.92 | 75.22 | 1 | 2 | 2 |
| $T_2$ | 61.77 | 3.78 | 19.78 | 64.97 | 4 | 3 | 4 |
| $T_3$ | 76.11 | 0.48 | 18.40 | 78.30 | 3 | 3 | 4 |
| $T_4$ | 73.31 | 0.38 | 39.10 | 83.08 | 3 | 3-4 | 4 |
| $T_5$ | 73.96 | 1.26 | 26.59 | 78.60 | 4 | 4 | 4-5 |
| $T_6$ | 66.05 | 3.15 | 37.73 | 76.14 | 4 | 4 | 4 |
| $T_7$ | 71.97 | 0.60 | 36.01 | 80.48 | 4-5 | 4-5 | 4-5 |
| $T_8$ | 73.18 | 0.52 | 37.93 | 82.43 | 3-4 | 4 | 4 |
| $T_9$ | 70.00 | 7.22 | 48.31 | 85.36 | 4 | 4 | 4 |
| $T_{10}$ | 70.53 | 0.78 | 32.68 | 76.86 | 4 | 3 | 4 |
| Mordant (A) | 0.08±0.01 | 1.08±0.18 | 5.46±0.90 | 2.42±0.40 | | | |
| Solvent (B) | 0.08±0.01 | 1.08±0.18 | 5.46±0.90 | 2.42±0.40 | | | |
| A×B | 0.06 ± 0.02 | 0.86±0.22 | 4.32±1.10 | 1.91±0.49 | | | |

Values are means ± SE of three independent determinations.

$T_1$ - Control, $T_2$ - stannous chloride + deionised water, $T_3$ - stannous chloride + 70% alcohol, $T_4$ - stannous chloride + (1N) 20%Dil.Hcl, $T_5$ – copper sulphate + deionised water, $T_6$ - copper sulphate + 70% alcohol, $T_7$ - copper sulphate + (1N) 20%Dil.Hcl, $T_8$ – Alum + deionised water, $T_9$ - Alum + 70% alcohol, $T_{10}$ - Alum + (1N) 20%Dil.Hcl

**Conclusion**

The present study showed that natural dye can be successfully extracted from the flowers of *Tagetes erecta* L. The Whole process of extraction is eco-friendly. The maximum dye extraction was observed at 95 ºC using aqueous extraction method. The obtained results have shown that the dyeing potential of the marigold flower could be huge which can be used as a source of textile dyeing. The various color shades can be obtained using safe and ecofriendly mordants. Good washing and light fastness using $CuSO_4$ mordants may be a pleasant source of thinking about the commercialization of natural dyes for such textile goods which is in its end uses needed to be color fast while washing and exposing to light specially. To cope with the existing traditional practices and so called disadvantages of natural dyeing, a much more researches and scientific practices are required to go in this area.

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