

## EXTRACTION, OPTIMISATION AND DYEING OF SILK YARN USING NATURAL DYE FROM *COSMOS* SP

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### ABSTRACT

The use of natural dye decreased in last few decades due to the discovery of synthetic dye in nineteenth century. Recently, non toxic, environment friendly natural dye started receiving much attention in order to avoid hazardous petrochemical raw materials which involve in synthetic dye production and also its non biodegradable effluent. In this study, extraction of a natural dye from *Cosmos* sp flower was optimised. Pretreated silk yarn was dyed with aqueous extract of the dye. Dyeing was carried out through pre, meta and post mordanting method with the help of permissible chemical mordants –Alum and Ferrous sulphate. Further, these mordants were able to develop different shades of hue with good fastness property. Standardised dyeing protocol was fixed on the basis of different parameters like time, temperature, pH and material to liquor ratio. Extracted dye was also evaluated to determine the principal compound responsible for dyeing the yarn. Preliminary analysis showed the presence of flavonoid in the dye extract. These results revealed extract of *Cosmos* sp can be a potent source of natural dye in textile industry.

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## INTRODUCTION

Natural dyes are colourants which can be derived from plants, minerals and animals; capable to dye other substances such as textile material, leather, food, medicine etc. This technique of dyeing was practiced by ancient people before thousands of years (Geelani *et al.*, 2015; Prabhu and Bhute, 2012). Ajanta, Ellora, Mithila wall painting, Egyptian pyramids, Spanish caves of Altamira, French Pyrene caves of Niaux are beautiful evidence of this ancient art (Prabhu and Bhute, 2012; Valladas *et al.*, 1992). The different sources of natural colourant has been found mentioned in Vedas and Bible (Ado *et al.*, 2014). Since accidental discovery of synthetic dye in 19<sup>th</sup> century, use of natural dyes in textile industries started decreasing due to its drawbacks like less availability, low dye uptake and low fastness properties. On other hand, synthetic dye replaced the natural dye market drastically due to its cheaper cost, wide

range of colours and good fastness properties (Savarino *et al.*, 1999; Paisan *et al.*, 2002). The synthetic textile dyes considered as the most harmful pollutant of all the industrial sectors. Synthetic dye production involves many hazardous chemicals which is now serious threat to eco system and human health. Textile consumption is around 30 million globally where synthetic dye production is  $7 \times 10^5$  annually (Rajendran and Tharamani, 2014; Ogugbue and Sawidis, 2011). Due to this huge amount of synthetic dye production, its non biodegradable effluent is now a major pollutant to environment (Geelani *et al.*, 2016; Aminoddin and Haji, 2010; Arun and Yogamoorthi, 2014). Recently a ban on synthetic dye use has been imposed by European Economic Community, Germany, USA and India to protect the eco system from harmful, carcinogenic chemicals (Prabhu and Bhute, 2012). As a result natural dye started regaining interest in textile industries for its non toxic, environment friendly nature (Yusuf *et al.*, 2016).

India is a potent land to produce natural dyes industrially, as India is considered as one of the seventeen mega diversity countries globally (Singh and Srivastava, 2015). In recent past considerable research work started to explore new sources to fulfill demands of natural dyes in industrial level with low cost, more dye exhaustion quality and better fastness properties (Geelani *et al.*, 2015). Natural dyes are mostly substantive dye which requires mordants to prevent fading off the colour from the yarn due to wash and exposure to light (Siva, 2007). Mordant is a chemical which can successfully bind to the yarn and also make a ligand with natural dye to the yarn. The word "Mordant" has been derived from latin word "modere" meaning "to bite" (Prabhu and Bhute, 2012). Mostly natural salts are used as mordants due to its easy availability. Common metal mordants are alum, ferrous sulphate, copper sulphate etc (Geelani *et al.*, 2016). Metal mordants are used as electron acceptor which makes coordinate bond with electron donor dye molecule to make water insoluble compound (Lee and Kim, 2004).

