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EXTRACTION OF NATURAL DYE FROM *Ixora coccinea* LINN. FLOWERS AND EVALUATION OF COSMETICS AS FACE POWDER DYEING USING DIFFERENT CHEMICAL MORDANTS

P. Narmatha and Dr. R. Sagaya Giri*

ABSTRACT

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. In the present study is to investigate the phytochemical screening on saponins, flavonoids, steroids, terpenoids, triterpenoids, antroquinone, polyphenol, glycoside and coumarins was percent in *Ixora coccinea* flower aqueous extract. The prepared cooler of the dye extract was found to be in magenta red colour. Among the three mordants method, pre-mordantning method gave excellent results as compared with normal cosmetics as face powder. Among the three mordants of dyeing, the ferrous sulphate mordants show excellent colour strength values as compared with $SnCl_2$ 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, 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.

Keywords: *Ixora coccinea* flowers, Natural dye, chemical mordants

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*Corresponding author:
Dr. R. Sagaya Giri,
Assistant Professor P.G. and research department of botany Kunthavai Naacchiyaar Government arts college for women (autonomous), Thanjavur - 613 007, Tamil nadu, India.

INTRODUCTION

Colour is one of the elements of nature that has made human living more aesthetic and fascinating in the world. Natural colors are always eco-friendly applications in various fields for mankind. India has a very rich biodiversity so there are more than 600 plants yielding various pigments, colors and dyes (Mahesh et al., 2011). 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).

Dyeing was practiced as early as 3000 BC in China and Egypt. The earliest records of Indian textiles belong to the period of about 2500 BC and they contained references of coloured silk and gold brocades. By about 1450 BC the Egyptians were making textile material of astonishing delicate structure and were able to dye them in a whole range of colours (Trotman, 1975). Natural dyes comprise those colourants (dyes and pigments) that are obtained from animal or vegetable matter without chemical processing (Gulrajani & Gupta, 1992). The word 'natural dye' covers all the dyes derived from the natural sources like plants, animal and minerals. Natural dyes are mostly nonsubstantive 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. The present study is to investigate the phytochemical screening and natural dye extractions from *Ixora coccinea* flower extract applied to Cosmetics as face powder.

MATERIALS AND METHOD

Collection of plant materials

The *Ixora coccinea* flowers were collected from 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.

Preliminary Phytochemical analysis

Preparation of flower extract

Take one gram of *Ixora coccinea* flowers powder was taken and dissolved and in 50 ml of aqueous solvent, the extract was shaken it well for 30 minutes by free hand and wait for 24 hours. After that extracts were filtered using whatman filter paper No.1 and 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 Dyes from *Ixora coccinea* flowers extract

Material

Cosmetics as face powder were purchased from local market at Thanjavur. It was used after bleaching for application.

Experimental methods

$CuSO_4$, $FeSO_4$, $SnCl_2$ used as mordants. For light shade, the Cosmetics 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 *Ixora coccinea* 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 Cosmeticss. 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 Cosmetics 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

Cosmetics 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

Cosmetics was bleached and dyed with dye extract at 70-80 0C.with a half on 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 cosmetics as powder (2gm sample). The pieces of the undyed cloth 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 cosmetics under a white light with standard as reference.

RESULTS AND DISCUSSION

The important phytochemical groups alkaloids, steroids, flavonoid, phenolic compounds, anthraquinones, and tannins present in various plant extracts are responsible for various colour. In the present study, *Ixora coccinea* flower extract revealed the presence of medicinally active constituents. Such as saponins, flavonoids, steroids, terpenoids, triterpenoids, antroquinone, polyphenol, glycoside and coumarins presence in aqueous *Ixora coccinea* flower extract while tannin and alkaloids were absent (Table 1). The attractive colours and fragrance produced by the plants is due to specific phytochemicals present in them. Secondary metabolites are reported to have many biological and therapeutic properties. Pharmacists are interested in these compounds because of their therapeutic performance and low toxicity (Inayatullah *et al*., 2012).

Table.1: Qualitative analysis of Phytochemicals in *Ixora coccinea* 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

Various organic 11 solvent extracts of *Pedalium murex* were subjected to preliminary phytochemical screenings by Thamizh mozhi *et al.* (2011). Flavonoids are a group of polyphenolic compounds with known properties which include free radical scavenging, inhibition of hydrolytic and oxidative enzymes and anti-inflammatory action. Flavonoids are 15 carbon compounds generally distributed throughout the plant kingdoms. Some isoflavones widely used in insecticides. They might also play a role in disease resistance. Some flavonoids such as quercetin and rutin, are known to support human health by serving anti-inflamatory, anti-histaminic and anti-viral agents (Okwu, 2004). Anthroquinones possess antiparastic, bacteriostatic, antidepressant and antimicrobial and antioxidant activities. Their potential effects against cancer through different mechanisms have been studied (Agoha, 1974). Alkaloids have been indicated as a starting material in the manufacture of steroidal drug. Pure isolated alkaloids and their synthetic derivatives are used as basic medicinal agents for their analgesic, antispasmodic and bactericidal effect (Okwu, 2004). Tannins are phenolic phytochemicals, which are natural constituents of green tea, are considered to have cancer preventive properties (Niraimathi *et al.*, 2012).

