MedCrave
Step into the World of Research

Journal of Textile Engineering & Fashion Technology

Research Article

Open Access

CrossMark

Spectrum of colors from reseda luteola and other natural yellow dyes

Abstract

In our search for a good natural yellow dye, Reseda luteola L fresh extract of the flower, stem and leaves was found to have good colorant quality. As the dyed cotton, silk and wool fabrics as well as yarn showed very good wash and light fastness properties, exploration of dyeing potential of this wonderful dye extract under different pretreatment conditions, mordanting- pre and post with metal mordants, biomordant, enzymes and surfactants were carried out. Our study with Reseda extract showed the following results- bright yellow color with alum, olive green with copper sulphate and dark brown with ferrous sulphate. Sodium salt of Dodecyl benzene-sulphonic acid (SDBS) and Cetyl trimethyl ammonium bromide (CTAB) as pretreatment have also been used. Combination of other natural dye extracts with Reseda extract has been attempted. A wide spectrum of colors has been obtained ranging from canary yellow to olive green to orange.

Keywords: reseda dye, mordants, biomordants, surfactants, combination of natural dyes

Volume 4 Issue 2 - 2018

Padma S Vankar, Dhara Shukla

Facility for Ecological and Analytical Testing, Indian Institute of Technology, India

Correspondence: Padma S Vankar, Facility for Ecological and Analytical Testing, Indian Institute of Technology, 204 A Southern Block, Kanpur-208 016, India, Email padma.vankar@gmail.com

Received: October 22, 2017 | **Published:** March 01, 2018

Introduction

Yellow dyes have always been used from ancient times and have been a color of marking festivity. Most of the yellow dyes are flavonoids which are not stable to UV radiation. The dyed fabric shows very poor wash and light fastnesses. In our quest for a stable yellow dye we started exploring all the possible sources of yellow natural dyes ranging from Curcuma rhizome, Eucalyptus barck Super Critical Fluid Extract (SCFE), Gaillardia flower, Thevetia peruviana flower, Tegetus flower, Punica granatum fruit epicarp, Cassia fistula flower, Cosmos sulphuerus flower, Carthamus flower and Reseda luteola flower, stem and leaves extracts. Since a lot of work had already been done for the identification of colorant in Reseda extract, some very pertinent questions arose to understand the mode of attachment of this colorant molecule on the fabric for stable dye adherence and unusual good fastness properties. Owing to its good thermal stability, the colorant can be extracted easily with water at alkaline pH or even with methanol: water mixtures.

A method was applied to evaluate the influence of soil fertility on the production of flavonoids in Reseda dye. The results showed that dye capacity is dependent on soil fertility and the origin of seeds. HPLC-diode array detector (DAD) methodology was developed to allow the simultaneous identification and quantification of Reseda luteola L. (weld) dye flavonoids, luteolin, apigenin, luteolin 7-O-glucoside, apigenin 7-O-glucoside, luteolin 3',7-O-diglucoside and luteolin 4'-O-glucoside[1] as shown in Figure 1. The method was developed by Gaspar et al, for the simultaneous identification of Reseda luteola L. (weld) flavonoids and quantification of the main compounds responsible for the yellow color. This method was applied to a large number of wild Portuguese welds to evaluate its potential application as dyestuff for textile factories, as a substitute for the synthetic dyes currently used. All these molecules had good chelation with metals due to presence of oxo and hydroxyl groups. The complex stoichiometry ratio of aluminium and luteolin was found to be 1:2.[2]

The most appropriate leaching solvent for luteolin from leaves, stems and flowers of *Reseda luteola* was found to be methanol optimal luteolin extraction was 8.6g/kg of plant material. Preliminary dyeing tests on pre-mordanted raw cotton and wool standard specimens gave greenish-yellow hue, acid perspiration fastness was found to be resistant to fading of dyed wool specimens was generally greater than that of cotton dyed samples.[3]