However, different mordants can produce different shades with same dye (Ado *et al.*, 2014). Natural fibres can successfully bond with natural dye. Mainly silk, wool, alpaca from animal origin and cotton, jute, linen from plant origin are considered as natural fibres. Animal fibres are protein based fibres which have strong affinity towards natural dye than plant based fibres (Ado *et al.*, 2014). Depending on different sources and different fabrics, method of applying dye varies which can be optimised through screening (Ado *et al.*, 2014). *Cosmos sp* is also known as sulphur cosmos/ orange cosmos/ yellow cosmos which belong to Asteraceae family. This plant is native of Mexico and widely spread in tropical and sub tropical region like Africa, Asia. They are annual herb with simple or pinnately divided leaves, large and long stock daisy like flower, grows in summer. The petals of the plant are bright yellow and orange in colour. If natural dye can be classified on the basis of its chemical structure, most of the yellow dye can be categorised under hydroxy and methoxy derivatives of flavones and isoflavones (Siva, 2007). The aim of this study is to extract textile dye from *Cosmos sp* flower and developing colour shades with different mordants. Further, evaluation of light, wash and perspiration properties of extracted dyes has been studied. The chemical nature of the dye was analysed to identify principal pigment present in it.

## MATERIALS AND METHODS

### Collection of plant material

The flower of *Cosmos sp* was used to extract the dye. Flowers were collected from Horticulture, Chennai.

### Collection of yarn

Silk yarn was purchased from TANSILK, Salem, Tamil Nadu.

### Optimisation of extraction of dye

In this study, extraction from the flower was done using water to avoid toxicity of harmful solvents. The extraction procedure was optimised on the basis of different parameters such as pH (5, 7, 9), Temperature (40°C, 50°C, 60°C, 70°C) and time (15 min, 30 min, 45min, 60min). Fresh flower of *Cosmos sp* were collected. Further, it was ground with distilled water and maintained different time, temperature and pH to extract dye. Sample to Liquor ratio of the extract 1:20 was maintained.

On comparison with standard calibration curve optimum pH, temperature and time were fixed for further analysis.

### Pre-treatment of Silk Yarn

Dirt and impurities like gum (serenin) of the silk yarn should be removed to increase the dye uptake. The yarn was soaked in distilled water with soft detergent and aqueous *Sapindus mukorossi* fruit extract at room temperature for 60 minute and then washed thoroughly. Then the yarn was ready to use for dyeing.

### Optimisation of Dyeing

The pretreated silk was dyed using *Cosmos sp* flower extract. The dyeing method has been standardised using different parameters such as dyeing time (15min, 30min, 45min, 60min), temperature (40°C, 50°C, 60°C, 70°C), pH (5, 6, 7, 8), and material (yarn) to dye ratio (1:20, 1:40, 1:60). On the basis of the percentage of dye exhaustion, optimum dyeing time, temperature, pH, and material to dye ratio were selected.

Measurement of dye exhaustion:

Percentage of dye exhaustion,

$$\% E = \frac{C_1 - C_2}{C_1} \times 100$$

where,  $C_1$  and  $C_2$  are concentration of dye bath before and after dyeing process respectively. Dye concentration was checked at specific wavelength ( $\lambda$  max) in UV Vis Spectroscopy. Dye exhaustion is the amount of dye which diffused to yarn (Jigar *et al.*, 2013).

### Mordanting

Permissible chemical mordants have been used to improve fastness property of the dyed yarn. In this study, chemical mordant alum and ferrous sulphate were used. Three different mordanting process have been carried out with both chemical mordants, i.e, pre mordanting, meta mordanting and post mordanting.

### Standardisation of Chemical mordant

Different concentration of alum and ferrous sulphate ranging from 1-5% ovy (on weight of yarn) has been screened. Wet yarn sample was brought into mordant solution and the various temperature was maintained at 40°C -100°C for different time between 30min - 1hour for both alum and ferrous sulphate (FeSO<sub>4</sub>). Further, the yarn was squeezed and soaked in dye bath. As few mordants are sensitive to light, therefore yarn has to be immediately dyed after mordanting. Similar process has been followed for pre, meta and post mordanting.

### Colour fastness

Dyed silk yarn was analysed for colour fastness to light (ISO 105 B02-2013), wash (ISO:105 C-06:2010) and perspiration (ISO 105 E04-2013) using standard ISO method.

