ISSN: 2395-4108

Abrahamia

An International Journal of Plant Sciences

Eulophia andamanensis

VOLUME 8 • NUMBER 2 • 2022

DEPARTMENT OF BOTANY, UNIVERSITY OF KERALA
Kariavattom, Thiruvananthapuram, Kerala, India - 695581

Abrahamia

An International Journal of Plant Sciences

EDITORIAL BOARD

Printed and Published by
Head
Department of Botany, University of Kerala, 2022

Cover Photo: Dr. Mehtab Jahan Bukhari
Cover Design & Layout: Godfrey's Graphics, 9447451314
Printed at: University Press, Palayam Campus, University of Kerala

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An International Journal of Plant Sciences

VOLUME 8 NUMBER 2 2022

DEPARTMENT OF BOTANY, UNIVERSITY OF KERALA
Kariavattom, Thiruvananthapuram

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Abrahamia
Vol. 8 (2)
45-50
2022
ISSN: 2395-4108

Spent Flowers of *Tagetes erecta* L. – An Alternative Source of Natural Dye

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ABSTRACT

***Tagetes erecta Linn.* is one of the important commercial flowers of Kanniyakumari district in Tamil Nadu, India. The flower occupies a prime position in various occasions and festivals. The production is high and requires only a minimum investment. Most of the flowers produced in the district are often going wasted. The present investigation was undertaken to utilize the spent flowers from floral markets as a source of natural dye. The dye was extracted in aqueous, acidic, and alkaline medium from the dried petals of the flower, and the resultant colors extracted were fastened onto a cotton cloth, the results of which are discussed.**

Key Words: - *Tagetes erecta* L. Natural dye, Mercerization, Mordants, Dye Fastening,

INTRODUCTION

Natural dyes are chemicals or colorants derived from natural sources such as animals, plants, minerals and microbes. Of these plants are the major sources of natural dyes. Various plant parts, including roots, leaves, twigs, stems, heartwood, bark, wood shavings, flowers, fruits, rinds, hulls, husks and the like serve as natural dye sources (Saxena and Raja, 2014). The natural dyes are mostly non substantive and must be applied on textiles with the help of a mordant which are metallic salts having the affinity for both the coloring matter and the fiber and can act as a bridging material to create substantively of natural dyes/colorants when the later is mordanted (Samantha, 2011). Apart from dyeing textiles, the natural dyes are used for dyeing hairs and nails, coloration of food, medicines, handicraft items, toys and leather processing (Saxena and Raja, 2014). They are related with cultural practices, rituals, arts and crafts and also to satisfy personal embodiment (Grover and Patni, 2011).

Natural dyes are ecofriendly, renewable, biodegradable and non-toxic may also provide health benefits to the wearer. Natural dyes exhibit several important properties that provide them a significant edge over synthetic dyes. Some of the advantages of natural dyes are they are biodegradable, non–toxic and non–allergic. Fabric dyed with natural dyes exhibits higher UV absorption that can result in reduced incidence of melanoma. Natural dyes are moth proof and can replace synthetic dyes in kid's garment and food stuff for safety (Arora, *et al.,* 2017). Plant kingdom is the major source of natural dyes. Some of the important dyes of plant origin are Blue dyes (*Indigofera tinctoria, Wrightia tinctoria*), Red dyes

(*Rubia cordifolia*, *Morinda citrifolia),* Yellow dyes (Turmeric, *Crocus sativus* (Saffron), *Tagetes spp.* (Marigold), Brown dyes (*Acacia catechu*), and Black dye (*Haematoxylon campechianum*).

Many of the plants used for dye extraction are classified as medicinal, and some of these have recently been shown to possess antimicrobial activity *Punica granatum* (Hussein, 1997), and many other common natural dyes are reported as potent antimicrobial agents owing to the presence of a large amount of tannins. Several other sources of plant dyes rich in naphthoquinones, such as lawsone from *Lawsonia inermis* (henna), juglone from walnut and lapachol from alkanet, are reported to exhibit antibacterial and antifungal activity (Siva, 2007). Optimized natural dye powders of *Acacia catechu, Kerria lacca*, *Rubia cordifolia* L. and *Rumex maritimus* showed antimicrobial activities (Singh *et al.* 2005). This is clear evidence that some natural dyes by themselves have medicinal properties.

