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Isolation and Characterization by Infrared Spectroscopy of Extracted Dye from the Petals of Magnolia *champaka*

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Abstract:

An increased interest for natural pigments started several years ago on the consumers is just beginning to be felt. This paper concerns with the development of process for the isolation of natural dye extracted from the flowers of *Magnolia Champaka* which is available almost everywhere in India. In India traditionally some plants were used for dyeing the fabrics. According to the dye yield and fastness properties the plant was chosen for fabric dyeing. So for the current need we have chosen the flowers of *Magnolia Champaka* to obtain the dye. To get this dye we have carried out different extraction methods Viz Aqueous extraction, Acid extraction, alkaline extraction, Solvent extraction, Soxhlet Method and obtained dye were characterized and confirmed the functional group of Luteolin and Tannin by Infrared Spectroscopy. The chemicals used to produce dyes today are often highly toxic and carcinogenic. Thus use of natural dyes has increased several folds in the past few years. The study concerns with the extraction of natural dye from flowers of *Magnolia Champaka.* Indian women ornament their hair with these flowers, devotees offer flowers to God and Goddesses. Afterwards these flowers are thrown but same can be used for dye extraction.

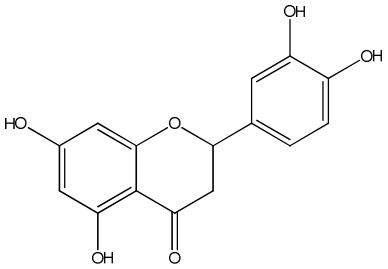
Keywords: Alkaline and aqueous extraction, Infrared Spectroscopy, *Magnolia Champaka*, Solvent extraction

1.0 Introduction:

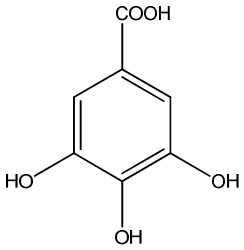
Dyes used for colouration of textiles are as old as textiles themselves. From naturally occurring sources without any chemical treatment natural dyes are derived for e.g. plants, insects and minerals (Sara K, 2008). Medicinal properties are also seen in natural dyes (Chengaiah B, 2010). Fabric colouration is also possible by natural dyes (Jothi D, 2008). Natural dyes and colourants play an important role in world's ecological and cultural heritage. Use of plants and few insects for creating colour is common to all civilization (Adeel et al, 2009). From prehistoric time art of dyeing is practiced, which shows human beings are always interested in colours and its use to decorate the textiles. Initially the use of various parts of plants for colouring rugs, carpets and clothing started and today its developed stages are there through many changes. Extraction of natural dyes is historic from Mesopotamian and Egyptian times. Due to increased awareness of environmental and health hazards associated with the use of synthetic dyes an international interest is arisen (Tanveer H et al, 2008; Kulkarni et al, 2011).Based on the origin of source from which the natural dyes are derived there are different dye classes. (Adrosko, 1971, Mohanty et al,1984 and Chandramouli 1993; Buchanan 1987, Siva R et al, 2007; Supaluk T et al 2012).

More than 500 dye yielding plant species are the gift of nature to human being (Mahanta D, 2005). One such dye yielding plant species is *Magnolia Champaka* (family –Mangoliaceae), commonly called as Son-Chafa in India. *Magnolia Champaka* is very well known flower native to the Himalayas in India and is popular for its fragrant flowers. It is easily available, much branched, widely cultivated as an ornamental shrub in garden for its flowers which are borne in all seasons. The flowers are used in Southeast Asia for several purposes. Especially in India they are primarily used for worship at temples and more generally worn in hair by girls and women as a means of beauty ornament as well as natural perfume.

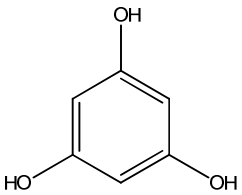
Figure 1: Close view of flower of *Mangolia Champaka*



