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Flowers extract uses in dyeing performance on cotton fibers obtained as flavanone glycosides from *Butea monosperma* (Lam.) Kuntze plant

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Abstract
The aqueous extract of flavanone glycosides were obtained from the flowers of *Butea monosperma* (Lam) Kuntze *(Palash)* as: 5,7-dihydroxy-4[1]-methoxy flavanone-5-D-glucopyranosyl-(1→2)-B-D-glucopyranoside (I) and 5,5[1]-dihydroxy-4[1]-7-dimethoxy flavanone-5,5[1]-di-O-β-D-glucopyranoside (II). The present investigation were carried out for the evaluation of their dyeing properties on cotton fibers. Flowers dye showed interesting shades with excellent washing and light fastness properties and had remarkable absorption percentages.

Keywords: natural dyes flavanone glycosides from flowers of *Butea monosperma* (Lam.**)**

Introduction
Butea monosperma (Lam.) Kuntze plant [1] belongs to the family- Papilionaceae and Fabaceae and commonly called as *Palash, Dhak, Tesu* or *Flame of the forest*. Plant is a medium to large sized upto 05-15m height. It is a native of Asia and India and distributed in Nepal, Indonesia, Thailand, Japan, China, Myanmar, Sri Lanka and Vietnam. Flowers have been used to prepare a traditional yellow dye in India [2]. We report the dyeing properties of flowers and their constituents to validate the traditional uses of the plants in dyeing properties on cotton fibers. The fresh flowers of *Palash* plant were collected from Agra and Mathura district during January to March, which were identified from Department of Botany, School of Life Sciences, Khandari Campus, Dr. Bhimrao Ambedkar University, Agra, U.P. (India).

Materials and Methods
Flowers of *Butea monosperma* (Lam) Kuntze *(Palash)* were collected for the identification of flavanone glycosides as dyeing performance on cotton fibers. *Palash* flowers have been used as blood purifier whereas the seeds as antiseptic and antihelmentic in Indian Traditional System of Medicine. The phytochemical reports on the plant have led to the isolation of alkaloids [3], flavonoids [4], proanthocyanidins [5], terpenoids [6] and tannins [7].

Dried crushed flowers (2 Kg) of *Palash* were successively extracted with *n*-hexane, chloroform and finally with methanol at 45-50 ^{0}C in a Soxhlet Apparatus. Solvents were evaporated upto dryness under reduced pressure to yield 50gm, 80gm and 140gm respectively. The methanolic extract was pre-adsorbed with silica gel (1:1) then applied on the top of a column (15 × 100cm) prepared by silica gel (400gm) in chloroform. The elution was first started with chloroform and then with chloroform-methanol mixture by increasing quantity of methanol up to 50%. The fraction obtained from column were collected every 100 ml and combined on the basis of TLC analysis. The elution 16% and 20% methanol in chloroform afforded two flavanone glycosides compounds were identified and characterized as follows

1. **5,7-dihydroxy-4[1]-methoxyflavanone-5-β-D-glucopyranosyl-(1→2)-β-D-glucopyranoside:** This flavanone glycosides is a yellow hygroscopic solid (70mg), had m.p. 206-208 ^{0}C; UV_{max}^{MeOH} 270, 320; IR (KBr), 3340, 1646, 1500, 1390, 1370, 1010cm^{-1}. Elemental analysis was found to be C, 55.13; H, 5.56; O, 39.28%, calculated value for $C_{28}H_{34}O_{15}$; C, 55.06; H, 5.62; O, 39.30%. ^{1}H NMR; δ5.39dd (J=42, 13.2, H_2; H-2); 2.68 dd (Trans, J=4.2, 12.5, H_2; H-3); 3.43m (H-4); 3.66m (H-5); 3.16m (H-6), 12.5 (OH-7) and ^{12}C NMR; δ 79.2 (C-2), 43.2 (C-3); 183.4 (C-4); 105.4 (C-4a); 163.2 (C-5); 99.5 (C-6); 158.5 (C-7), 97.6 (C-8), 156.8 (C-8a) and 55.4 (C-OMe-4).
2. **5,5[1]-dihydroxy-4[1]-7-dimethoxy flavanone-5,5[1]-di-O-β-D-glucopyranoside:** It is a yellow needles shaped flavanone glycosides (56mg) had m.p. 194-196 ^{0}C, UV_{max}^{MeOH}; 230, 270, 312nm; IR-(KBr), 3324, 1664, 1478, 1400, 1354, 996cm^{-1}. Elemental analysis was found to be C, 54.32; H, 5.64; O, 39.8%, calculated for $C_{29}H_{36}O_{16}$; C, 54.36; H, 5.64; O, 39.94, ^{1}H NMR; δ5.37 dd (J=4.2, 13.4, H_2; H-2), 2.65 dd (J=4.2, 12.5, H_2, H-3); 6.88d (J=2.5, H_2, H-6), 6.98 d (J=2.5, H_2,H-8), 7.25 dd (J=25, 10.4 H3, H-2), 3.51m (H-5), 3.14m (H-6), 5.12d (J=6.7, H_2, H-1), 3.89m (H-2), 330m, (H-3), 13.38m (H-4); 3.62m (H-5), 3.14m (H-6); 3.485 (OMe-4), 3.255 (-OMe-7) and ^{13}C NMR, δ79.8 (C-2) 43.66 (C-3), 183.4 (C-4); 108 (C-4a); 164 (C-5); 99.6 (C-6), 158.9 (C-7), 97.2 (C-8), 157 (C-8a), 133.8 (C-1), 562 (-OMe-4) and 55.8 (C-OMe-7).