According to Waghmonde *et al*., 2018, floral waste is one of the major concerns. Flowers have applications in many industries viz; perfumes, cosmetics, food, liquor and textile industries. Disposal of flowers in rivers, oceans, etc. leads to water pollution as well as affects the living organisms present in the waters. The dyes and pigments from floral wastes will have applications in various textile industries. The production and application of synthetic dyes release vast amounts of waste and unfixed colorants causing serious health hazards to humans and other important organisms. They are allergic, carcinogenic and detrimental to human health. They also cause skin diseases. The synthetic dyes disturb the eco-balance of nature and generate water pollution as well as waste disposal problems (Arora, *et al.,* 2017).

According to Shetty *et al.* 2015, flowers of *Tagetes erecta* is used traditionally from ancient times. Different parts of this plant including flower are used in folk medicine to cure various types of diseases. The flower are used to cure fever, epileptic fits according to Ayurveda, astringent, carminative and stomachic, scabies and liver complaints and is also employed in diseases of the eyes. To purify blood and bleeding piles, rheumatism and bronchitis, flower juice is given as a remedy for bleeding piles (Kadam *et al.,* 2013). Mexicans use decoctions of flowers and leaves as diuretics and carminative. Other folklore uses of *Tagetes* include its use in anaemia, irregular menstruation, abdominal pain, muscular and bone pain. Internally *Tagetes* used for indigestion, colic, cough and dysentery.

Besides, it is also popular for having medicinal properties, such as analgesic, antispasmodic, immune stimulant, laxative and anthelmintic (Priyanka *et al*., 2013) and bactericidal (Jain *et al*., 2012). The petals are useful in fevers, epileptic fits (Ayurveda), astringent, carminative, stomachic, scabies and liver complaints and are also employed in diseases of the eyes. The present work was done in the spent flower petals of *T. erecta*, which is one of the most important commercial flowers in Kanniyakumari district of Tamil Nadu. An attempt was made to extract the natural dye from spent *T. erecta* flowers. These flowers were also phytochemically profiled and experimental dyeing of the flower extract was undertaken to bring out its importance as a natural dye.

MATERIALS AND METHODS

The spent flowers of *T. erecta* were collected from various floral markets of Kanniyakumari District in Tamil Nadu, India. It was rinsed under running tap water to eliminate dust. After that, the petals were separated from the whole cut flower and were air dried for 25 days under room temperature. The dried sample was powdered using mixer grinder and the powdered samples were kept in a clean, dried, air tight glass container to protect it from sunlight and labeled.

Dye Extraction Processes

Extraction procedure of Patil *et al*., 2016 was followed. This procedure was slightly modified as follows. A control was also used in which no mordant was used.

1) **Aqueous extraction:** By preparing an aqueous solution of flowers (10g in 50 ml distilled water) in the ratio 1:5 and the extraction process was carried out in a heating mantle at a temperature of 85^0 C for 1 hour (Sample A)

2) **Alkaline extraction:** In second method, the flowers (10 g) were placed 50ml of 1% NaOH for extraction in the ratio 1:5. This pasty mass was kept for 60 minutes in a mantle at a temperature of 85-100^0C to extract the dye (Sample B).

3) **Acidic extraction:** In the third method of dye extraction, the flowers (10 g) were placed 50ml of 2% acetic acid for extraction in the ratio 1:5. This was kept for 60 minutes in a mantle at a temperature of 85-100^0C to extract the dye (Sample C).

Mercerization

i. Cloth used for dyeing was boiled in NaOH solution (10%) for 15-30 minutes to remove starch from the cloth, and then washed with lukewarm distilled water.

ii. Cloth used for dyeing was boiled in vinegar for 30 minutes and it is taken out. The excess vinegar is washed with lukewarm distilled water and it is then introduced into the dye bath.

