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EXTRACTION OF NATURAL DYE FROM *Tagetes erecta* LINN. FLOWERS AND EVALUATION OF COTTON FABRIC DYEING USING DIFFERENT CHEMICAL MORDANTS

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ABSTRACT

Indians are considered as forerunners in the art of natural dyeing. The environment - friendly natural dyes are recently enjoying resurgence in popularity because of the concern with the carcinogenic, mutagenic and sensitizing characteristics of many synthetic dyes. The use of non-toxic and eco-friendly natural dyes on textiles has become a matter of significant importance because of the increased environmental awareness in order to avoid hazardous synthetic dyes, The phytochemical screening on saponins, flavonoids, steroids, terpenoids, triterpenoids, antroquinone, polyphenol, glycoside, tannins, alkaloids and coumarins were presence in aqueous *Tagetes erecta* flower extract. The prepared colour of the dye extract was found to be in yellowish pink colour. Among the three mordants method, pre-mordantning method gave excellent results as compared with normal cotton fabric. Among the three mordants of dyeing, the ferrous chloride mordants show excellent colour strength values as compared with stanneous and copper sulphate. In light fastness, pre-mordant has prevent the light fastness followed by post and simultaneous mordant. The stannous chloride mordant has retained the colour as compared to copper and ferrous sulphate chemical mordan. In wash fastness, post and pre-mordant has prevent the wash fastness followed by simultaneous mordant. The ferrous sulphate mordant has wash fastness the colour as compared to copper and stannous chloride chemical mordant. The Pantone Matching System (PMS) used as color reference system to monitor the colour and observed the codes were different for various mordant.

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INTRODUCTION

Colour is such a vital and vibrant ingredient of our existence that it is difficult to imagine what life would be like without it. Colour is one of the elements of nature that made the human living more aesthetic and fascinating in the world. Even in the prehistoric times, our ancestors must have noticed the abundance of a multitude of colours worn by nature. With the growth of civilization, it was realized that most of the colours he saw in nature (Clouds at Sunset, Flowers on Plants etc) were not permanent; the fading of the colours of flowers took place when their petals fell. The green colour of leaves changed in yellow or brown due to ageing. In his urge to make his world colourful, he tried extracting the colours from flowers plants and even animals. He found that these colours could be retained unchanged for a longer period unlike the natural thing wearing these colours. This led to the use of different types of natural colouring matters to dye clothes prepared from different natural fibres such as cotton, Linen, Wool, and Silk etc. He observed that some dyes, which produce intense action on woollen fabrics, did not even stain cotton clothes. A new class of dyes had to be used to dye cotton. Indigo, cutch, logwood, tyrian purple and henna were some of the natural dyestuff used for dyeing cotton fabrics (Shenai and Saraf 1991). This practice was common and prevalent in different periods of history, with the development of civilization; there was improvement in dyeing techniques. Use of natural dyes was common in most of the ancient civilizations e.g. India, Egypt, Greece, Aztec and others. Natural dyes or colorants derived from flora and fauna are believed to be safe because of its nontoxic, noncarcinogenic and biodegradable in nature (Cristea & Vilarem, 2003). Most of the natural dyes have no substantively on cellulose or other textile fibers without the use of a mordant. The majority of natural dyes need a mordanting chemical (preferably metal salt or suitably coordinating complex forming agents) to create an affinity between the fiber and dye or the pigment molecules of natural colorant. These metallic salts as mordant form metal complexes with the fibers and the dyes (Samanta et. al., 2011). Keeping in view, the present study is to investigate the phytochemical screening and natural dye extractions from *Tagetes erecta* flower extract applied to Cotton cloth.

MATERIALS AND METHOD

Collection of plant materials

The *Tagetes erecta* flowers were collected from the market "Poosanthai" in Thanjavur district, Tamil Nadu, India during December 2019. The collected flowers were washed several times with distilled water to remove the traces of impurities from the flower. Then examined carefully, old infected and fungus damaged portion of the flowers were removed. Healthy flowers were dried in room temperature and grind using grinder mixture. The powder was stored for further analysis.

Preliminary Phytochemical analysis

Preparation of flower extract

One gram of *Tagetes erecta* flowers were taken and made a fine powder, powder was added in 50 ml of aqueous solvent, the extract was shaken well for 30 minutes by free hand and wait for 24 hours. After the extracts were filtered using whatman filter paper No.1, the filtrate was used for further analysis.

Phytochemical screening

Chemical tests were carried out on the extract using standard procedures to identify the constituents as described by Sofowara (1993), Trease and Evans (1989) and Harborne (1973 and 1984).