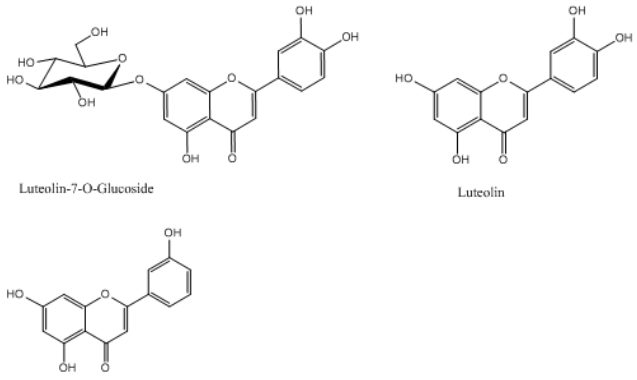

Figure 1 Reseda luteola L. (weld) dye flavonoids, luteolin, apigenin, luteolin 7-O-glucoside.

The structural difference in the various yellow flower extracts gives further insight into the uniqueness of Reseda extract. Curcuma rhizome has main colorant as Curcumin with some Rutin flavonoid[4] as shown in Figure 2.

Gaillardia flower extract consists of Sesqiterpene lactones, aglycones and glycosilated flavonoids, dihydroxy flavonol and 6-methoxyethers[5] as shown in Figure 3.

Thevetia peruviana flowers have main flavonoids as quercetin, Kaempferol and quercetin-7-O-galactoside[6] as shown in Figure 4.

Cosmos sulphureus flower extract consisted of as Fustin and quercetin[7,8] as shown in Figure 5.

Tegetus flower consists of Patuletin and Patulitrin flavonoids as their main constituents[9] as shown in Figure 6.

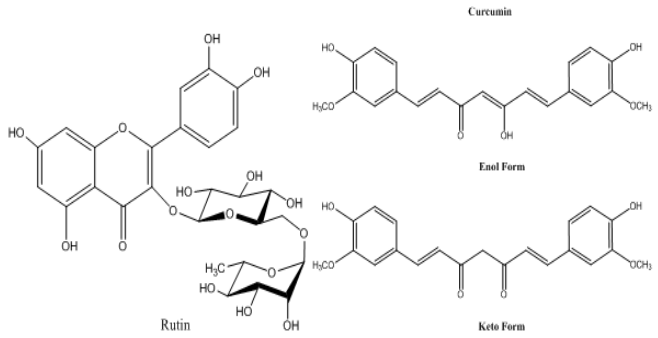

Figure 2 Curcuma rhizome has main colorant as Curcumin with some Rutin flavonoid.

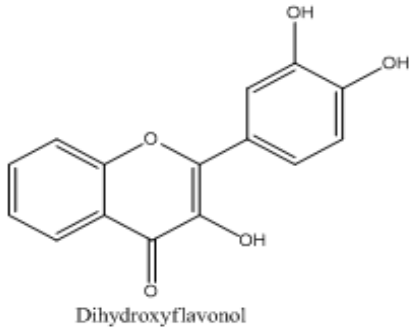

Figure 3 Gaillardia flower extract consists of glycosilated flavonoids, dihydroxy flavonol.

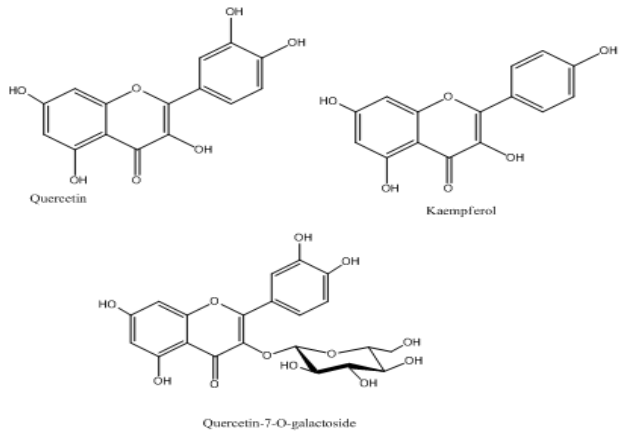

Figure 4 Thevetia peruviana flowers have main flavonoids as quercetin, Kaempferol and quercetin-7-O-galactoside.

Punica granatum consist of Myricetin, quercetin, Luteolin and Kaempferol in the epicarp of its fruit[10] as shown in Figure 7.