### Identification of Pigment

#### Phytochemical analysis

Initial analysis of chemical constituents of dye solution was carried out by phytochemical analysis (Raaman, 2006; Divya and Ravi, 2013; Wanyama *et al.*, 2011) following standard procedure.

### Thin layer chromatography

Silica gel coated plate was used to separate pigments. Different solvent systems such as Chloroform: Methanol (96:4); Methanol: Chloroform (9:1); Petroleum benzene: Methanol (5:5); Petroleum ether: Acetone: Chloroform (3:1:1); nButanol : Acetic acid : Water (4:1:5) were screened. Rf value has been calculated.

### UV Visible Spectroscopy

The extract was analysed in UV Visible Spectrophotometer SHIMADZU 1650PC at wavelength of 400-800nm and the corresponding peaks were recorded.

### FTIR Spectroscopy

FT-IR spectral system (Shimadzu, IR Affinity 1, Japan), equipped with a DLATGS detector with a mirror speed of 2.8mm/sec. scan range: from 400-4000 $\text{cm}^{-1}$  with a resolution of 4 $\text{cm}^{-1}$  was used for this analysis. The dried samples were finely grounded using potassium bromide (KBr) in 1:10 ratio. The IR pellet was recorded in the region 4000-400  $\text{cm}^{-1}$ . This range was used to study the fundamental vibration and associated rational vibration structure.

## RESULTS AND DISCUSSION

The initial screening showed the flower of *Cosmos sp* (figure 1) yield orange colour dye (Figure 2). Therefore, detailed study was carried out to standardise the extraction and dyeing of silk yarn with the help of mordants.



Fig. 1. *Cosmos sp*



Fig. 2. *Cosmos sp* flower extract

### Optimisation of extraction

Natural dye can be extracted using different techniques such as aqueous method, super critical fluid method, complicated

solvent system and ultrasonic extract. Among which the extraction with water is the most common, cheap and the oldest method (Prabhu and Bhute, 2012). Aqueous extraction depends on time, temperature, pH and sample to liquor ratio. Therefore, optimum extraction process can be determined by maximum absorbance value at lamda max (Prabhu and Bhute, 2012). Different pH such as 5, 7, 9 has been analysed. Result revealed that pH 5 gave light yellow colour where pH 7 showed dark orange and pH 9 showed orangish brown colour. Extraction of more colouring component in alkaline solvent might be due to ionisation of hydroxyl groups of colour components in alkalinity which increases the solubility of dye component (Tayade and Adivarekar, 2013). Neutral pH has been selected as optimum pH for extraction. Like pH, time and temperature also have significant effect in dye extraction. When temperature and time increases the extract of dye gets darker in colour which ultimately remains unchanged on further increase of time and temperature. In comparison with standard curve 60°C and 45 min has been standardised as best extraction method. Increase flavonoids content extraction with increase in time due to *de novo* formation in the aqueous solvent (Sukrasno *et al.*, 2011). Hence, water soluble orange dye from flower might be a type of flavonoid. The sample to liquor ratio was maintained at 1:20. Optimisation of aqueous extraction of dye has been successfully done by M A Khan *et al.*, (2006), S R Maulik and S C Pradhan (2005).

### Mordanting and Dyeing of Silk Yarn:

Natural dyes mostly need mordants to fix to yarn and to improve fastness. One molecule of yarn bind with one dye molecule, whereas, one mordant molecule can bind with more than one dye molecule which makes mordanted yarn uptake more dye than unmordanted yarn (Bhattacharya and Shah, 2000; Uddin, 2014). Most commonly used less toxic chemical mordants are alum and ferrous sulphate. Saravana and Chandramohan (2011) supported the fact that ferrous sulphate and alum can help natural dye to attach with silk yarn successfully. Different shades of hue can be formed by using different metal salts as mordants due to formation of metal ion complex (Saranya *et al.*, 2011; Ghurde *et al.*, 2016). Ferrous sulphate can increase strong bonds between dye and yarn due to strong coordination tendency of Fe. Therefore, dye exhaustion can be increased by yarn and also chromophore of dye can be protected from photolytic degradation (Jothi, 2008). When Silk yarn interacts with ferrous sulphate, few coordination sites remain free which can be occupied by yarn, so ferrous sulphate can form ternary complex with yarn and dye (Uddin, 2015).