Results and Discussion
Fresh flowers of *Butea monosperma* (Lam.) Kuntze (500 gm) were crushed and extracted exhaustively in water (2 ltr) for 4hrs at 60 ^{0}C and used for the experimental analysis. The cotton threads and cloths were pretreated before dyeing by dipping them in flower flavanone. Soap solution and then washed with clean water thoroughly till completely free

from the detergent in order to remove dirt, dust or greases from the cotton yarn. The extracted dye solution was divided into 4 parts and dye solution (100 ml) of each was taken in a separate dye baths. The first dye bath contains pure dye solution, $SnCl_2$ and $FeCl_3$ (10ml, 0.0016g/L each) were added to second, third and fourth dye bath respectively. The scoured, skinned pre-soaked cotton thread (1gm) and cotton cloth ($6cm^{-2}$) were dipped in each dye bath separately. These dyes bath were heated upto 4 hrs with mechanically stirring and allowed to cool at room temperature. Each sample was removed from the dye bath and dried in shade. The optical density of each left over solution was determined one by one-using UV/VIS spectrophotometer and finally percentage absorption was calculated by following formula:

[P.A. = {CO.D, before dyeing-O.D. after dyeing ÷ O.D. before dyeing} × 100]

Each dyed sample was placed on a cardboard frame alongwith blue standard rating. The dyed cotton thread and cloth samples were covered with a black object in such a manner that half of the samples exposed to light. Now the samples were placed inside the Fadometer to determine the rating for light fastness. On other hand, the colour fastness to washing was determined by dipping the dyed samples in the detergent solution for 30 min. and then rinsed in running water. After washing the dye, samples were dried and rating of colour fastness was determined by Launtrometer and identified the dye compounds are as follows:

