International Journal of Agriculture, Environment & Biotechnology
Citation: IJAEB: 6(3):497-501 September 2013
DOI Number 10.5958/j.2230-732X.6.3.023

Agricultural Chemicals

Extraction of *Palas* dye for its use in Textile Coloration

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Paper No. 143 Received: June 7, 2013 Accepted: August 18, 2013 Published: September 03, 2013

Abstract

Natural colorants are emerging globally as the safer and eco-friendly leaving synthetic colorant behind in the race. An attempt has been made to extract the dye from fresh, shade dried; field dropped and sun dried flowers of *Butea monosperma (Palas)*. A good quantity of crude dye was recovered to the extent of 4, 18 and 9 per cent respectively on weight basis. The compounds present in the flowers were analyzed through spectral means and identified as Chalcone, butein ($C_{15}H_{12}O_5$), orange yellow needles and a small quantities of colourless isomeric flavanone, butin ($C_{15}H_{12}O_5$), and its glycoside, butrin. The crude dye after further purification and refining has tremendous scope as a medicine or ingredient for other medicine and can also serve as colouring material in soft drinks and other food products like ham/sausages, jam, chowmin noodles etc.

Highlights

- Method of palas dye extraction methodology have been standardized
- The yield of dye using cold percolation and soxhlet method of extraction were 4% and 2%, respectively
- The dye extract was fractionated through solvent extraction techniques using solvent like hexane, chloroform, ethyl acetate and n-butanol
- The separated constituents were characterized through physical determination of melting points, boiling points and instrumental methods like UV and Infra red (IR) and GC-MS
- Compounds identified from the palas dye were chalcone, small quantities of colourless isomeric flavanone, butin and its glycoside, butrin.

Keywords: *Butea monosperma*, palas dye, butein, food, pharmaceuticals

Introduction

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 (Rungruangkitkrai and Mongkholrattanasit, 2012.). 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). Since the advent of widely available and cheaper synthetic dyes in 1856 having moderate to excellent colour fastness properties, the use of natural dyes having poor to moderate wash and light fastness has declined to a great extent. However, recently there has been revival of the growing interest on the application of natural dyes on natural fibers due to worldwide environmental consciousness (Samanta and Agarwal, 2009).

Butea is a genus of *flowering plant* belonging to the pea family, *Fabaceae*. Several species belonging to this family produce resin used in cosmetics and ayurvedic medicine. *Butea monosperma* or *Butea frondosa*, known as Dhak. Flame of the forest, Palas (primarily in Bengal) or Batsard Teak, is native to India and South Asia, where it is used for timber, resin, fodder, medicine, and dye; it is also a host to the lac insect, which produces resin and natural lacquer.

Palas tree is commonly found all over India, except in very arid region. Palas flower is bright reddish yellow in color, blooms in huge quantity in the beginning of summer season. Flowers on the necked branches appear like Flame of Fire from a distance. Each flower is 2 to 4 cm in diameter. Petals of these flowers are great source of natural dye, which can be used as medicine and food constituent. Nazimuddin *et al.* (1981) demonstrated that aqueous extract of *Gul - e - teesu* (flower of *Butea monosperma*) gave protection against experimentally induced liver injury by CCl_4 in albino rats. Bhatwadekar *et al.*, (1999) studied antistress activity of water soluble part of ethanoic extract of *Butea monosperma* on water immersion stress induced ulceration, elevation of sertonin (5-HT) in brain and corticosterone in plasma in rats. Kasture *et al.*, (2000): carried out the bioassy-guided fraction of dry flower of *Butea monosperma* to isolate the active principle responsible for its anticonvulsant activity. The antibacterial activity of the fresh and dried aqueous extracts (at 10, 50 and 100%, v/v) of flowers of *Butea monosperma* was investigated against *Staphylococcus aureus, S. epidermidis, Pseudomonas aeruginosa, Escherichia coli* and *Salmonella* spp. Seven strains of each bacterial species were used. The fresh aqueous extract had a marked antibacterial effect at 50 and 100% against *S. aureus, S. epidermidis* and *P. aeruginosa*. Further trails are also going on to investigate its usefulness in the treatment of Epilepsy (Kalorey *et al*., 2003). In Thai pharmaceutical handbook of the School of Traditional Medicine Association, mentioned 92 kinds of flower, which are medicinal and mild tasting (Tui Jumsai, 1972). THONGKWO (*Butea monosperma*) is one of those flowers, used for remedy of fever and laxative described in the book. The other medicinal uses of the flowers are well documented in Indian Medicinal Plants (Kirtikar and Basu, 1935). Thus dye obtained from this flower is used as medicine or ingredient for other medicine, hence is usually edible and may find its usage in soft drinks, beverages and other food products like ham/sausages, jam, chowmin noodles etc. In 1995, UK made export of natural colour and dyes of value £ 21.8 million and imported worth £ 23.7 million (Ash and Ash, 1995). Thus, a very big foreign market exists for natural colour and dyes. The present investigation deals with extraction of natural dye from the petals of *palas* flower (*Butea monosperma*) and its probable applications in the field of food and pharmaceuticals.