In case of alum, it can form strong bond with dye molecule whereas it forms lesser strong bond with yarn (Cotton and Wilkinson, 1972). In this study, 1% (oww) ferrous sulphate was selected as optimised concentration to fix the dye. The mordanting process was optimised at 60°C for 1 hour. Further, 3% (oww) alum at 80°C for 1 hour was standardised mordanting method. Mordanting was carried out in three process as pre mordanting, meta mordanting and post mordanting, where meta mordanting revealed better result than pre and post mordanting. Similar result was found by Sayed Maqbool Geelani (2015), though experimental setup was different. Silk yarn is polypeptide chain so it contained many active sites to bind with natural dye through different bondings like covalent, hydrogen bonding, hydrophobic interaction (Yusuf, 2016). Finally, anions of dye and cations of metal

mordants show attraction to  $\text{NH}^+$  bonds and  $\text{COOH}^-$  bonds of silk yarns which forms strong ionic bonds between silk, mordants and dye (Uddin, 2015). Therefore, metal mordants can successfully overcome drawbacks of natural dyes.

#### Effect of time on dyeing:

Figure 3 showing effect of time on dye exhaustion percentage. Both the mordants show different results as they form different chemical complex with silk yarn. In case of ferrous sulphate 15min -45min dye uptake increases, following which the uptake was reduced. Hence, 45min was selected as optimum dyeing time, this can be due to disruption of dye at high temperature or silk yarn became saturated with dye molecule (Saranya *et al.*,2011; Haji,2010). Further, for alum, dye exhaustion increased gradually from 15min -1hour ,therefore 1 hour was selected as optimum dyeing time with alum. Similar result was found by M Chairat *et al.*,(2007) and M Srivastava *et al.*, (2015).

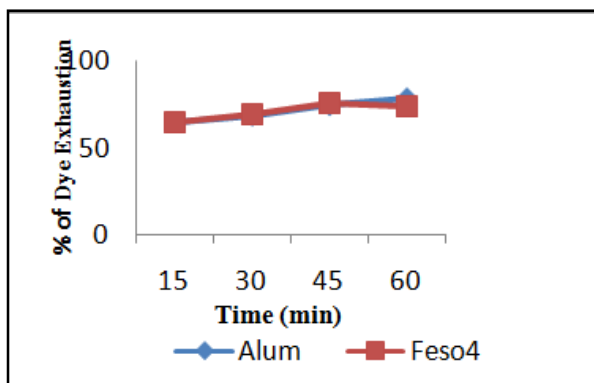


Fig. 3. Effect of Time on dye exhaustion

#### Effect of temperature on dyeing:

Effect of various temperature on percentage of dye exhaustion showed in figure 4 .Dye uptake was found to increases with temperature rise from 40°C-60°C. After which there was a sharp decline of dye uptake percentage for both mordants, alum and ferrous sulphate. Therefore, for both the mordants 60°C was selected as standardised temperature to maximise the percentage of dye exhaustion by silk yarn. This result can be supported by M Chairat *et al.* (2007). With increase in temperature, silk yarn gets swelled, which enhances its dye uptake in higher temperature (Jung and Dae, 2014). After 60°C decrease of dye uptake attributes maybe due to instability of functional groups of dye in high temperature (Saranya *et al.*, 2011).