Cassia fistula yellow flower has Kaempferol, Catechin and proanthocyanidins as the main flavonoids[11] as shown in Figure 8.

Carthamus tinctorius is also another plant with flavonoids such as acacetin, luteolin, quercetin, and their glucuronide, cinaroside, 5-O-methylluteolin and rutin[12] as shown in Figure 9.

The Supers Critical fluid extract of Eucalyptus Bark also is rich in flavonoids such as Eriodictyol, naringenin and isorhamentin[13,14] as shown in Figure 10.

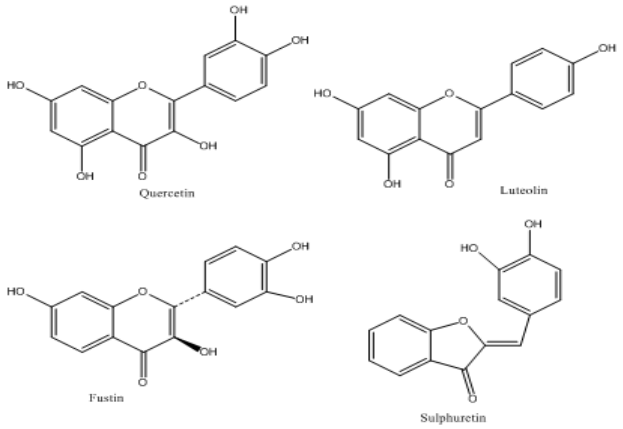

Figure 5 Cosmos sulphureus flower extract consisted of as Fustin and quercetin.

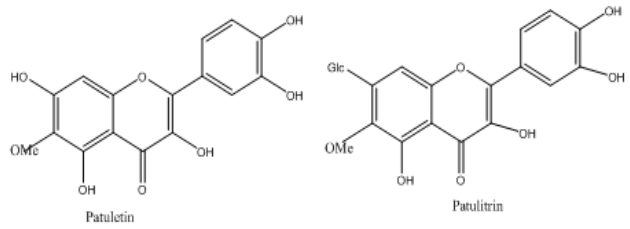

Figure 6 Tegetus flower consists of Patuletin and Patulitrin flavonoids.

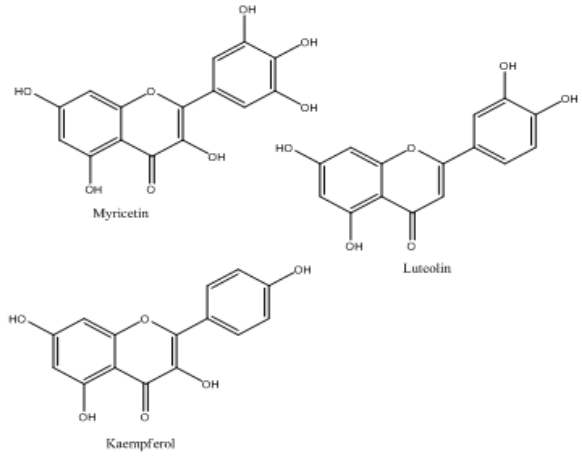

Figure 7 Punica granatum consist of Myricetin, quercetin, Luteolin and Kaempferol.

It well known that flavonol based molecules- quercetin and kaempferol are found to be more prone to light induced fading than flavones based luteolin and apigenin.[15] Infact the latter ones darken with time.

The preferred binding site depends on the flavonoids, metal and the pH. Even the binding sites differ for flavonol and flavones with Al^{+3} at different pH. At alkaline pH flavonols have strongest affinity with Al^{+3} through their ortho dihydroxy group while flavones show binding through site 4 and 5 of A and C rings or the catechol sites in ring B[16,17] and shown in Figure 11.

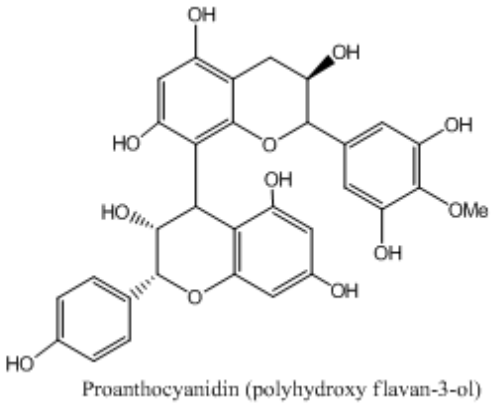

Figure 8 Cassia fistula yellow flower has Kaempferol, Catechin and proanthocyanidins.

