AATCC Review

13 January 2023: Received
11 May 2023: Revised
12 July 2023: Accepted
13 September 2023: Available Online

www.aatcc.peerjournals.net

Research Article Open Access

Chemical Characterization of *Spathodea campanulata* for natural dye

P.S. Devanand*, B. Sivakumar, K. Sivakumar, , N. Raja, K. Hemaprabha, K. B. Sujatha, P. S. Vijayayand[a], K. Nelson Navamani Raj, S. Utharasu, P . Kumar, R. Revathi and K. Kumaran

Faculties, Forest College and Research Institute, Mettupalayam 641301, India
[a]Faculty, Banariamman Institute of Technology, Sathyamangalam 641251, India

ABSTRACT

Spathodea campanulata a perennial tree identified the potential source of natural dye obtained from the flower at forest college and research institute, mettupalayam during 2016-2017. Spathodea campanulata produces a flower dye that has a wide variety of uses like antimicrobial, anti-fungal, antitumor, etc. So the natural dye extracted from African tulip flowers is found to be environmentally friendly and medical value in nature. To identify various components, the dye is analyzed through Gas chromatography and Mass spectroscopy (GC-MS) using the extract prepared from Soxhlet apparatus using methanol. The following compounds were identified in GCMS analysis, Pectinose Beta-GALACTOSIDASE (yellow), 1-Hexadecanol(Cetyl alcohol), Dodecane (substitute for kerosene fuel), Methyl stearate (ester). The spectrum of the unknown component was compared with the spectrum of the known components stored in the inbuilt library. The extraction of dye from Spathodea campanulata can be done easily and a wide spectrum color range is also available which will be helpful to replace the synthetic dyes. This study may give information on the possible production of natural dyestuff components on a large scale as a source of natural dye.

Keywords: *Spathodea campanulata, natural dye, Chemical Standardization, GCMS analysis*

INTRODUCTION

Natural dyes have become a part of human life since time immemorial. The alchemy of colors started its use from an early time. A dye is a colored substance which can be made to adhere to fabrics such as cotton, silk, wool, jute, polyester, nylon, linen, etc. Natural dyes are obtained from flowers, seeds, barks, roots, shrubs, berries, leaves, insects, and minerals. These dyes have been used for centuries to produce colours for fabrics, yarns, leather, foods, cosmetics, drugs, etc. Natural dyes can give subtle and soft colors brightest colour to the yarns and fabrics. Recently, interest in the use of natural dyes has been growing rapidly due to the result of stringent environmental standards imposed by many countries in response to toxic and allergic reactions associated with synthetic dyes.

Nowadays with global concern over the use of eco-friendly and biodegradable materials, considerable research work is being undertaken around the world on the application of natural dyes in textile industry. The effluent problems of synthetic dyes occur not only during their application in the textile industry but also during their manufacture and possibly during the synthesis of their intermediates and other raw materials. The use of non-allergic, non-toxic and eco-friendly natural dyes on textiles has become a matter of significant importance due to the increased environmental use of natural dyes for the colouration of textiles has mainly been confined to artisan / craftsman, small scale / cottage level dyers and printers as well as to small-scale exporters and producers dealing with high-valued eco-friendly textile production and sales . Recently, some commercial and small textile export houses have started looking at the possibilities of using natural dyes for regular basis dyeing and printing of textiles to overcome environmental pollution caused by the synthetic dyes. Natural dyes produce very uncommon, soothing and soft shades as compared to synthetic dyes. For the successful use of natural dyes, the appropriate and standardized dyeing techniques need to be adopted without sacrificing the required quality of dyed textile materials.