Materials and Methods

Palas flower were collected from Institute farm and flower petals were taken for extraction of natural dye. The flowers petals were collected in three different ways for the experiment.

A. Flowers on fresh weight basis: The flower petals were weighed after green portion of the fresh flowers was removed. The weighed petals were used for the extraction of *palas* dye.

B. Flowers on shade dried basis: The fresh flowers were collected from trees and green portion of the flowers removed thereof. The flower petals were dried under shade. Final weight of dried petals was recorded after keeping the same in shaded place for a week. The dried petals were used for the extraction of *palas* dye.

C. Flower on field dried basis: The flowers which were naturally dropped from the tree and dried in the sun, were collected and used for the extraction of *palas* dye.

Pre-extraction for removal of impurities: Impurities from the flower petals were removed before extraction of *palas* dye. Hexane, a non-polar solvent was used to remove a large amount of unwanted fat and chlorophyll from the flowers by keeping the flower petals completely dipped in hexane for overnight.

Extraction Techniques: Extraction of *palas* dye from the flower petals was performed by using two methods as described below.

1. Cold Solvent Percolation Method

The flower petals were weighed and taken in a beaker, continuously flushed with cold and fresh pure grade ethanol at room temperature after allowing these petals to be soaked in ethanol atleast for 2 hours. The extracted material was removed and a fresh solvent was again poured in it. This process of extraction remains continued till all the color was extracted from the flower petals. All the extracted material was collected and subjected to concentration by distilling off the extra solvent from the material under ambient temperature (27^0 C) and under vacuum.

2. Soxhlet method: The flower petals were taken in a semipermeable thimble and are flushed with a suitable warm solvent. Ethanol was used as a solvent medium for the extraction of palas dye. Every Percolation of the solvent takes coloring material down in the round bottom flask. This technique is very useful for thermally stable compound. Extracts were collected and concentrated under ambient temperature (27^0C) and under vaccum.

Results and Discussion

The yield of *palas* dye obtained in all the three different ways varied greatly under both Soxhlet and Cold Percolation Methods. Besides yield, difference in colour shade of *palas* dye was also observed while followed two methods of extraction. The per cent yield of *palas* dye (w/w basis) and dye content (per cent) obtained through two extraction techniques which were applied on three different ways of collecting flowers are given in Figure1 and 2.