Natural Dye extractions from *Ixora coccinea* flower extract and Pre Mordant, Simultaneous mordanting and Post Mordant Application to Cosmetics as face powder.

Aqueous Extract of *Ixora coccinea* 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 Magenta red colour as shown in Figure 1.

Fig.1: Prepared dye from *Ixora coccinea* flower using distilled water

Application of different mordants as PM, SM, and POM on Cosmetics as face powder

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 and 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. 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 *Ixora coccinea* flower that produce Magenta red 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 Cosmetics (Table 2 to 6). From the results, it was observed that *Ixora coccinea* flower showed better colour strength values. In all the three dyeing methods, pre-mordantning method gave excellent results as compared with normal Cosmetics. In all the three methods of dyeing, the mordants copper sulphate, ferrous sulphate and stannous chloride showed excellent colour strength values. Among the various mordants, ferrous sulphate possess potential colouring dye.

Table.2: Colour produced by different mordants as PM, SM, and POM on Cosmetics as face powder by conventional method, dyed with flower extract of *Ixora coccinea*

Chemical mordant	Colour shades obtained in Cosmetics as face powder			
	Pre-mordant (PM)	Simultaneous mordant (SM)	Post-mordant (POM)	Normal (Without mordant)
$CuSO_4$	 PMS 400	 PMS 663	 PMS 538	 PMS 656
$FeSO_4$	 PMS 5507	 PMS 5295	 PMS 5455	
$SnCl_2$	 PMS 422	 PMS 627	 PMS 663	

Light fastness

Present study light fastness of Cosmetics dye was measured light fastness grade method followed and represent in table 4. In Pre-mordant the light fastness grade was 7 indicating as very slight fading, light fastness grade was 5 indicating as Moderate 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 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 7 indicating very 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. 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 Cosmetics 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

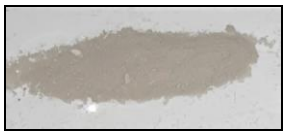
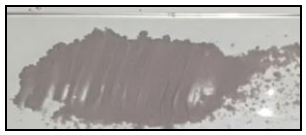
Table.4: Colour lightness after PM, SM, and POM on Cosmetics as face powder

Chemical mordant	Light fastness grades			
	Pre-mordant	Simultaneous mordant	Post-mordant	Normal
$CuSO_4$	 **7**	 **5**	 **6**	 **6**
$FeSO_4$	 **7**	 **6**	 **7**	
$SnCl_2$	 **6**	 **7**	 **7**	

Wash Fastness

Table 5 showed the wash fastness after PM, SM, and POM on Cosmetics as face powder. Present study light fastness of Cosmetics dye was measured wash fastness grade as excellent very good, good, moderate and poor. In Pre-mordant the wash fastness grade was 4 indicating as very 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 $CuSO_4$ 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 mordant. In Pre-mordant the wash fastness grade was 3 indicating as 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, 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 cosmetics as powder post-mordanted with ferrous sulphate have given relatively very good wash fastness.

Table.5: Wash fastness after PM, SM, and POM on Cosmetics as face powder

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

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. 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). Table 6 shows the dyed Cosmetics as face powder were compared with reference standard Pantone matching system (PMS). 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: The dyed Cosmetics as face powder with Pantone matching system (PMS)

Chemical mordant	Colour shades obtained in Cosmeticss as face powder			
	Pre-mordant	Simultaneous mordant	Post-mordant	Normal
Colour lightness				
$CuSO_4$	PMS - 602	PMS - 615	PMS - 5807	PMS - 614
$FeSO_4$	PMS - 5875	PMS - 5865	PMS - 5797	
$SnCl_2$	PMS - 607	PMS - 616	PMS - 586	
Wash fastness				
$CuSO_4$	PMS - 461	PMS - 609	PMS - 581	PMS - 600
$FeSO_4$	PMS - 5787	PMS - 5875	PMS - 5865	
$SnCl_2$	PMS - 587	PMS - 602	PMS - 600	

CONCLUSION

The dye was prepared from *Ixora coccinea* flower extract and possesses potential dyeing capability to cosmetics as face powder. The ferrous sulphate has important dyeing mordant than other mordants used in this study.

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