Today, in the world of growing environmental consciousness, natural colourants have attracted the attention of everyone. There is a tendency to assume that consumable natural products are safer and better than synthetic products because they came naturally. Presently there is excessive use of synthetic dyes, estimated at around 10,106 tons per annum, the production and application of which release vast amounts of waste and unfixed colorants causing serious health hazards and disturbing the eco-balance of nature. Nowadays, fortunately, there is increasing awareness among people towards natural dyes. Natural dyes are preferred in developed countries because they are non-allergic, non-carcinogenic and have lower toxicity than the synthetic dyes. Natural dyes are mostly non-substantive and must be applied on textiles with the help of mordants, usually metallic salt, having an affinity for both the coloring matter and the fibre. The source for the natural dye can be made easily available from tree waste and grown in gardens. There is been lots of demand for a decade in using natural dye for coloration. Reports have been published on the use of natural dyes over synthetic dyes on silk and cotton. There is a greater need in the present day to revitalize the sacrament of natural

*Corresponding Author: ***P. S. Devanand***
*Email Address: ***devps25@gmail.com***

DOI: https://doi.org/10.58321/AATCCReview.2023.11.04.48

dye and dyeing techniques as an alternative to the use of unsafe synthetic dyes. Hence the present study was undertaken with the objectives of extraction and application of eco-friendly natural dyes from fresh and dry flowers of Spathodea campanulata on silk fabrics and cotton. The effect of UV, sunlight and heat fastness on the fadedness of color was studied. The present study also deals with the effect of mordanting and dyeing properties of cotton and silk fabrics.

Spathodea is a monotypic genus in the flowering plant family Bignoniaceae Spathodea campanulata is commonly known as the Fountain tree, or African tulip tree. It is native to tropical Africa. It grows between 7-25 m (23-82 ft) tall. This tree is planted extensively as an ornamental tree throughout the tropics and is much appreciated for its very showy reddish-orange or crimson (rarely yellow), campanulate flowers. It is commonly planted as a street tree in India. It is considered evergreen but it sheds leaves in dry summers and hence it is a dry-season deciduous tree. The generic comes from Ancient Greek words. Since there are no reports on the phytochemical aspects of the flower of Spathodea campanulata, it was chosen as the subject for this study. This paper aims to validate a rapid method for the quantitative determination of organic compounds in the flower of *Spathodea campanulata* using a rapid fingerprint procedure. Keeping this in view along to produce an ecologically sound product the following objectives are formulated to determine Chemical characterization of *Spathodea campanulata* using GCMS for dye.

MATERIALS AND METHODS

The present study is to find the chemical composition of *Spathodea campanulata* to extract the dye-yielding potential of the plant material used. The dye extraction experiment was conducted at Forest College and Research Institute, Mettupalayam , located at $11^0 19^{'}$N latitude and $77^0 56^{'}$E longitude and an altitude of 300 m AMSL and GC-MS analysis was conducted at Forest College and Research Institute, Mettupalayam. The climate is semi-arid tropical type with hot summer and cold winter. The dry season starts from early February to mid-June and wet season from mid-August to early November. The mean annual rainfall in this region is 895 mm distributed over 49 rainy days with northeast monsoon contributing to 60 percent and the balance through summer showers and the southwest monsoon (Swaminathan et.al 1991).The mean maximum and minimum temperatures are 30^0C and 20.5^0C respectively.

Targeted species

The study is restricted to one target species *Spathodea campanulata* which the currently famous and highly cultivated species owing to its high Antimicrobial content and other ecosystem services that it provides.

Collection of plant material

The picked-up green flower of *Spathodea campanulata* were collected from Forest College and Research Institute and shade dried at room temperature with constant turning to inhibit fungal growth. The dried flower were later crushed using Willey mill to obtain a fine powder to ease extraction using soxhlet apparatus.

Preparation of methanol dye extract

Exactly 5.0 grams of the finely powdered flower of *Spathodea campanulata* were extracted with 25 mL of methanol in an automated soxhlet apparatus (SOXTEC 2043 FOSS). The extraction was performed at 210^0 C for 2 hours and 30 minutes completing three cycles. All the phytoconstituents were extracted from the flower at the end of the third cycle. The extract was then dried at room temperature and the dried extracts were stored at 4^0C in air-tight sterile vials in the refrigerator.