Dyeing Process

Dyeing procedure of Grover and Patni, 2011 was followed. This procedure was slightly modified as follows. The extracts obtained through above mentioned methods were filtered and used for dyeing. Cotton fabric was used for the procedure.

i. This cloth was then transferred in the dye bath containing the mixture of mordant and dye solution and kept for 60 minutes in warm temperature (65-70°C). It is then kept undisturbed for 30 hours. Then the cloth was taken out. It was then washed with distilled water followed by washing with a herbal hair wash (Color fixative) and dried in sunlight.

ii. Effect of dye without mordanting the fabric is also studied. Then the cloth was treated with herbal hair wash (Color fixative) and dried in sunlight.

The mordants used here is 3% $CuSO_4$, and 4% ferrous sulphate.

Dye Fastening Test

The dye fastening procedure of Grover and Patni, 2011 will be followed. The sundried cloth will be further evaluated for its color, lightness and wash fastness.

Wash fastness was tested by washing with soap water (10% w/v).

The heat resistance was tested by keeping the cloth at various temperatures, viz. 50^0C, 60^0 C, 70^0C for 30 minutes in the oven without water.

RESULTS AND DISCUSSION

Biodegradable dyes have emerged as important alternative to synthetic dyes. There is a report on use of petal part of the saffron flower to extract dye for application on the Pashmina shawl (Raja *et al*., 2012). The potential use of Hibiscus as a natural dye in textile coloration has been reported by Teli *et al*., 2013. The main advantage of floral dyes is that they are very eco-friendly and have no allergic action on skin. Also, flowers are cultivated widely; easily available and inexpensive, which facilitates their use for dyeing paper at the level of small and medium enterprises as well as larger commercial scale. Earlier reports by Jhathod and Rathod, in 2013 have reported that patuletin dye extracted from marigold (*Tagetes erecta*) and French marigold floral wastes (*Tagetes patula*) is used in textile industries and also in antioxidant treatment.

These days environmental protection has become a challenge for the textile industry because it utilizes a lot of chemicals for coloration of textile materials. These chemicals are

harmful to humans as well as environment and are even carcinogenic and mutagenic. In this era of green minded consumer, interest in natural dyes as growing mainly because natural dyes have been shown to possess health-promoting and eco-friendly properties (Singh and Srivastava, 2015). In this present investigation an attempt was made to extract natural dye from spent flowers of *Tagetes erecta* and used to dye cotton. The shade dried flower powder was subjected to dye extraction, mercerization and dye fastening processes.

Table 1
Showing Natural dye extraction and dyeing processes.

EXTRACT	MERCIRIZATION	MORDANT	COLOR DEVELOPED
Acidic extract	Vinegar	Control	Yellow green
	NaOH		Olive green
	Vinegar	$CuSO_4$	Dark orange
	NaOH		Brown
	Vinegar	$FeSO_4$	Dark grey
	NaOH		Black

Aqueous extract	**Vinegar**	**Control**	**Lemon yellow**
	NaOH		**Light olive**
	Vinegar	**$CuSO_4$**	**Sienna**
	NaOH		**Saddle brown**
	Vinegar	**$FeSO_4$**	**Dark olive green**
	NaOH		**Black**
Alkaline extract	**Vinegar**	**Control**	**Lemon green**
	NaOH		**Dark lemon green**
	Vinegar	**$CuSO_4$**	**Dark orange**
	NaOH		**Golden brown**
	Vinegar	**$FeSO_4$**	**Slate grey**
	NaOH		**Light grey**

Table 1 shows the mordant and mercerization solvents used and the colors developed in the cloths. For dyeing, cotton fabric was first mercerized with either vinegar or NaOH for 30 minutes and after that it is was exposed to mordants $CuSO_4$ or $FeSO_4$ and then transferred to the dye bath. In each step the cotton fabric not treated with mordant served as the control. In the whole dyeing process, the cloth mercerized with vinegar produced light colors of the cloth mercerized with NaOH produced darker shades. The different color shades were produced with different mordants of the same extract.