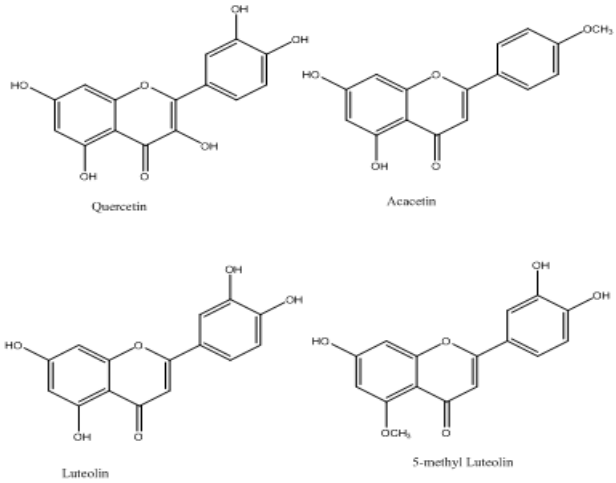

Figure 9 Carthamus tinctorius is also another plant with flavonoids such as acacetin, luteolin, quercetin, and their glucuronide, cinaroside, 5-O-methylluteolin.

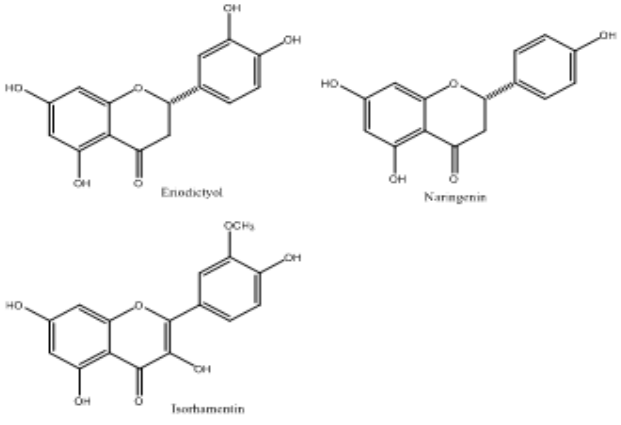

Figure 10 Eucalyptus Bark is rich in flavonoids such as Eriodictyol, naringenin and isorhamentin.

Most of the yellow dye plants contain colorant molecule from the group of hydroxyl flavones, however the combination of components may differ from plant to plant and species to species.[18] Some plausible explanation can be given to the fact that the Table 1 shows such vast difference in wash, light fastnesses, solubility and dyeability of the natural extracts (Figure 12) (Figure 13). It is very apparent that among 10 different sources, Reseda excels in all the three parameters (Tables 2) (Table 3) (Figure 14).

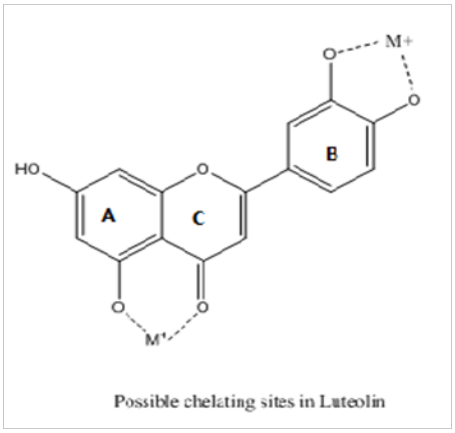

Figure 11 Ortho dihydroxy group while flavones show binding through site 4 and 5 of A and C rings or the catechol sites in ring B.