Extraction Methods – solvent extraction

The color compounds in plants may vary in polarities depending on their chemical structure; therefore they need different solvents for extraction. So the parts are repeatedly extracted with various organic solvents. The least polar solvents (eg. carbon tetra chloride) are used first and then proceeded towards higher polar solvents (eg. n-butanol) in succession and finally with water. The water extract mostly contains the glycosides of the coloring principles along with other non-coloring organic compounds, minerals etc. The organic solvent extracts consist of compounds in non-glycosidic form. The glycosidic linkage is opened up by hydrolysis of the water extracts with mineral acids to yield colour constituents (Sivakumaran, 1989). Natural dyes are extracted by boiling the dye-yielding parts of the plant with water for appropriate period and pH. In the aqueous method, dyes can be extracted either in alkaline, neutral, or acidic medium.

These are the extraction process are involved.

Alcoholic Extraction – Alcohol medium
Aqueous Extraction – Neutral medium
Alcoholic Extraction – Alcohol medium

Natural coloring matters depending upon their nature can also be extracted by using organic solvents such as acetone, petroleum ether, chloroform, ethanol, methanol, or a mixture of solvents such as mixture of ethanol and methanol, a mixture of water with alcohol, and so on. Feer (1891) reported extraction of finely powered madder with the treatment of six times its weight of alcohol, containing 10% HCL When the dissolution is complete, the alcoholic solution is concentrated and is precipitated by adding water. The precipitate is filtered and dried at low temperature.

Methanolic extract of the natural dye source is prepared by extracting the raw material in methanol in soxhlet for suitable time period and then evaporated under reduced pressure to make a concentrated extract. This technique enhances mass transfer in supercritical fluid extraction by using modifier for the successful extraction of natural products. Natural colorants such as quercetin, rhamnetin, and other flavonoids are isolated by this technique. In a study, balsam flowers are extracted in aqueous as well as methanolic medium. Flowers are extracted in methanol for 4 hrs in soxhlet to yield the dye (Tiwari et al., 2000b). Annato dye is purified by soxhelation process using methanol at 55°C for 36 hrs. After soxhelation, methanol is evaporated and the dye crystallized from methanol solution, dried and stored (Gulrajani et al 1999).

Aqueous Extraction - Neutral medium

Paul et al. (1996) reported dye extraction from dried rhizomes of turmeric using the aqueous method of dye extraction. The same method is used for fruits of kapila. Dried rhizome/fruits are powdered and boiled in water for 45 min. The extract is filtered and used for dyeing. In a sjfcwiy Carina flowers are crushed and dissolved in distilled water and allowed to sitr in sonicator for quick extraction for hours. (Ghorpade et al. 2006). 'Catechin is extracted from the dried and ground cutch in

aqueous medium by adding cold water to it. The solution is filtered, the residue is dissolved in hot water, and charcoal is added, the solution is boiled and is filtered hot. The filtrate is cooled when crystals of catechin crystallizes. The cochineal dye is obtained by boiling the cochineal insect in water or by placing on a hot oven or by exposing to sun. The latter method produces the highest quality dye. To produce 1 kg of dye 1,50,000 dried insects reared on 0.16 hectares of cactus are required (Glover & Pierce, 1993). For dye extraction from mesta calyx, four methods such as, aqueous, alkaline, acid and alcohol are tried by Katyayini & Jacob 1999 ': however aqueous method is found best to extract the pure form of dye the wood can be chipped and powdered and extracted under soxhlet apparatus.

5g of the coarsely powdered bark was used for the solvent extraction process. The 5g powdered bark was filled in the thimble of Soxhlet apparatus using methanol as a solvent. The process of extraction continued for an hour at 55^{0}C. The solvent extracts collected in airtight containers were preserved in refrigerated condition at 5^{0}C for further use

Chemical composition analysis by GC - MS

The Gas chromatography - Mass spectroscopy (GC-MS) analysis affords the advantage of identifying the chemical entities present, which constitutes the chemical picture of a plant extract, and the whole mixture can be resolved in to individual components. The chemical composition of the extract was analyzed using Thermo GC - Trace Ultra Ver: 5.0 and Thermo MS DSQ II fitted with a DB 35 - MS capillary standard non - polar column (30 m, ID: 0.25 mm and film thickness of 0.25 µm). 0.5 µl of methanol extract was injected for analysis and Helium was used as a carrier gas at 1 mL/ min. The instrument was set as follows, Injector port temperature was set to 250^{0}C, source was kept at 220^{0}C. The oven temperature was programmed from 70^{0}C to 260^{0}C at the 6^{0}C/ min rate. The MS was set to scan from 50 - 650 Da. The MS also had an inbuilt pre - filter which reduced the neutral particles. The data system has two inbuilt libraries for searching and matching the spectrum, NIST4 and WILEY9 containing more than five million references.