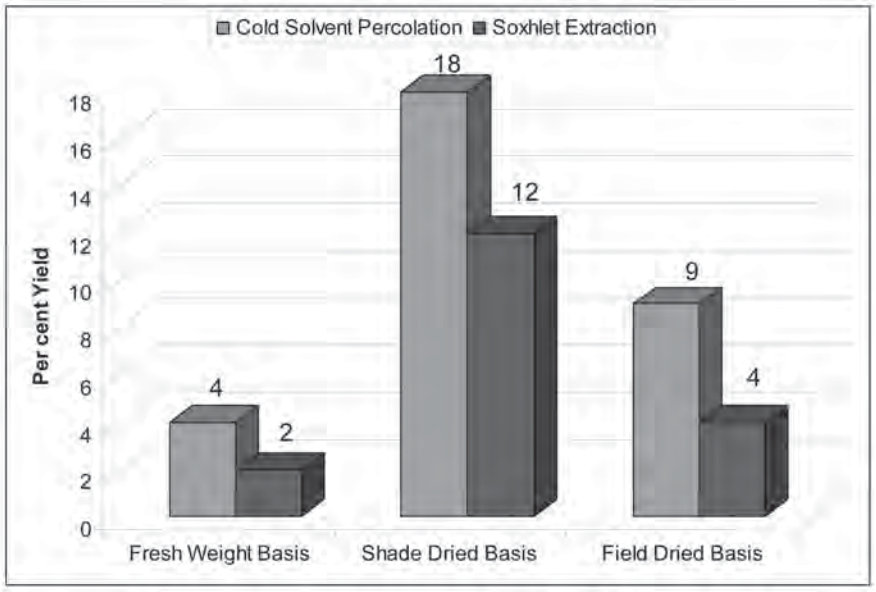

Fig. 1: Yield of *palas* dye recorded per kg weight of flower petals

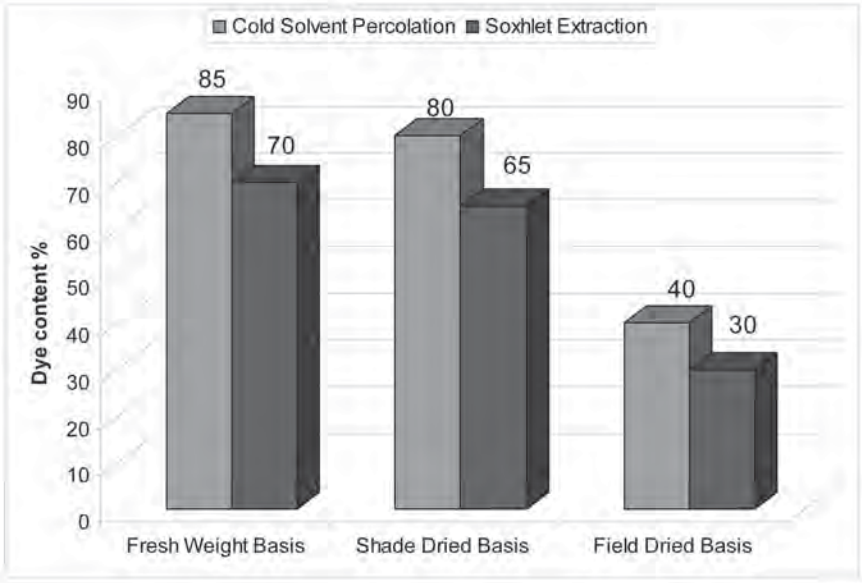

Fig. 2: Dye content of *palas* dye

Yield of dye from fresh flowers

Using Cold Percolation and Soxhlet method of extraction techniques, yield of dye was 4% and 2% respectively whereas dye content recorded was 85 and 70 respectively. The yield and dye content of *palas* dye obtained through extraction using Cold Percolation technique was higher than the one through Soxhlet method. Yield and dye content recorded in flowers on fresh weight basis was comparatively lower which might be due to the reason that more than 75 % moisture was present in fresh flowers.

Yield of dye from shade dried flowers

The shade dried petals were subjected to Cold Percolation Method and Soxhlet method of extraction techniques. Yields of crude orange colour dye obtained through above methods was recorded 18% and 12% respectively whereas dye content recorded was 80 and 65 respectively. The per cent yield, quality of color shade and dye content of *palas* dye was better in Cold Percolation Method than the one which was obtained through Soxlet method. In this case, final percent yields of *palas* dye for both the extraction techniques were good in flowers dried under shade basis.

The reason for higher yield and dye content of dye obtained in flowers on shade dried basis was probably due to the moisture present in the flower petals was optimally reduced during drying and also the drying took place in the absence of direct sunlight which ultimately help to retain natural colour pigment of flowers and thus, better yield of *palas* dye is obtained.