Extraction of Dye from *Tagetes erecta* flowers extract

Material

Cotton fabric were purchased from local market at Thanjavur. It was used after bleaching for application.

Experimental methods

$CuSO_4$, $FeSO_4$ and $SnCl_2$ were used as mordants. For light shade, the cotton fabric is kept in the dye bath in short time and dark shade kept it for overnight and change the proportion.

Extraction of Dyes

The flowers of *Tagetes erecta* was cut into small pieces and put into distilled water and heated in a water bath for one hour to filtrate the extract. These dyes were used for dyeing cotton fabric. All material was thoroughly cleaned with distilled water and then after ground finely in the machine. Extraction was carried out throughout in aqueous media.

Dyeing Techniques

Pre-mordanting dyeing

The cotton fabric sample was dipped in one of the required mordanting solution M: L ratio as 1:20 for one and half an hour at temperature range 70-80^{0}C. Dyeing was done with extracted dye at the same temperature for one hour. The cloth was washed with cold water, squeezed and dried in air.

Simultaneous mordanting dyeing

Cotton fabric was dyed with dye extract and selected mordant simultaneously with keeping material to liquor ratio 1:20 at 70-80 ^{0}C with one and half hour and done the further process.

Post-mordanting dyeing

Cotton fabric was bleached and dyed with dye extract at 70-80 0C.with a half an hour. The dyed cotton fabric was taken out and squeezed, then the sample was treated with selected mordant without any washing to same material to liquor ratios as above process, washed with water and dry in air.

Method of Wash Fastness

As the sample to be tested is in cotton fabric form a piece measuring 10cm width by 4cm length was cut from each of the cotton fabric. The pieces of the undyed cloth enable the degree of staining during test to assessed. The sample was washed with 5g/l of soap in a solution with liquor ratio 50:1, at a temperature of 50˚C, for 45mins followed by rinsing and drying. The change in colour of the tested specimen and the staining of the adjacent undyed cloths were assessed with the appropriate grey scales.

Method of Light Fastness

The artificial light source method of determination of light fastness was used in this study. The specimens were exposed behind a glass and inserted into the light fastness testing machine. Exposure was carried out for 48hrs. Exposure was terminated after the contrast between the exposed and the unexposed portion of the specimen is equal to the grades on the grey scale, for assessing change in colour. Change in colour was assessed by comparing the tested cotton fabric under a white light with standard as reference.

RESULTS AND DISCUSSION

Phytochemicals are naturally occurring in the plants that have defense mechanism and used as protection from various ailments. The important phytochemical groups alkaloids, steroids, flavonoid, phenolic compounds, anthraquinones, and tannins present in various plant extracts are responsible for various colour. Organic pigments are large and often complex organic molecules responsible for the different colours of plants and foods. Besides giving the vegetable their characteristic colour, they are also responsible for critical plant functions. The different variations of colours are due to combinations of pigments (Gupta et al., 2013). In the present study was carried out on the *Tagetes erecta* flower revealed the presence of medicinally active constituents. The phytochemical characters of the *Tagetes erecta* flower investigated and summarized in Table-1. The phytochemical screening of *Tagetes erecta* flower aqueous extract exhibits on saponins, flavonoids, steroids, terpenoids, triterpenoids, antroquinone, polyphenol, glycoside, tannins, alkaloids and coumarins. The attractive colours and fragrance produced by the plants is due to specific phytochemicals present in them.

Table.1: Qualitative analysis of Phytochemicals in *Tagetes erecta* flower extract

S. No	Phytochemicals	Aqueous extract
1	Tannin	+
2	Saponin	+
3	Flavonoids	+
4	Steroids	+
5	Terpenoids	+
6	Triterpenoids	+
7	Alkaloids	++
8	Anthroquinone	+
9	Polyphenol	+
10	Glycoside	+
11	Coumarins	+

(+) Presence, (++) High concentrations and (–) Absences

Natural products such as plant extracts provide unlimited opportunities for new drug discoveries because of unmatched availability of chemical diversity, either as pure compounds or as standardized extracts (Sasidharan *et al.*, 2011). These medicinal plants can be rich in phenolic compounds, alkaloids, diterpenoid, steroid and other compounds which inhibit the development of various microorganisms (Ranjitham *et al.*, 2013).

Natural Dye extractions from *Tagetes erecta* flower extract and Pre Mordant, Simultaneous mordanting and Post Mordant Application to cotton fabric.

Aqueous Extract of *Tagetes erecta* flowers were found to discharge colour in hot water very easily. Increasing the quantity of flowers 5 g to 20 g per 100 ml water boiled for 1 hour is accompanied with the increase in colour strength and depth in colour. It was observed that, colour of the dye extract was yellowish pink colour as shown in Figure 1.