The dye stuff components of *Spathodea campanulata* were extracted with water, alkaline medium and acidic medium are feed to GC-MS (gas chromatography and mass spectroscopy) to analyze the chemical components of the heartwood (Lioe H.N. et al). The extract from the soxhlet apparatus is collected and analyzed under Gas Chromatography and mass spectroscopy for the chemical characterization. The extract fed in to the equipment and the total running time is of 42 minutes.

RESULTS AND DISCUSSION

Natural has been traditionally used as a food and beverage colorant all around the world. The dye is widely used in oriental medicine, particularly against inflammation. The methanol extract were subjected to GC-MS analysis.

The methanol dye extract after crystallization has a reddish brown color. The color of the extract varies from red to violet color (Kim et al., 1997; Ye et al.,2006). The color pattern is as follows Mephisto , Rose Soiree , Sundown Yesenrite , Onion Skin Pink, Mellow glow, Mineral, Bois de rose, London smoke (chandraprabha et al ., 2005). Identification of compounds and interpretation of mass spectrum of GC - MS was done using the database of National Institute Standard and Technology (NIST4) and WILEY9 (Dool and Kratz, 1963). Pectinose Beta-GALACTOSIDASE (Graph 1 and 2: yellow), Pectinose is used extensively in winemaking, 1-Hexadecanol(Cetyl alcohol), Cetyl alcohol is used in the cosmetic industry as an opacifier in shampoos (Graph 3), Dodecane (Graph 4: substitute for kerosene fuel) has garnered attention as a possible surrogate for kerosene-based fuels such as Jet-A, S-8, and other conventional aviation fuels.., Methyl stearate (ester) (Graph 5), Methyl Linoleate is classified as a biodiesel due to its long methyl ester chain and is used as a fuel in standard diesel engines. The spectrum of the unknown component was compared with the spectrum of the known components stored in the inbuilt library.

In this century, the natural resources are protecting the environment and earth from pollution. However; the present study is more focused on 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 conform 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 into 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.

The result of the GC-MS analysis of *Spathodea campanulata* is given below

THE SOUTH INDIA TEXTILE RESEARCH ASSOCIATION
COIMBATORE - 641 014

Sample ID:	GC-209	Sample Name:	SPATHODYA SP	Operator:	DSQ
Low Mass(m/z):	50	High Mass(m/z):	650	Comments:	
Run Time(min):	37.53	Instrument Name:	DSQ	Acquisition Date:	05/11/17 05:40:21 PM

