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Research Article

A case study of natural dye extraction and phytochemical screening using the flower *Spathodea campanulata*

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Abstract

The word natural dye derived from the natural sources like plants, animal and minerals. Due to the current eco-consciousness, the researcher's attention has been drawn to the use of natural dyes for dyeing textile materials. The flower of *Spathodea campanulata* dye was used to dye silk at optimized dyeing conditions, using combination of mordant and evaluate the resultant colour fastness of the dyed samples to washing, rubbing, perspiration and light. However; the colour fastness of the test samples exhibited excellent fastness to washing with different mordants and some extracts shown good results after mordanting .The present scenario is more dedicated to the proper utilization of vast diversity of natural resources of colour pigments uses in food materials, pharmaceutical and textiles, rather than their synthetic counterparts.

Keywords: natural dye, spathodea, mordants and textiles

INTRODUCTION

India harbours a wealth of useful germplasm resources and there is no doubt that the plant kingdom is a treasure-house of diverse natural products[1]. One of the products from nature is the dye. Textile materials (natural and synthetic) used to be coloured for value addition and as per the desires of the customers. Anciently, this purpose of colouring the textile was initiated using colours from natural source, until synthetic dyes were invented and these are commercialized. The word natural dye refers to dyes, derived from the natural sources like plants, animal and minerals.

These natural dyes are mostly non-substantive which are on textiles by the help of mordants, usually a metallic salt. The metallic salt has an affinity for both the colouring matter and the fibre. After textile material being impregnated with such metallic salt (i.e. mordanted).It is subjected to dyeing with different natural dyes, usually having some mordantable groups which help in fixation of such dye. These metallic mordants after combining with dye in the fibre, it forms an insoluble precipitate and thus both the dye and mordant get fixed to become wash fast to a reasonable level.

Natural dye from spathodia

The prominence of natural dyes slacked because with the invention of synthetic dyes had some advantages over natural dyes like colour fastness, good reproducibility of shades, brilliance of colour and easy to use[2, 3].

These synthetic dye stuffs produced hazardous by-products some of which possess carcinogenic intermediates and hence a ban has been imposed by Germany and some other European countries on the use of benzidine dyes in textile garments exported into their countries. Hence, due to the current eco-consciousness, the researcher's attention has been drawn to the use of natural dyes for dyeing textile materials[4]. The present study has been undertaken so as to revive the age-old are of dyeing with natural dyes.

In the present work, the flower of *Spathodea campanulata* dye was used to dye silk at optimized

dyeing conditions, using combination of mordants[5, 6] and evaluate the resultant colour fastness of the dyed samples to washing, rubbing, perspiration and light.

MATERIALS AND METHODS

Source: Dried and semidried sample without calyx of *Spathodea campanulata* were collected from Rishi Herbal Technologies Pvt Ltd, Bangalore. *Spathodea campanulata* is a medium sized, reaching height of about 10-35 m. These plants are deciduos, with heavy crown of dense, dark foliage and young bark pale in color. The Flowers are generally large in size, red in color, hermaphrodite in nature, orange insde; calyx is green, about 1cmlong and split on the posterio side, ribed and tomentlous; having 5 petals of 1.5 cm long each; stamens 4with orange filaments; extruding with a2-liped stigma; flower buds curved and contain ared sap. Generally the flowering occurs from early January until early March in India. For the present study the flower samples were collected in March 06, 2012 from trees available in the campus of Rishi Herbal Technologies Pvt Ltd, Bangalore, India.

Materials:

Grinder, Whatmann filter paper, cotton cloth, Soxhlet apparatus, Water bath, colorimeter, Hot air oven, Laminar Air flow.

Chemicals:

1% starch ,sodium carbonate solution, Brine solution, 1M NaOH, 70% Ethanol, 1M H_2SO_4 Solution, Tween solution, Detergent , DMSO(Di Methyl Sulfoxide), 0.08% Stannous chloride.

Mordants:

0.15% alum, 0.08% $CuSO_4$, 0.04% $FeSO_4$, 0.06% $K_2Cr_2O_7$, Myrobolan solution (1:10 Myrobolan : water) soaked for overnight.

PROCEDURE

(a) Extraction:

The present study was undertaken to dye cotton with the semi-dried flowers of *Spathodea campanulata*. 5 grams of flowers of *Spathodea campanulata* was semi-dried, powdered and wrapped in Filter paper and kept for SOXHLET extraction in different solvents like NaOH, Ethanol solution and H_2SO_4.The extract was obtained and kept for boiling on hot water bath at 80°C to evaporate the solvent and allowed to cool, finally filtered and then dried. Even water extract was obtained by boiling the dried sample in hot water for 90°C for 1 hour[7].