Fig.1: Prepared dye from *Tagetes erecta* flower using distilled water

Application of different mordants as PM, SM, and POM on cotton fabric

Mordant can help the dyestuffs achieve a strong and bright colour on cellulose fibers. They combine with the dyestuff and are then permanently fixed onto the fibre. Intensity of the hue and the fastness of the resultant colour can be improved (Dalby, 1992). However, mordants have their own colours which may affect the colour of the dyed textile. In addition, the mordants combined with the dyeing molecules lead to a significant influence on the hue produced with a particular dyestuff (Horrocks & Anand, 2000).

A natural dye is obtained from *Tagetes erecta* flower that produce yellow colors. In the present study the important mordants used are ferrous sulphate, copper sulphate and stannous chloride. The strength of color depends upon the use of pre-mordant, simultaneous mordant and post-mardant and these are metal sources to form a coordinate bond with dye and cotton fabric (Table 2 to 6). From the results, it was observed that *Tagetes erecta* flower showed better colour strength values. In all the three dyeing methods, pre-mordantning method gave excellent results as compared with normal cotton fabric. In all the three methods of dyeing, the mordants copper sulphate, ferrous sulphate and stannous chloride showed excellent colour strength values.

Table.2: Colour produced by different mordants as PM, SM, and POM on cotton fabric by conventional method, dyed with flower extract of *Tagetes erecta*

Chemical mordant	Colour shades obtained in cotton fabric			
	Pre-mordant (PM)	Simultaneous mordant (SM)	Post-mordant (POM)	Normal (Without mordant)
$CuSO_4$	 PMS 132	 PMS 129	 PMS 146	 PMS 105
$FeSO_4$	 PMS 133	 PMS 103	 PMS 5815	
$SnCl_2$	 PMS 123	 PMS 127	 PMS 1215	

Light fastness

Present study light fastness of cotton fabrics dye was measured light fastness grade method followed and represent in table 3. Present study light fastness of cotton fabric dye was measured light fastness grade method and represent in table 4. In Pre-mordant the light fastness grade was 7 indicating as very slight fading, light fastness grade was 6 indicating as slight fading in Simultaneous mordant while light fastness grade was 6 indicating slight fading in post mordant for chemical mordant as $CuSO_4$ when compared to normal (Without mordant) light fastness grade was 3 indicating as Significant fading..

In Pre-mordant the light fastness grade was 6 indicating as slight fading, light fastness grade was 4 indicating as appreciable fading in Simultaneous mordant while light fastness grade was 7 indicating very slight fading in post mordant for chemical mordant as $FeSO_4$ when compared to normal (Without mordant) light fastness grade was 3 indicating as Significant fading..

In Pre-mordant the light fastness grade was 4 indicating as appreciable fading, light fastness grade was 7 indicating as very slight fading in Simultaneous mordant while light fastness grade was 4 indicating appreciable fading in post mordant for chemical mordant as $SnCl_2$ when compared to normal (Without mordant) light fastness grade was 3 indicating as significant fading.

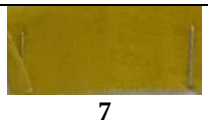
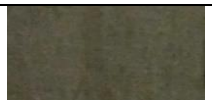
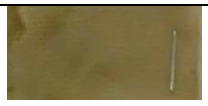
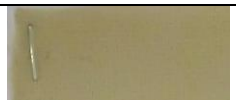
Among the various mordant, pre-mordant has prevent the light fastness followed by post and simultaneous mordant. The stannous chloride mordant has retained the colour as compared to copper and ferrous sulphate chemical mordant.

Metallic mordants were used the complex formed with transition metal protects the chromophore from photolytic degradation. The chromophoric group absorb the photons which by resonating within six- member ring dissipate their energy thereby protects the dyes (Padma et al., 2008). Hence the metal mordant stannous chloride used in the present study protects the dye from photolytic degradation, thereby giving excellent light fastness than copper and ferrous sulphate mordant. In the light fastness result shows that the cotton fabric have a higher light fastness characteristics might be due to the chemical structure of the colour and because the resistance of a dye or pigment to chemical or photochemical attack is directly related to its chemical structure. Dyes with large chemical structure exhibit higher light (Yang & Edward 1996).