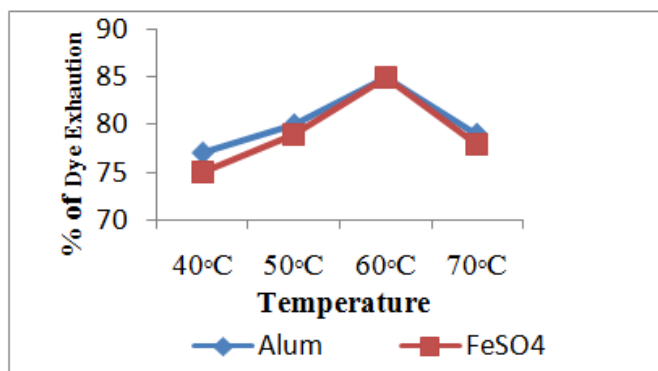


Fig. 4. Effect of Temperature on dye exhaustion

#### Effect of pH on dyeing

Effect of various pH on silk dyeing depicted in figure 5. Dyeing of silk yarn with alum showed optimum dye uptake at pH 7 and with ferrous sulphate pH 6. In this study, dye exhaustion percentage was more in acidic pH where alkaline pH showed drastic decline in dye uptake. Similar results were reported by H T Deo and B K Desai (1999) and Sayed Maqbool Geelani *et al.*,(2016). Further they stated that silk as protein yarn having equal amount of positive and negative group , i.e, amino and carboxyl group respectively. At low pH carboxylic group absorbs hydrogen ion , whereas at high pH protein loses hydrogen ion, so protein yarn shows maximum colour depth in acidic dye bath (Deo and Desai,1999; Geelani *et al.*, 2016).

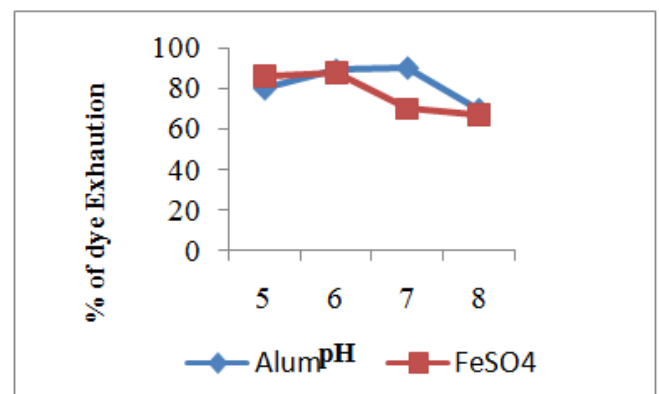


Fig. 5. Effect of pH on dye exhaustion

#### Effect of concentration on dyeing

Figure 6 shows the percentage of dye exhaustion of silk yarn using alum and ferrous sulphate in different material to liquor ratio such as 1:20, 1:40 and 1:60. All three ratios showed good results. There was an increase in dye uptake percentage from 1:20 to 1:40, however, 1:60 shows similar result as 1:40. Therefore 1:40 was selected as optimised material to liquor ratio for both mordants. Similar results were found by Ado A *et al.*, (2014). Dye uptake increases with increase of dye concentration as concentration gradient increase with increase of dye concentration in dye bath (Shabbir *et al.*,2016) . After a certain concentration dyed yarn reaches to equilibrium after that dye uptake does not increase with increase of dye concentration (Saranya *et al.*,2011) .

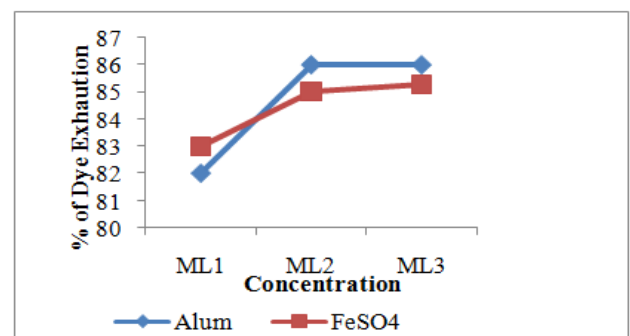


Fig. 6. Effect of concentration on dye exhaustion

#### Fastness Properties

Fastness properties of dyed yarn with both alum (Fig 7) and ferrous sulphate (Fig 8) have been tested through standard

methods in laboratory. Further, depending on best dye uptake and fastness properties, dyed silk yarn treated with alum was tested using standard ISO method. Light, wash and perspiration fastness was analysed with respect to grey scale. Light and perspiration fastness showed better result than wash fastness. Both perspiration (acidic and alkaline) and light showed fair to good(3-4) result. Result shows mordants not only help the yarn to uptake more dye, but also forms a water insoluble metal-dye complex with yarn which improves fastness properties (Tayade and Adivarekar,2013).



Fig. 7. Dyeing with *Cosmos sp* flower with alum



Fig. 8. Dyeing with *Cosmos sp* flower with Ferrous sulphate

### Characterization of Dye

Phytochemical screening of plant extract were carried out to simplify the characterization of dye (Wanyama *et al.*, 2011). Phytochemical analysis revealed the presence of carbohydrates, phenols and flavonoids in the extract. Presence of flavonoids, tannins, terpenoids, reducing sugar in the extract considered as significant characters for textile dyes (Wanyama *et al.*, 2011).