Flavanone Dye Compound – I

It was isolated as dark yellow compound, hygroscopic solid, soluble in chloroform and recognized as flavanone glycoside by means of positive Gibbs, $FeCl_3$ and Molish tests. Molecular formula was deduced as, $C_{28}H_{34}O_{15}$ from its molecular ion peak at m/z 610.5584 $[M]^{Y}$, (Calcd for 610.5604) in its positive HR-FABMS. IR bands at 3340, 1645 and 1500 cm^{-1} were due to presence of chelated –OH, C=O and C=C functions. UV-absorption at 268.320 (MeOH); 277, 296 (NaOMe) and 256 Sh, 276, 300 in $AlCl_3$ showed glycosidic nature of compound (I) which was supported by two downfield signals for anomeric centres at, δ 5.22 (d, J=6.8 H_2) and 4.8 (d, J=6.6 H_2) in ^1H NMR and 102.8 and 103.4 in ^{13}C NMR. The coupling constant J=6.8 and 6.6 H_2 were indicated for 3-D configuration of both sugars. DEDT (130^0) spectrum exhibited 28 signals corresponding to 1-methyl, 3-methylene, and 17-methine and 7-quanternary carbon. The ^1H NMR displayed 3ABX type signals at δ 5.38 (dd, J=4.2 and 13.2 H_2) for H-2 with 62.68 (dd, J=4.2 and 12.6H_2) for H-3 Trans and 3.20 (dd, J=12.4, 16.2H_2) for H-3Cis, characteristic of flavanone [8]. In ^{13}C NMR, three downfield signals at δ 145.6 (C-7) 158.4 (C-7) and 163.2 (C-5) were suggested to three oxygen substitute Carbon including one methoxy (δ 3.06 cm^{-1} H and δ 55.6 in ^{13}C NMR), one hydroxyl (δ 12.5 in ^1H NMR) and one glyosidic substituted. The linkages of substitution was confirmed by means of COSY, NOESY and HMBC correlation. The long-range correlation of δ 12.5 (OH) to 97.6 (C-8) /99.4 (C-6) /158.4(C-7) in HMBC confirmed the position of hydroxyl group at C-7. Similarly the linkages of methoxyl group at C-4 was confirmed by NOESY correlation of δ 3.06 (-OCH_3) to 6.86(H-3, 5) and HMBC of δ 55.4 (-OCH_3) to 145.6 (C-4). Correlation of δ 5.22 (H-1) to ^{1}63.4(C-5) in the HMBC confirmed the linkages of glycosidic at C-5. The ^{13}C NMR value of the sugar carbon at δ 77.6 (C-2) showed that C-2 was the linkage to another sugar unit. Hydrolysis (12% Me OH – HCl, 10 ml, 60-80 ^{0}C, 8 hrs) as well as methanolysis (methylation and hydrolysis) followed by paper chromatography confirmed the presence of two glucose molecules with (1→2) linkages [9]. Mass spectra was found very informative in respect of the structure elucidation in which the fragments at m/z 449.26 and 285.24 were corresponding to the loss one and two glucosyl unit respectively from molecular ion of the fragmented as m/3 151.42 (100%), 161.16, 947.20 and 91.34 were typical for flavanone[10]. It was identified as 5,7-dihydroxy-4^1-methoxyflavanone-5-β-D-glucopyranosyl-(1→ 2)-β-D-glucopyranoside.

Flavanone Dye Compound – II

It was obtained as needles shape compound had molecular formula, $C_{29}H_{36}O_{16}$ from Quasi molecular ions at m/h 641.5902 $[M+H]^+$ and molecular ions at m/z 640.5800 (calcd, 640.5864) in its positive HRFABMS. It was also recognized flavanone glycoside by means of characteristic chemical tests by IR and UV –spectra. The DEPT (135^o) spectrum showed 29 signals out of them 2 methyl, 3 methylene, 16 methine and 18 carbons. The downfield signals 4 in the ^{13}C NMR at δ 148.4 (C-4^1), 164.0(C-5), 152.4(C-6) and 159 (C-7) were corroborated to 4 oxygen substituted carbons. Among them 2 signals were assigned for methoxyl functions (δ 55.8 and 56.2). Anomaric signals 2 at δ 5.14and (J=6.8Hz) in ^1H NMR and δ102.8 and 103.4 in ^{13}C NMR were clearly showed that the presence of 2 sugar moieties. Partial hydrolysis as well as methanolysis were confirmed by HMBC, NOESY and COSY studies. The NOESY correlation of δ 3.48 (-OCH_3-4') to 7.02 (H-3'), 3.24 (-OCH_3-7) to 6.88 (H-6)/6.98 (H-8) and HMBC of 5.16 (H-1") to 164(C-5), 5.12(H-1") to 152.4(C-5'), 3.48 to 148.4 (C-4'), 3.24 to 158.8 (C-7) confirmed the linkages of methoxy groups at C-4' and C-7 whereas glucose molecule at C-5 and C-5'. Mass spectra exhibited a molecular ion at m/z 640.56, which produced two daughter ions at 477.42 and 315.32 due to loss of 2 glucosyl moieties. The fragmentation pattern of compound –II was found similar to that of compound –I, characteristic for a flavanone. On the basis of above discussion the flavanone dye compound-II was characterized as 5, 5'-dihydroxy-4', 7-dimethoxy flavanone-5, 5'-di-O-β-D-glucopyranoside.

The aqueous *Palash* flower extract showed UV- absorption at 320nm whereas compound-I and II showed at 322 and 314 nm respectively as the characteristic for flavonoids. The flowers extract from compound I and II showed remarkable absorption percentage by 41.76, 51.14 and 49.88 respectively. The flower extract with natural mordant from *Butea monosperma* (Lam.) Kuntze (Palash) plant exhibited better absorption as compared to that of the synthetic dye.

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