EQUIPMENT : THERMO GC - TRACE ULTRA VER: 5.0,
THERMO MS DSQ II

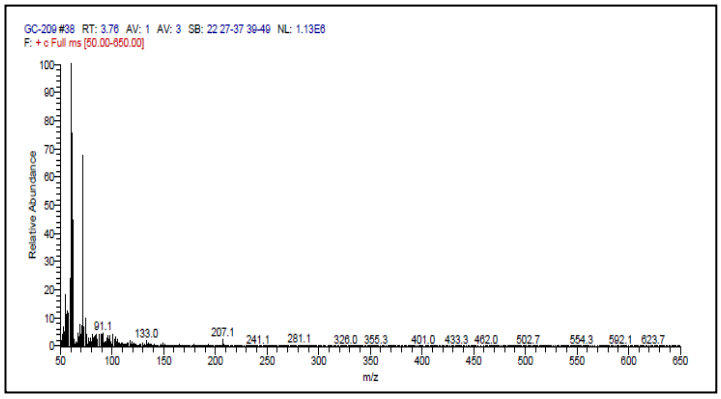
COLUMN : DB 35 - MS CAPILLARY STANDARD NON - POLAR COLUMN

DIMENSION : 30 Mts, ID : 0.25 mm, FILM : 0.25 µm

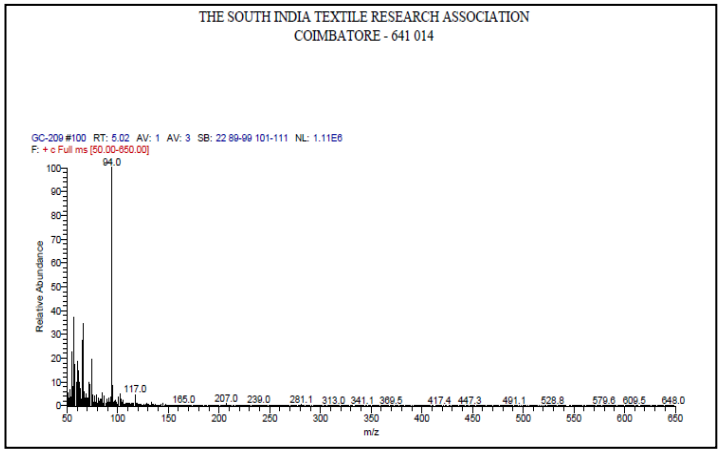
CARRIER GAS : He, FLOW : 1.0 ML/Min

TEMP PROG : OVEN TEMP 70 C RAISED TO 260 C AT 6 C /MIN

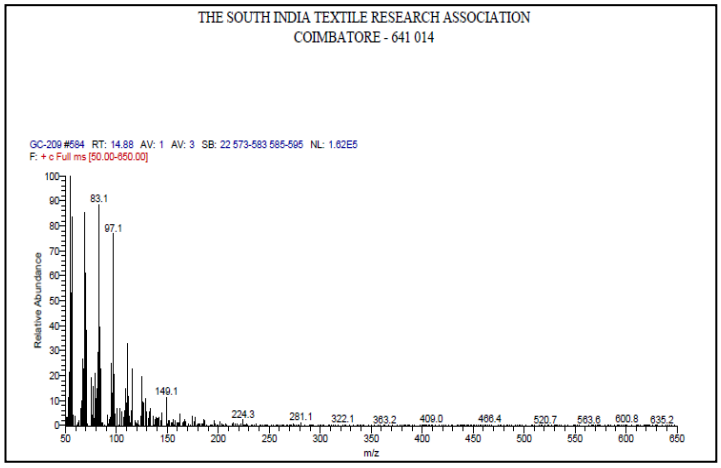
INJECTION
VOLUME : 1 MICRO LITER



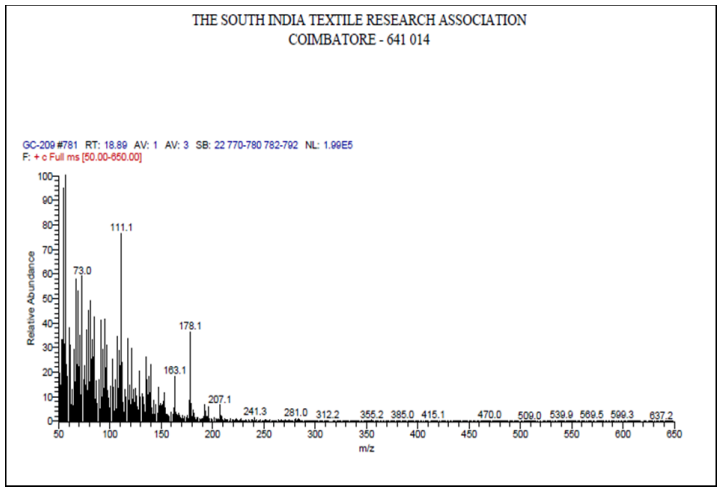
Graph 1: Pectinose



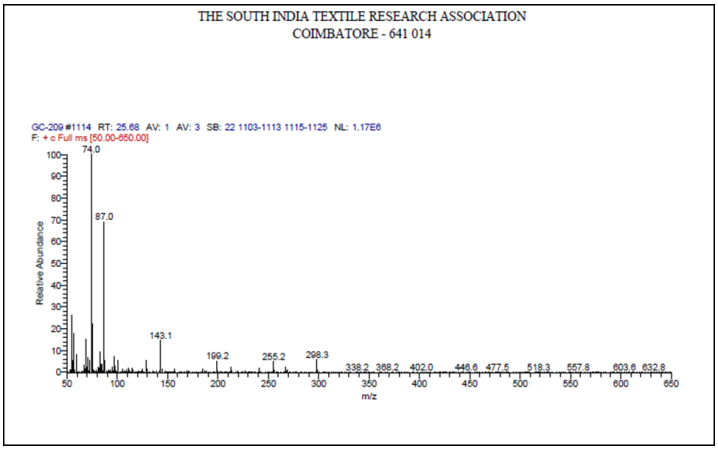
Graph 2: Phenyl-á-D-glucoside



Graph 3: 1-Hexadecanol(Cetyl alcohol)



Graph 4: 2,6-DIENOATE



Graph 5:Methyl stearate (ester)

REFERENCE

1. Quisumbing E.1954. Medicinal Plants of the Philippines.Manila Bureau of Printing p 1234.
2. Dipalma J R.1971. Drills Pharmacology Medicine. (4th ed.) McGraw –Hill Book Ltd.London. p 21-43.
3. Farnsworth N.R, Akarela A.S, Bingel D.D, Soejarto Z.G. 1985.Medicinal plants in therapy.
4. Padua L.S, Bunyaprapkatsara N, Lemmens R.H.M.J.1999. Medicinal and Poisonous Plants. Blachuys Publisher, Leiden p 711.
5. Corral L.G, Post L.S, Montville T.J. 1988. *J. Food Sci.* 53:11-16.
6. Guerrero R.D, Guerrero L.A, Garcia L.L.1990. *Philippines Technol. J.* 15:15-18.
7. Hema,R,Kumaravel S, Gomathi S,Sivasubramaniam C.2010. Newyork Science Journal, 3(11):141-143.
8. Gledhill, D. (2008).The Names of Plants (4th ed.).Cambridge University Press.p.357.
9. Anderson, B., Creative spinning, weaving and plant dyeing, Angus and Robinson, Singapore, 1971,24-28.
