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EXTRACTION OF NATURAL DYE FROM *Nerium oleander* Linn. FLOWERS AND EVALUATION OF CRAFT AS ICE STICKS DYEING USING DIFFERENT CHEMICAL MORDANTS

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ABSTRACT

The phytochemical screening *Nerium oleander* flower extract showed tannin, saponins, flavonoids, steroids, terpenoids, triterpenoids, antroquinone, polyphenol, glycoside, alkaloids and coumarins present in aqueous *Nerium oleander* flower extract. The prepared colour of the dye extract was found to be in dark blue colour, among the three mordants method, post-mordantning method gave excellent results as compared with normal craft. Among the three mordants of dyeing, the ferrous sulphate mordants showed excellent colour strength values as compared with stannous chloride 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 mordant. In wash fastness, simultaneous and pre-mordant has prevent the light fastness followed by pre 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. The dye was prepared from *Nerium oleander* flower extract and possesses potential dyeing capability to craft as ice sticks. The ferrous sulphate has important dyeing mordant than other mordants used in this study.

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INTRODUCTION

Natural dyes are known for their use in colouring of food substrate, leather, wood as well as natural fibers like wool, silk, cotton and flax as major areas of application since ancient times. Natural dyes may have a wide range of shades, and can be obtained from various parts of plants including roots, bark, leaves, flowers, and fruit (Allen, 1971). Since the advent of widely available and cheaper synthetic dyes in 1856 having moderate to excellent colour fastness properties, the use of natural dyes having poor to moderate wash and light fastness has declined to a great extent. However, recently there has been revival of the growing interest on the application of natural dyes on natural fibers due to worldwide environmental consciousness (Samanta and Agarwal, 2009). The

present study is to investigate the phytochemical and Natural Dye extractions from *Nerium oleander* flower extract.

MATERIALS AND METHODS

Collection of plant materials

The *Nerium oleander* flowers were collected from the market "Poosanthai" 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.

Preparation of flower extract

One gram of *Nerium oleander* flowers were taken and made a fine powder, powder was added in 50 ml of aqueous solvent, the extract was shaken it well for 30 minutes by free hand and wait for 24 hours. After the extracts were filtered using whatman filter paper No.1 and 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 Dyes from *Nerium oleander* flowers extract

Material

Craft as Ice sticks 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 crafts is kept in the dye bath in short time and dark shade kept it for overnight and change the proportion.

Extraction of Dye

The flowers of *Nerium oleander* were cut into small pieces and put into distilled water and heated in a water bath for one hour and filter the extract. These extract were used for dyeing crafts. All materials were 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 Crafts 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

Crafts 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

Crafts was bleached and dyed with dye extract at 70-80 0C.with a half 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 craft form a piece measuring 10cm by 4cm was cut from each of the craft. The pieces of the undyed ice sticks enable the degree of staining during test to the 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 craft under a white light with e standard as reference.

RESULTS AND DISCUSSION

Qualitative analysis of Nerium oleander flower extract

In the present study was carried out on the Nerium oleander flower revealed the presence of medicinally active constituents. The phytochemical characters of the Nerium oleander flower investigated and summarized in Table-1 and figure 3. The phytochemical screening Nerium oleander flower carried on tannin, saponins, flavonoids, steroids, terpenoids, triterpenoids, antroquinone, polyphenol, glycoside, alkaloids and coumarins presence in aqueous Nerium oleander flower extract. The attractive colours and fragrance produced by the plants is due to specific phytochemicals present in them.

Table.1: Qualitative analysis of Phytochemicals in *Nerium oleander* flower extract

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

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

Gupta et al. (2013) screened Qualitative and Quantitative Analysis of phytochemicals and pharmacological value of some dye yielding medicinal plants. The qualitative analysis carried out for Emblica officinalis, Acacia catechu, Acacia concina and Hibiscus rosa-sinensis showed that tannins, saponins, flavonoids, terpenoids and alkaloids are present in all the plants except phlobatannins that is only present in Acacia catechu. The pet-ether and chloroform extract of Emblica officinalis does not show potential for oil and fat components where as all the extract of Emblica officinalis showed positive test for carbohydrates.

Various organic 11 solvent extracts of Pedalium murex were subjected to preliminary phytochemical screenings by Thamizh mozhi et al. (2011). Selected 53 traditionally used medicinal plants from western region of India for their qualitative phytochemical screenings, total phenol and flavonoids contents. Pascaline et al. (2011) screened phytochemical constituents of some medicinal plants used by the Nandis of South Nandi District, Kenya.

Natural Dye extractions from Nerium oleander flower extract and Pre Mordant, Simultaneous mordanting and Post Mordant Application to Crafts as Ice sticks.

Aqueous Extract of Nerium oleander 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 dark blue colour as shown in Figure 4

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Fig.4: Prepared dye from *Nerium oleander* flower using distilled water

Application of different mordants as PM, SM, and POM on craft as Ice sticks

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).

Mordants helps in absorption and fixation of natural dyes and also prevents bleeding and fading of colours i.e., improves the fastness properties of the dyed fabrics. The majority of natural dyes, whether chemical dyes or natural dyes, adhere to the fiber through a chemical bond. With straight dye and fiber, this bond is easy to degrade and break. However, some compounds can cause the dye to adhere to the fiber. These compounds are called mordants, and are usually metal salts. Different types of mordants yield different colours even for the same natural dye (Vankar, 2009).