Table 1 and Plate 1 shows the colors produced by cotton fabric dyed with various extract of *T. erecta* flower petals. The acidic extract of *T. erecta* flower petals when drenched in the control cotton fabric treated with vinegar or NaOH had yellow green or olive green color respectively. When the fabric was mercerized with vinegar and exposed to $CuSO_4$ (mordant) dark orange color was observed and the cotton after mercerizing with vinegar and the exposing to mordant $FeSO_4$ a dark grey color was observed **(Plate 1& Fig 1)**. The cotton fabric treated with NaOH and mordant $CuSO_4$ produced brown color while that treated with NaOH and mordant $FeSO_4$ produced dark olive shade. The fabric dipped in aqueous extract, and treated with vinegar and not exposed to any mordant produced lemon yellow color while the cotton fabric treated with vinegar and exposed to mordant $CuSO_4$ produced sienna color and the fabric treated with vinegar and mordant with $FeSO_4$ dyed as dark olive green colored one **(Plate 1& Fig 2).** While the cloth dipped in aqueous extract was treated with NaOH and no mordant was dyed as light olive green color. Sodium hydroxide mercerized aqueous extract dipped cotton fabric when exposed to $CuSO_4$ mordant showed saddle brown color and when $FeSO_4$ mordant was used instead of $CuSO_4$ black color was dyed in the cloth **(Plate 1& Fig 3).** In the alkaline extract, the cloth treated with vinegar and no mordant produced lemon green color; the cloth treated with vinegar and $CuSO_4$ produced dark orange color and the cloth treated with vinegar and $FeSO_4$ produced slate grey color. While the cloth treated with NaOH and no mordant produced dark lemon green color; the cloth treated with NaOH and $CuSO_4$ produced golden brown color and the cloth treated with NaOH and $FeSO_4$ produced dim grey color **(Plate 1& Fig 3).**

During the dye fastening test, when the dyed clothes were exposed to 50^0C, 60^0C and 70^0C for 30 minutes in a hot air oven there no change in color. When washed in soap water, there was no change in the color of the dyed fabric nor was there any bleeding of color from the fabric which was dyed using mordant. The control fabric (cotton fabric clothes dyed without using mordant) was washed there is a slight change in color due to the alkalinity of the soap water but regained its original color when the soap particles is completely washed off from the fabric. There is no bleeding of color and staining other fabric occurred in the control cotton fabric. For estimating the amount of carotenoids present in 1g of the sample spectrophotometry was used and it was found that dried 1g of flower sample contains 2.5 mg of carotenoids.

Floral dye sources are more important for dyeing of textile materials as it provides both dye as well as fragrance. Dye pigments of flowers provide color to the textile while fragrance of the floral dye will help in retaining freshness of the textile material by keeping body odour away from the garment for a long period of time (Singh *et al.*, 2005). Natural dyes in textile industry are going to be one of the thrust areas in the near future. However newer techniques for dyeing without the use of metal mordant have to be coined so as to still reduce artificiality. The importance of *T. erecta* has been brought out again and again by many reviews and works the latest of which is by Singh *et al*., 2005. *Tagetes* is stated in literature as popular dyeing plant (Kaffková, and Kaššák 2018); however, the use of metal mordant as an essential ingredient is common to obtain high levels of color intensity. The main coloring component present in the petals of *T. erecta* is a carotenoid compound called lutein. The carotenoids, xanthophylls and isoflavonoids give color to the extract. Lutein is an oxycarotenoid, or xanthophylls, containing 2 cyclic end groups (one beta and one alpha-ionone ring) and the basic C-40 isoprenoid structure common to all carotenoids. It is one of the major constituents and the main pigment of *T. erecta* (Dixit *et al*., 2013).

The present work agrees with Li-Wei (2011) that the flower petals of *T. erecta* serves as a promising source for natural dye extraction. African marigold (*T. erecta*), a major source of carotenoids and lutein, is grown as a cut flower and a garden flower, in addition to being grown for its medicinal values. Marigold flowers which are yellow to orange red in color, are a rich source of lutein, a carotenoid pigment. 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 which is due to the lack of information on its safety, stability, and compatibility in textile coloration. The present investigation has standardized a protocol for extracting natural dye from spent flowers of *T. erecta* one of the important commercial flowers of Kanniyakumari district in Tamil Nadu thereby an application of these spent flowers (waste). This initiative is a basic work of post graduate level which can serve as an alternative source of income to the student community as most of them (eighty percent) comes from seasonal fresh floral merchant families.