Fig. 9. TLC

Thin layer chromatography (TLC) is widely used chromatographic technique due to its simplicity, rapid nature and low cost (Dias,2011).Figure 9 shows the TLC plate of *Cosmos sp* flower extract, where mobile phase is nButanol:Acetic acid:water (4:1:5). According to Harborne (1998) the presence of yellow spot on the TLC plate in day light shows the presence of flavonoid. According to Harborne *et al.* (1998) ,mostly yellow to orange coloured flowers of Asteraceae family contains carotenoids as principal pigment except *Dahlia sp* , *Coreopsis lanceolata* L. and *Cosmos sulphureus* Cav. Their principal pigments are butein and coreopsin , sub group of flavonoid. Flavonoids contain unique UV active chromophore that is phenyl ring which make it easily detectable. Flavonoids mainly has two UV absorbance peak, one is range between 240-285 nm and another range between 300-400nm (Dias, 2011). In this study, figure 10 shows maximum absorbance peak at 387.50nm. Harborne (1998) reported that UV spectrum of yellow flavonoid ranges between 365-430nm and 240-260nm. Therefore it can be concluded that extract of *Cosmos sp* contains flavonoid as major dyeing pigment

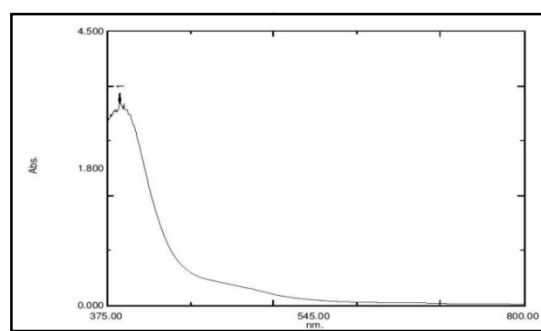


Fig.10. UV Vis Spectroscopic analysis of Dye extract

FTIR helps to identify functional groups of chemical constituents by their characteristic frequencies. Therefore, FTIR considered as a simple but reliable instrument to categorise compound in specific class (Harborne,1998). Peaks between  $1625-1650\text{ cm}^{-1}$  supports the presence of alpha beta unsaturated carboxyl group which is a characteristic of yellow flavonoid pigment (Nissankararao, 2011). Figure 11 shows presence of specific peaks in between above mentioned range. Stretches around  $1610-1570\text{ cm}^{-1}$  implies the presence of aromatic ring and stretches at  $2850-3000\text{ cm}^{-1}$  represents presence of C-H bond which usually considered as prominent band for flavonoid pigment (Aksoz and Ertan, 2012). Presence of distinct characteristic peaks between  $2850-3000\text{ cm}^{-1}$  and  $1610-1570\text{ cm}^{-1}$  range in Figure 11 determines the presence of flavonoid in the dye pigment.

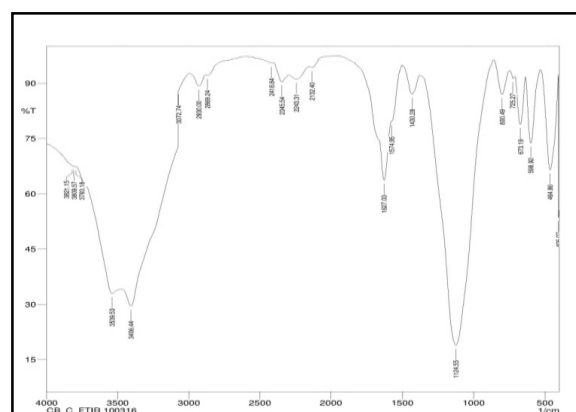


Fig. 11. FTIR analysis of dye extract

## Conclusion

Present study revealed that orange natural dye can be efficiently extracted from *Cosmos sp* flower. The extracted dye could be successfully attached to silk yarn with the combination of chemical mordants having good fastness property. The extracted dye was analysed for its chemical constituent and found the presence of flavonoid as orange pigment.

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