NaOH extract obtained = 12.1 gram

Ethanol extract obtained = 4.6 gram

Ethanol extract was dissolved in DMSO solution in 1:5 ratio, and NaOH extract dissolved in distilled water in 1:3 ratio.

Pre-mordanting

Scouring was done with sodium carbonate and Tween mixture (1:1) for 30 minutes.

Cotton cloth pre-mordanted with 1% starch for 20 minutes.

Mordanting

Cotton clothes were each treated with individual and different combinants of mordants.

0.15% alum, 0.08% $CuSO_4$, 0.04% $FeSO_4$, 0.06% $K_2Cr_2O_7$, Myrobolan, 0.08% $SnCl_2$,Myrobolan: $K_2Cr_2O_7$(1:1), Myrobolan : $SnCl_2$

(b)Dyeing:

Cloth kept for dyeing in extract for overnight. Dyed solution with cloth was heated for 75° C so that the cloths take up the dye. Washed in water, dried and kept in brine solution and then washed with detergent and then allowed to shade dry.

It was seen that almost all cloths after washing dint retain dye except the NaOH extract that retain colourupto some extent. The cotton when mordanted with Myrobolan was found to retain much good colour with water extract.

(c) Characterization of dye in table 1:

Initial absorbance was taken of NaOH and Ethanolic extract at 540 nm

Ethanol extract = 1.46

NaOH extract = 1.09 (1:10 diluted with water)

Exposing the dye for 4 hours to different parameters like

Direct exposure to sunlight

U.V

Temperature

Final absorbance after exposure in table 1:

Thus, only Ethanol extract was found to be stable with sunlight. Mostly the stability of dye is decreased with different parameters.

(d) Phytochemical analysis

In the final stage the phytochemical analysis has been carried out and the step wise descriptions are explained below:

Material collection and extraction: Dry flowers were collected, ground into powder and passed through 60 meshes. About 5g of each powder added in 10ml of both the solvents and kept it for 48 hours at room temperature. The extracts were filtered through Whatman No.1 filter paper. This extracts were tested for the presence of active chemical compounds by the following the methods described by Trease and Evans (1989) [7].The different tests i.e. from test are carried out and the details about the test are presented.

Tests for Tannins

One millilitre of the extract was added with 5ml of distilled water and kept for boiling in hot water bath. After boiling, sample was cooled down and to these two to four drops of 0.1% ferric solution was added. Appearance of brownish green or blue black coloration confirms the presence of tannins.

Test for Phlobatannins

One percentage of HCl was added to the extract (1ml) and boiled in hot water bath. Formation of red precipitation indicates indicates the presence of phlobatanins.

Test for Saponins

One millilitre of the extract was taken in a test tube and distilled water (2ml) was added to it. The test tube was then kept in boiling water bath for boiling and was shaken vigorously. Existence of froth formation persisted for next one hour confirms the presence of saponins.

Test for Flavonoids (Ammonia test)

One millilitre of the extract was taken in the test tube and ammonia solution was added (1:5) followed by the addition of conc. sulphuric acid. Appearance of yellow colour and its disappearance on standing indicates the positive test for flavonoids. Here the ammonia is kept into the refrigerator for 30mints before use.

Test for Terpinoids

Five millilitre of extract was taken in a test tube and 2ml of chloroform was added to it followed by the addition of 3ml of conc. Sulphuric acid formation of reddish brown layer at the junction of two solutions confirms the presence of terpinoids.

Test for Glycosides

Five millilitre of each extract was added with 2ml of glacial acetic acid which was followed by the addition of 2ml glacial acetic acid, 1drop of ferric chloride solution and 1ml of conc. sulphuric acid. Formation of brown ring at interface confirms the presence of glycosides.

Test for alkaloids (Hager's test)

0.5ml of the extract. Few drops of 0.1% picric acid were added. Formation of the yellow colour which indicates the presence of the alkaloids.

Test for phenols (Ferric chloride test)

The0.5ml of the extract was added with few drops of neutral ferric chloride (0.5%) solution. Formation of dark green colour indicates the presence of the phenolic compounds.

Test for steroids

Two millilitre of acetic acid was added to 0.5ml of the extract and then added 2ml of H_2SO_4 change of colour form violet to blue or green indicate the presence of steroids.