Plate 1

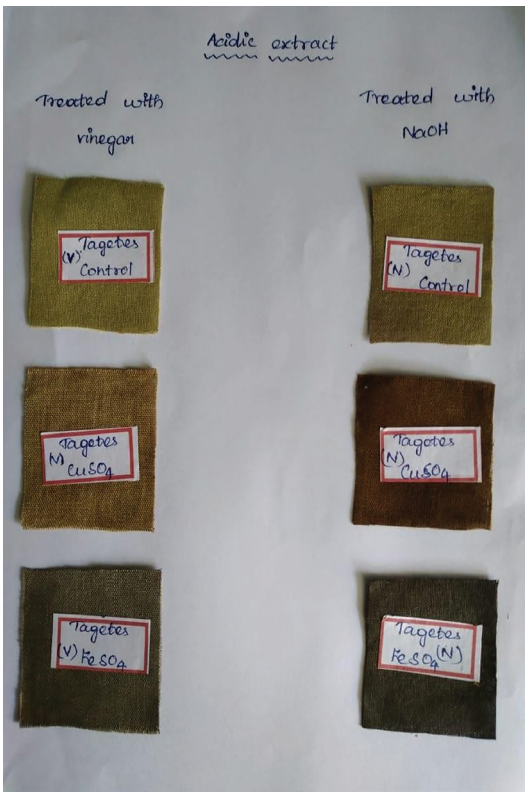

Fig 1

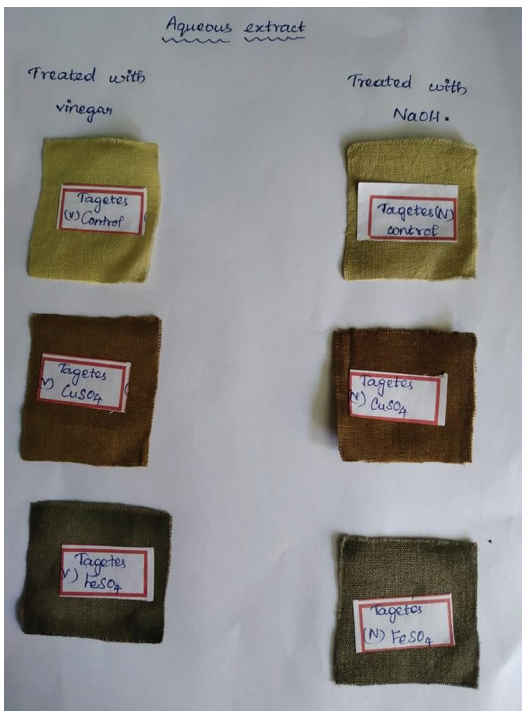

Fig. 2

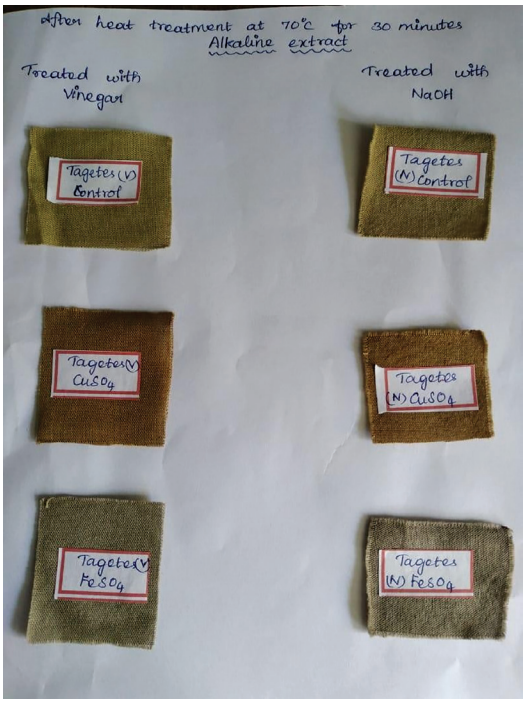

Fig 3

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Received: 9 February 2023
Revised & Accepted: 13 February 2023

Abrahamia
VOLUME 8 • NUMBER 2 • 2022

ISSN: 2395-4108

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Published by Department of Botany, University of Kerala
Kariavattom, Thiruvananthapuram, Kerala, India - 695581