10. Samanta, A.K., Agarwal Priti, and Datta Siddhartha, Dyeing of jute and cotton fabrics using jack fruit extract: part –I: Effects of mordanting and dyeing process variables on colour yield and colour fastness properties, Indian Journal of Fibre and Textile Research, 2007, 32, 466-476.
11. Sandeep bains, Sumeet kang, and Koshal preet kaur, Dyeing of wool with Peach dye using combination of mordants, Journal of the Textile Association, 2005,127-131.
12. Sandeep Bains, Singh, O.P., Ganganpreet Goraya, and Manpreet Kang, Dyeing of cotton with Goldendrop (Onosma Echioides) dye, Journal of the Textile Association, 2003, 183-186,

13. Kumaresan, M., Palanisamy, P.N. and Kumar, P.E, Application of eco-friendly natural dyes on Cotton obtained from the stem of *Achras sapota* using combination of mordants, Nature Environment and Pollution Technology,2010, 9(3),547-552.

14. Kumaresan, M., Palanisamy, P.N. and Kumar, P.E, Dyeing of cotton using flower of *Cordia sebestena*:

15. Effects of mordanting and fastness properties, Journal of Asian Scientific Research,2011, 1(4), 137-142.

16. Kumaresan M., Palanisamy P.N. and Kumar P.E, Dyeing of cotton and silk fabrics using stem of *Achras sapota* extract: Effects of mordanting and dyeing process variables on colour yield and colour fastness properties, The Asian Journal of Experimental Chemistry, 2011, 6(1),1-7.

17. Kumaresan M., Palanisamy P.N. and Kumar P.E, Application of eco-friendly natural dye obtained from *Cordia sebestena* on cotton using combination of mordants, Journal of Natural Product and Plant Resources,2012, 2(1), 32-38.