Table.3: The Light Fastness Grades

Grade	Degree of fading	Light Fastness type
8	No fading	Outstanding
7	Very slight fading	Excellent
6	Slight fading	Very good
5	Moderate fading	Good
4	Appreciable fading	Moderate
3	Significant fading	Fair
2	Extensive fading	Poor
1	Very Extensive fading	Very poor

Table.4: Colour lightness after PM, SM, and POM on cotton fabric

Chemical mordant	Light fastness grades			
	Pre-mordant	Simultaneous mordant	Post-mordant	Normal dye
$CuSO_4$	 7	 6	 6	 3
$FeSO_4$	 6	 4	 7	
$SnCl_2$	 4	 7	 4	

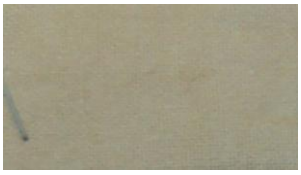
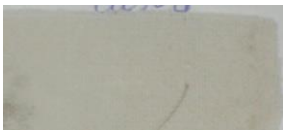
Wash Fastness

Table 5 shows the wash fastness after PM, SM, and POM on cotton fabric. Present study wash fastness of cotton fabric dye was measured wash fastness grade as excellent very good, good, moderate and poor. In Pre-mordant the wash fastness grade was 2 indicating as moderate, wash fastness grade was 3 indicating as good in simultaneous mordant while wash fastness grade was 2 indicating moderate in post mordant for chemical mordant as $CuSO_4$ when compared to normal (Without mordant) wash fastness grade was 2 indicating as moderate fastness..

In Pre-mordant the wash fastness grade was 3 indicating as good, wash fastness grade was 2 indicating as moderate in Simultaneous mordant while wash fastness grade was 3 indicating good in post mordant for chemical mordant as $FeSO_4$ when compared to normal (Without mordant) light fastness grade was 2 indicating as moderat. In Pre-mordant the wash fastness grade was 2 indicating as moderate, wash fastness grade was 2 indicating as moderate in simultaneous mordant while wash fastness grade was 2 indicating moderate in post mordant for chemical mordant as $SnCl_2$ when compared to normal (Without mordant) wash fastness grade was 2 indicating as moderate.. Among the various mordant, post and pre-mordant has prevent the wash fastness followed by simultaneous mordant. The ferrous sulphate mordant has wash fastness the colour as compared to copper and stannous chloride chemical mordant.

The washing solution influences the relation between dye removal and dye mordant nature. If the number of groups which is capable of forming hydrogen bonding and metal complex is higher, the magnitude of dye removal will be lower (Ali and El-Mohamedy, 2011). Similarly the cotton fabric pre-mordanted with $FeSO_4$ have given relatively very good wash fastness.

Table.5: Wash fastness after PM, SM, and POM on cotton fabric

Chemical mordant	Wash fastness grades			
	Pre-mordant	Simultaneous mordant	Post-mordant	Normal
$CuSO_4$	 2	 3	 2	 2
$FeSO_4$	 3	 2	 3	
$SnCl_2$	 2	 2	 2	

KEYS: 5 = EXCELLENT, 4 = VERY GOOD, 3 = GOOD, 2 = MODERATE, 1 = POOR

Pantone Matching System (PMS)

The Pantone Matching System (PMS) has become the leading color reference system for "selecting, specifying, matching and controlling ink color" in the graphic arts and printing industries. With their forever-expanding variety of specialized colors, Pantone has created multiple color systems and guides that all types of designers look to when wanting to create a uniquely colored piece. When much time and effort is put into designing something that includes specific Pantone colors, designers would expect the final printed product to be accurately reproduced. When digitally printed, the file that includes the Pantone colors must go through a raster image processor (RIP) that interprets the colors and is then printed with the use of cyan, magenta, yellow and black toner (Pantone, 2008). Table 6 shows the dyed cotton fabric were compared with reference standard Pantone matching system (PMS).

Table.6: The dyed cotton fabric with Pantone matching system (PMS)

Chemical mordant	Colour shades obtained in cotton fabric			
	Pre-mordant	Simultaneous mordant	Post-mordant	Normal
Colour lightness				
$CuSO_4$	PMS 123	PMS 124	PMS 1405	PMS 105
$FeSO_4$	PMS 1265	PMS 147	PMS 103	
$SnCl_2$	PMS 1255	PMS 100	PMS 113	
Wash fastness				
$CuSO_4$	PMS 128	PMS 1252	PMS 117	PMS 121
$FeSO_4$	PMS 1265	PMS 1215	PMS 147	
$SnCl_2$	PMS 1255	PMS 1205	PMS 120	

CONCLUSION

Overall, it can be concluded that the dye was prepared from *Tagetes erecta* flower extract has possesses potential dyeing capability to cotton fabric. The ferrous sulphate is a best dyeing mordant than the $SnCl_2$ and $CuSO_4$ mardants.

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