Luteolin



Gallic Acid



Phloroglucinol

1.1 Chemistry of Dye:

As dyes have complex chemical structures, their chemical names are difficult to understand and remember and common names are in the local language and area specific, a color index has been developed for identifying the dyes. It serves as reference for both the chemical and technical properties of dyes. Natural dyes have a complex chemical composition, unlike synthetic dyes; they are usually not a single entity but a mixture of closely related chemical compounds. On the basis of major chemical constituents present they are divided into 1) Indigo dyes 2) Anthraquinone dyes 3) Napthaquinone , Benzoquinone dyes 4) Flavonoid dyes 5) Caratenoid dyes 6) Tannin based dyes etc. Sources for yellow dyes are enormous. Most of the yellow natural dyes have a hydroxyl or methoxy substituted flavones and tannin based structure (Ferreira E, et al,2004., Harbone J, 1994 and Haslam E, 1998). Yellow dyes are less resistant to fading (Bohmer,Hand Koekboya,2002, Heitor M et al 2007 and Melo J, et al 2006). Dyes with this chemical constitution are found in a wide variety of natural resources. The coloring matter present is flavone Luteolin and its structure is given as below. Tannins are containing multihydroxy compounds. The coloring matter present is tannin and can be obtained as gallic acid and phloroglucinol etc.

2.0 Materials and Methods:

2.1 Materials:

2.1.1 Source: *Magnolia Champaka* plant locally known as son-chafa was collected from Dadar (west) flower market.

2.1.2 Chemicals used: Hydrochloric acid, sodium hydroxide, ethanol and methanol.

2.1.3 Equipments used: A Heidolph distillation rotaevaporator and Soxhlet was used for methanolic extraction of the dye.

2.1.4 Instrument used: IR were recorded on Shimadzu IR-408, a Shimadzu FTIR instrument. The spectra were recorded as KBR pellets and expressed in wave number (cm^{-1}).

Figure 3: Heidolph distillation rotaevaporator

Figure 4: Soxhlet apparatus

2.2 Methods:

2.2.1 Production techniques for Natural dyes:

Synthetic dyes which are synthesized from chemical precursors. Natural dyes are mostly obtained from various plants parts. These dyes bearing materials contain only a small percentage of dye usually 0.5-5%. These plant materials cannot be directly used for dyeing textiles. Many plant materials such as flowers and fruits are seasonal and contain large amount of water and hence cannot be stored as is. Therefore, in order to make them suitable for textile dyeing purposes and to make them available throughout the year many materials can also be sun dried. Dried material is then powered in pulveniser to reduce particle size and to facilitate better dye extraction. At some materials are not stable at room temperature. So storage under nitrogen can further prolong their shelf life.

Extraction Methods:

As natural dye bearing materials contain only a small percentage of coloring matter or dye along with a number of other plant and animal constitutes such as water insoluble, fibers, carbohydrates, protein, chlorophyll and tannins, along others extraction is an essential step not only for preparing purifying natural dyes but also required to be carried out by users of crude dye bearing materials. Extraction of natural dye is a complex process. The different methods for the extraction of colouring materials are 1) Aqueous extraction 2) Alkali and acid extraction 3) Solvent extraction 4) Alcoholic extraction etc.

2.2.2 Aqueous extraction :

Aqueous extraction was traditionally used to extract dyes from plants and other materials. In this method, the dye containing material is first broken into small pieces or powdered and sieved to improve extraction efficiency

2.2.3 Acid and alkali extraction method :

As many dyes are in the form of glycosides. These can be extracted under dilute acidic or alkaline conditions. The addition of acid or alkali facilitates the hydrolysis of chemical functional group resulting in better extraction and higher yield of coloring materials. Alkaline extraction is suitable for dyes having phenolic groups as they are soluble in alkali which improves the dye yield.

2.2.4 Solvent extraction :

Natural coloring matters depending upon their nature can also be extracted by using organic solvents such as acetone, petroleum ether, ethanol, methanol, chloroform or a mixture of solvents such as mixture of ethanol and methanol, mixture of water with alcohol and so on. Thus extraction yield is higher as compared to the aqueous method as a larger number of chemicals and colouring material can be extracted. Purification of extracted color is easier as solvents can be easily removed by distillation and reused.

2.2.5 Extraction by Soxhlet method :

Soxhlet was originally designed for the extraction of lipid from a solid material. Typically, Soxhlet

extraction is used when the desired compound has a limited solubility in a solvent and the impurity is insoluble in that solvent. It allows for unmonitored and unmanaged operation while efficiently recycling a small amount of solvent to dissolve a larger amount of material. The solvent is heated to reflux. The solvent vapour travels up a distillation arm. The condenser ensured that any solvent vapour cool and drips back down into the chamber. Some of the desired compound dissolves in the warm solvent and when Soxhlet chamber is almost full and the chamber is emptied by the siphon and the solvent in return to the distillation flask. This cycle may be allowed to repeat many times over hours or days. After extraction the solvent is removed, typically by means of rotary evaporator yielding the extracted compound. The non-soluble portion of the extracted solid remains in the thimble and is usually discarded.