A natural dye is obtained from *Nerium oleander* flower that produce blue colors. In the present study the important mordants used are ferrous sulphate, copper sulphate and stannours 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 craft (Table 2 to 6). From the results, it was observed that *Nerium oleander* flower showed better colour strength values. In all the three dyeing methods, post-mordantning method gave excellent results as compared with normal craft. In all the three methods of dyeing, the mordants copper sulphate, ferrous sulphate and stannous chloride show excellent colour strength values.

Light fastness

Table.3 shows the Light Fastness Grades and compared with the fastness of craft. Present study light fastness of crafts dye was measured light fastness grade method and represent in table 4. 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 $CuSO_4$ when compared to normal (Without mordant) light fastness grade was 6 indicating as Slight fading.

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 7 indicating very slight fading in post mordant for chemical mordant as $FeSO_4$ when compared to normal (Without mordant) light fastness grade was 6 indicating as Slight fading

In Pre-mordant the light fastness grade was 6 indicating as slight fading, light fastness grade was 7 indicating as very slight fading in Simultaneous mordant while light fastness grade was 6 indicating slight fading in post mordant for chemical mordant as $SnCl_2$ when compared to normal (Without mordant) light fastness grade was 6 indicating as Slight 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.

The variability of the emission from light sources, both natural and artificial, and the variability of exposure conditions (e.g temperature, humidity) in the case of daylight makes it difficult to know the exact conditions of exposure and to reproduce them precisely, unlike in the case of other fastness determinations (e.g. washing, etc.). Hence the grey scales cannot be used as standards of reference. Rather, standard dyed material of known light fastness exposed alongside the specimens under test serve as scales for the assessment of light fastness.

In the light fastness result shows that the craft 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 craft as Ice sticks

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

Table.2: Colour produced by different mordants as PM, SM, and POM on craft as Ice sticks by conventional method, dyed with flower extract of *Nerium oleander*

Chemical mordant	Colour shades obtained in craft as ice stick			
	Pre-mordant (PM)	Simultaneous mordant (SM)	Post-mordant (POM)	Normal (Without mordant)
$CuSO_4$	 PMS 488	 PMS 407	 PMS 410	 PMS 4685
$FeSO_4$	 PMS 437	 BLACK	 PMS 419	
$SnCl_2$	 PMS 469	 PMS 4655	 PMS 4665	

Table.5: Wash fastness after PM, SM, and POM on craft as Ice sticks

Chemical mordant	Wash fastness grades			
	Pre-mordant	Simultaneous mordant	Post-mordant	Normal
$CuSO_4$	 3	 4	 4	 2
$FeSO_4$	 3	 3	 4	
$SnCl_2$	 4	 4	 3	

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

Wash Fastness

Table 5 shows the wash fastness of after PM, SM, and POM on craft as Ice sticks. Present study light fastness of crafts dye was measured wash fastness grade as excellent very good, good, moderate and poor. In Pre-mordant the wash fastness grade was 3 indicating as good, light fastness grade was 4 indicating as very good in Simultaneous mordant while wash fastness grade was 4 indicating very good 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 3 indicating as good in Simultaneous mordant while wash fastness grade was 4 indicating very good in post mordant for chemical mordant as $FeSO_4$ when compared to normal (Without mordant) wash fastness grade was 2 indicating as moderat..

In Pre-mordant the wash fastness grade was 4 indicating as very good, wash fastness grade was 4 indicating as very good in Simultaneous mordant while wash fastness grade was 3 indicating good 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, simultaneous and pre-mordant has prevent the wash fastness followed by pre 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 ice sticks post-mordanted with ferrous sulphate have given relatively very good wash fastness.

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).

The PMS consists of thousands of unique color mixtures and is separated into different types of categories for specific purposes and usages. There are systems dedicated strictly for the graphic arts, including printing and publishing, clothing, home furnishing and interior decorating, paints and plastics Sharma, (2008). The Pantone Matching System (PMS) used as color reference system to monitor the colour and observed the codes were different for various mordant. Table 6 shows the dyed Craft as Ice sticks were compared with reference standard Pantone matching system (PMS).

Table.6: The dyed Craft as Ice sticks with Pantone matching system (PMS)

Chemical mordant	Colour shades obtained in crafts as ice stick			
	Pre-mordant	Simultaneous mordant	Post-mordant	Normal
Colour lightness				
$CuSO_4$	PMS-461	PMS-393	PMS-5835	PMS-460
$FeSO_4$	PMS-141	PMS-Black 7	PMS-462	
$SnCl_2$	PMS-608	PMS-1205	PMS-600	
Wash fastness				
$CuSO_4$	PMS-453	PMS-617	PMS-5875	PMS-127
$FeSO_4$	PMS-466	PMS-451	PMS-582	
$SnCl_2$	PMS-601	PMS-608	PMS-459	

Natural dyes are non-toxic, non-allergic and biodegradable hence natural dye used in dyeing various cloths. Metal salts were used as mordants. A naturally dyed textile cloth product not fully environmental friendly. We have investigated bark, flowers, leaves and fruit extracts for dyeing cotton fabric, selected metal salts being used as a mordant. The extracted natural dye was examined for pre, simultaneous and post mordant with metal salts (Bajirao Ahire, 2018).

CONCLUSION

The dye was prepared from *Nerium oleander* flower extract and possesses potential dyeing capability to craft as ice sticks. The ferrous sulphate has important dyeing mordant than other mordants used in this study.

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