RESULT AND DISCUSSION

It was found from the study that flower of *Spathodia campanulata*dye can be successfully used for dyeing of cotton to obtain a wide range of soft and light colours by using combination of mordants. However; the colour fastness of the test samples exhibited excellent fastness after washing with premordanting using Myrobolan: Potassium dichromate combination and Myrobolan: $SnCl_2$ and good mordanting with NaOH and water extract but stability of H_2SO_4 extract is lost. But both the ethanol and NaOH extract the colour intensity decreased so it is not suitable dye for the application of textiles.

Table 1 shows the ethanol extract is more stable against sunlight and UV exposure in comparison to NaOH extract. From the phytochemical study of dyes i.e. Table 2, here the Tannins, Cardiolides and Terpenoids are positive in both ethanol and NaOH extract but in the Saponins, Tannins, Cardiac glycosides, Cardiolides and Terpenoids are positive only in ethanol extract. It is been reported that fastness of the dye is influenced by the rate of diffusion of the dye[8], which is well supported by the experiment. Mordants play important role in imparting color to the fabric. However; the mordants used in combination in different ratios give different type of shades[9] and this experiment shows that the extraction comparison between Ethanol and NaOH are quite comparable for the extraction, but test with ethanol extraction gives more positive values than NaOH. Complexing the fiber with mordant has the effect of insolubilizing the dye, making it color fast is also observed and it is well compared with the past studies[10, 11]. Basically for the better colour strength dependents on the metal salt used[12,13] so in the present set up metal salts are being used and which gives reasonable results. The mordanted cotton cloth was immediately used for dyeing because some mordants are light sensitive.

It is found that the whole process of extraction and dyeing is ecologically safe and valuable and cost effective. The obtained results have shown the dyeing potential of all three natural plant sources as source for dyeing. There is need for proper knowledge, documentation and assessment of dye yielding plants as well as the dying techniques so as to increase the use of natural dyes and some of work already presented in past[14,15], and there is also need for the viability and cost effectiveness of the adopted methods. Among differently mordanted, silk dyed with 0.1 N NaOH extract with Myrobolan:

$SnCl_2$(1:1)by simultaneous mordanting technique, causes the fabric relatively higher K/Svalue as compared to other mordanting system[16], which is also supported by this present study.

Ethanol Extract:
When cotton cloth was treated with different mordants like 0.08%$CuSO_4$, 0.04% $FeSO_4$, 0.06% $K_2Cr_2O_7$, 0.08% $SnCl_2$, then $CuSo_4$ and $K_2Cr_2O_7$ gave good colour intensity but colour intensity got decreased gradually when detergent washed and stability was decreased when checked under different parameters. Thus, it is not a suitable dye for application of textiles.

NaOH Extract:
It gave good colour intensity with all the above four mordants but colour intensity got decreased slightly after detergent wash and stability was lost when checked under different parameters. Thus, it is also not a suitable dye for application of textiles.

H_2SO_4 Extract:
When the fabric was pre-mordanted, mordanted and dyed then it was broken into small pieces due to acidic reactions.

Water Extract:
When the fabric was pre-mordanted, mordanted with individual mordants and its combinations with Myrobolan (1:1) gave good colour intensity with this extract and showed best result under different parameters.

CONCLUSION
In this century, the natural resources are protecting the environment and earth from pollution. However; the present study is more focused towards the utilization of the natural resources of color pigments for textiles, food materials and towards medicines in place of their synthetic counterparts. This trend is aimed at safeguarding human health as well as protecting and prolonging life on earth. Detailed scientific studies with natural dyes have established that in most cases their properties are comparable to those of synthetic dyes.
Therefore, if natural dyes have to be commercialized, they need to confirm to the same stringent standards of performance that are applied to synthetic dyes .It thus follows that much more research and developmental effort needs to go in this area. From the above study it was seen that the cotton fabric dyed with water extract was found to have best results and can be used for textile application.

Table 1
Final absorbance after exposure in different light

Dye extract(O.D)	Sunlight	U.V.	Temperature(30°)
Ethanol extract	1.98	1.23	-
NaOH extract	0.33	0.43	0.32

Table 2
Phytochemical Analysis of dye

S.No	TEST	ETHANOL EXTRACT	NaOH EXTRACT
1.	Alkaloids	Negative	Positive
2.	Flavonoids	Negative	Negative
3.	Saponins	Positive	Negative
4.	Phenols	Negative	Negative
5.	Tannins	Positive	Positive
6.	Anthraquinone	Negative	Negative
7.	Cardiac glycosides	Positive	Negative
8.	Cardiolides	Positive	Positive
9.	Volatile oils	Negative	Negative
10.	Terpenoids	Positive	Positive

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