3.0 Result and Discussion:

3.1 Aqueous extraction:

Aqueous extraction was carried out by adding 20 gms of long flower petals of *Magnolia Champaka* in 200.0ml water. The mixture was stirred by rod and kept at boiling point for 2 hrs on heating mantle. The mixture was cooled to room temperature and finally filtered. The filtrate was evaporated on rotary evaporator. Weight of residue = 1.0 gms. Obtained residue was chracterised by Infrared spectroscopy. The I.R. spectrum of this residue shows characteristic band at 1071 cm^{-1} indicates the presence of -C-O- stretch of alcohol. The band at 1634 cm-1 indicates the presence of isolated –C=C-. The peak at 3252 cm^{-1} indicates the presence of O-H stretching of alcohols.

3.2 Acidic extraction:

Acidic extraction was carried out by adding 20 gms of long flower petals of *Magnolia Champaka* in 100 ml 3 Molar solution of Hydrochloric acid. The mixture was stirred by rod and heated and kept at boiling point for 2 hrs on heating mantle. The mixture was cooled to room temperature and finally filtered. The filtrate was evaporated on rotary evaporator. But the residue is black coloured and is not dissolved in any solvent and also insoluble in water.

3.3 Basic extraction:

Basic extraction was carried out by adding 20 gms of long flower petals of *Magnolia Champaka* in 100ml of 3 molar solution of sodium hydroxide in water. The mixture was stirred by rod, heated and refluxed for 2 hrs on heating mantle. The mixture was cooled to room temperature and filtered. The Filtrate was evaporated on rotary evaporator. Weight of residue= 0.70 gms. Obtained residue was characterized by Infrared spectroscopy. The I.R. spectrum of this residue shows characteristic band at at 1088cm^{-1} indicates the presence of -C-O- stretch of alcohol. The band at 1634cm^{-1} indicates the presence of isolated –C=C- and the peak at 3397 cm^{-1} indicates the presence of O-H stretching of alcohols.

3.4 Solvent extraction:

The 20 gms of long flower petals of *Magnolia Champaka* was weighed and 200ml of ethanol was added and heated to reflux for 2 hrs in round bottomed flask and reaction mass was cooled to room temperature and filtered. The filtrate was evaporated on rotary evaporator. Weight of residue = 0.75 gms. Obtained residue was chracterised by Infrared spectroscopy. The I.R. spectrum of this residue shows characteristic band at 1077cm^{-1} indicates the presence of -C-O- stretch of alcohol. The peak at 3394 cm^{-1} indicates the presence of O-H stretching of alcohols. The band at 1667cm^{-1} indicates the presence of isolated –C=C- .

3.5 Extraction by Soxhlet method :

20 gms of long flower petals of *Magnolia Champaka* was weighed and charged Soxhlet chamber and charged 200 ml methanol in round bottomed flask and heated to reflux. During refluxing the solvent vapour travels up a distribution arm and when distillation arm is full the solvent return in the distillation flask. This procedure of reflux and returning of solvent to be done for 1 hr. cooled the solvent from distillation flask and remove the solvent on rotary evaporator. Weight of residue was 3.5gms. The obtained residue was characterized by I.R spectroscopy. The I.R. spectrum of this residue shows characteristic band at at 1100 cm^{-1} indicates the presence of -C-O- stretch of alcohol. The band at 1652cm^{-1} indicates the presence of isolated –C=C- and the peak at 3333 cm^{-1} indicates the presence of O-H stretching of alcohols.