Yield of dye from field dried flowers

The petals were separated, weighed and subjected to Cold Percolation Method and Soxhlet method of extraction techniques. Yields recorded in above methods were 9% and 4% respectively whereas dye content recorded was 40 and 30 respectively. The per cent yield, quality of color shade and dye content obtained by Cold Percolation Method was better than obtained through Soxlet method. But while comparing the total yield, it was found low in both the methods of extraction for flower collected from field dried basis. The most obvious reason for decrease in the yield and dye content was exposure of the flowers to the direct sunlight and heat which might have caused the degradation of colour components in the flowers.

In general, it was observed that the Cold Percolation Method of extraction technique gave higher yield and dye content of *palas* dye for the flowers collected in all the three different methods.

Shade of natural dye obtained through above technique was orange. The yield and dye content both were reduced considerably when Soxhlet Method of extraction technique was applied to extract *palas* dye from flowers collected in all the three methods. There was also a marked difference in color shade and dye content of *palas* dye when Soxhlet Method of extraction technique was used.

As such, it could be concluded that the extraction of *palas* dye obtained through the combination of flower petals dried under shade and use of Cold Percolation Method technique not only gave higher yield but also produced the natural colour of *palas* dye.

Characterization of Natural Coloring / Dye

Seperation and Purification technique

After having obtained the concentrated extract, it is necessary to obtain the desired compounds or material in the pure form.

Solvent Partition

In this step, the extract was acquified and subjected to liquid - liquid extraction in a separating funnel starting from low polar solvent like hexane, chloroform, ethyl acetate and n-butanol. The Thin Layer Chromatography (TLC) study of each separated fraction gave the number of compounds present.

Characterization of Natural Coloring / Dye

The separated constituents by the above method were studied for their physical constants like melting points, boiling points, UV and Infra red (IR) spectra provided information regarding unsaturation (useful tool in detecting the color intensity) and functional groups of the compounds. And finally GC-MS provide the molecular mass of each separated compounds. The compounds present in the flowers identified through above spectral means were as Chalcone, butein ($C_{15}H_{12}O_5$), an orange yellow needles (m.p., 213-215^{0}C). It was obtained as amorphous yellow powder (210 mg), Rf - 0.63 (chloroform: benzene: methanol, 15:2:3), red colour with aqueous NaOH indicated the presence of chalcone aglycone.

UV(MeOH): 234, 275 and 370nm; IR (KBr/cm-1) 3389 (chelated -OH group) 2371, 1595, 766 (phenolic-OH group), 1640cm-1

(>C=O of chalcones); 1H NMR signals at ä 6.25 to 7.66 (aromatic protons), ä 4.906 to 5.314. Molecular ion peak

appeared at m/z 272. It also showed mass peaks at 255 (M-OH), 227 (255 - > C = O), 213 (227-CH2) further confirm the structure.

Small quantities of colourless isomeric flavanone, butin ($C_{15}H_{12}O_5$) (m.p. 224-26^{0}C) and its glycoside, butrin were also obtained. The results obtained here are in full conformity with literature reported earlier on characterization of *palas* dye by Lal and Dutt (1935) and Oberoi and Ledwani (2010). Pal and Bose (2011) also conducted experiment with palas for identification of some new compounds and they reported few novel compounds from this plant. Similar findings were reported by some other author with their medicinal properties (Sharma and Shukla, 2010; Somani *et al.*, 2006).

Conclusion

Many countries have restricted the use of synthetic color in their food products, since it's unsafe for human health and environment. India has a great opportunity for the export of natural dye due to its vast plant wealth and rich traditional knowledge of using color as dye. The development of an economically viable and suitable work to promote the use of natural dye will be accompanied by this effort.

There is a particular need for the development of natural dye world wide, as its use in favour of human health and enviornment. Palas dye has got tremendous potential as a colouring material in soft drinks and other food products like ham/sausages, jam, chowmin noodles etc. This dye has got hidden tremendous medicinal properties such as antistress, anticonvulsant properties, antibacterial effect and its usefulness in fever & laxative which can be flashed by emphasizing the research on the further isolation and characterization (structure elucidate by spectral analysis) of novel bioactive compounds. Hence, isolation and characterization of novel bioactive compounds may lead into the development of new drug or prototype for the preparation of new drugs with better efficacy and lower side effects in the area of pharmaceuticals.

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