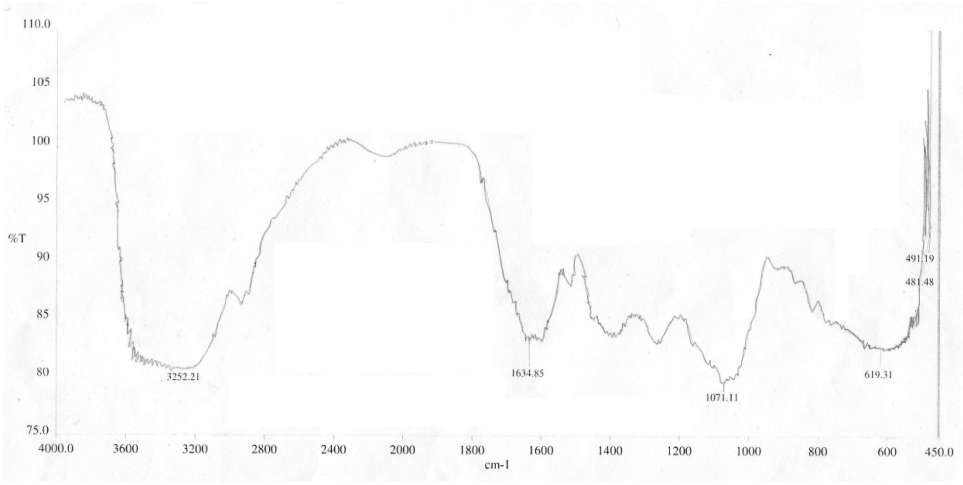


Figure 5: IR Spectrum of aqueous extract of Magnolia Champaka

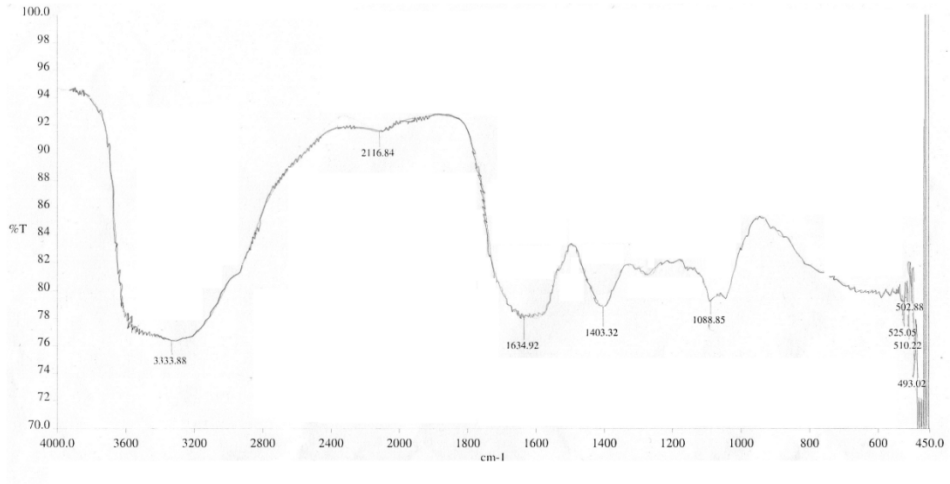


Figure 6: IR Spectrum of basic extract of Magnolia Champaka

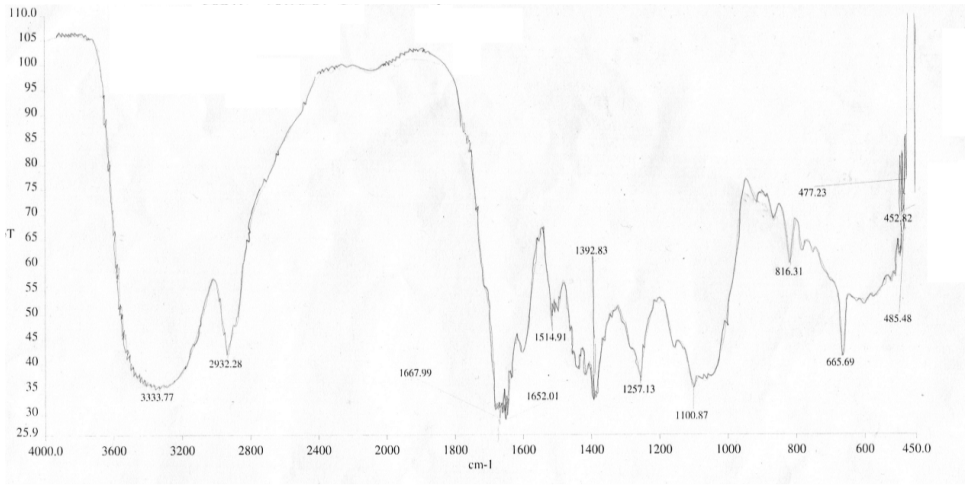


Figure 7: IR Spectrum of soxhlet extract of Magnolia Champaka

4.0 Conclusions:

All methods for the extraction of dye are ecologically safe. The obtained dyes were characterized by Infrared Spectroscopy to check the functional group in flavonoids structure and all the I.R. values matches with the structure of flavonoids. There is lot of scope to use the *Magnolia Champaka* dye for obtaining various color shades using safe mordants under ecofriendly textile dyeing. Thus application and use of this dye will contribute significantly in attaining a safe, ecofriendly and green environment. The obtained dyes can be evaluated on textiles further the possibility of dyeing and printing of textile material. Future research can be carried out on application of dyes on textiles.

5.0 Acknowledgement:

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