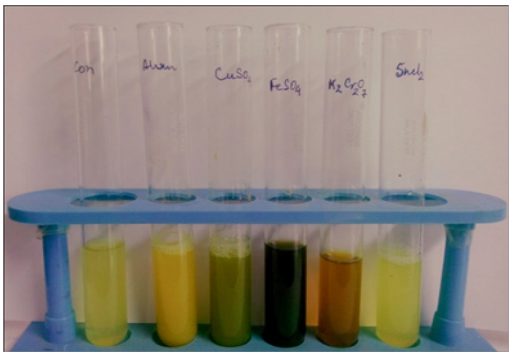

Figure 12 Reseda extract with different mordants.

Figure 13 Reseda plant.

Citation: Vankar PS, Shukla D, Spectrum of colors from reseda luteola and other natural yellow dyes. *J Textile Eng Fashion Technol.* 2018;4(2):106-119. DOI: 10.15406/jteft.2018.04.00127

Table 1 Relative dyeability and fastness properties of extracts

Dye plant	Wash fastness	Light fastness	Dyeability	Solubility in water
Reseda	Very Good	Very Good	Very Good	Good
Gaillardia	Poor	Poor	Very Poor	Poor
Thevetia	Poor	Poor	Poor	Poor
Tegetus	Good	Good	Good	Poor
Punica	Good	Good	Good	Good
Cassia	Very poor	Very Poor	Very Poor	Poor
Cosmos	Good	Good	Good	Poor
Carthamus	Good	Good	Good	Poor
Curcuma	Poor	Poor	Poor	Good
Eucalyptus SCFE	Good	Good	Good	Poor

Table 2 Reseda extract with different mordants

CIE lab	Reseda	RI + maddar	RL+catechu	RL+eupatorium	RL+indigo
L*	64.565	43.242	15.778	48.442	64.867
a*	-6.463	14.572	25.994	7.394	-33.62
b*	47.577	42.009	27.175	45.367	12.703
C*	48.014	44.465	37.605	45.966	35.94
H*	97.769	70.841	46.254	80.711	159.31
dE*	-----	30.465	62.047	21.374	44.202
Color	Yellow	Orange	Brown	Olive green	Turquiose

Table 3 Reseda extract in combination with other natural dyes

CIE lab	Reseda	RI + maddar	RL+ctechu	RL+eupatorium	RL+indigo
L*	64.565	43.242	15.778	48.442	64.867
a*	-6.463	14.572	25.994	7.394	-33.62
b*	47.577	42.009	27.175	45.367	12.703
C*	48.014	44.465	37.605	45.966	35.94
H*	97.769	70.841	46.254	80.711	159.31
dE*	-----	30.465	62.047	21.374	44.202
Color	Yellow	Orange	Brown	Olive green	Turquiose

UV-Visible analysis of reseda dye and other yellow dye yielding plants: The UV- Vis spectra of most of the flavonoids show two main absorption bands such as a) the benzoyl band at 240-280 nm range and b) the cinnamoyl band at 320-385 nm range.[15]

Reagents: all reagents should be of analytical purity: Methanol, Reseda dye and other natural yellow extracts

Apparatus and equipment: Ultra Violet -Visible spectrophotometer machine, Quartz sample tube (cuvette) having 1.00 cm light path.

Sample preparation: The Reseda dye was weighed separately (0.1gm) in 1000 ml methanol and scanned through UV-Visible spectrometer. For visible spectrum this solution is used and for UV spectrum the solution is further diluted by 5 times. Identification of the dye by this method is through the ultraviolet (UV) region scanning from 200 to 400 nm, and the visible portion is from 400 to 800 nm (Table 4) (Figures 15−21).

Different yellow flowers and natural reseda dye by HPLC method

Reagents: All reagents should be of analytical purity: Methanol, De-ionised water, Ethyl acetate (HPLC grade)

Apparatus and equipment: HPLC Machine (Waters), Ultra Violet-Visible Detector (Waters 2998), C-18 reverse phase column (RPC -C18), Binary pump system, Micro syringe

Sample preparation: All the different types of yellow flowers

were extracted in methanol, while Reseda dye sample was weighed separately (0.1gm) in 1000 ml of methanol and then the sample is diluted to 100 times, 1µL of this diluted prepared solution was injected to the C-18 reverse phase column and eluted through column. The base line showed response within a run of 15 mins for natural Reseda dye. The parameters of this assay were made to be such that a clean peak of the both the samples are observed from the chromatograph.

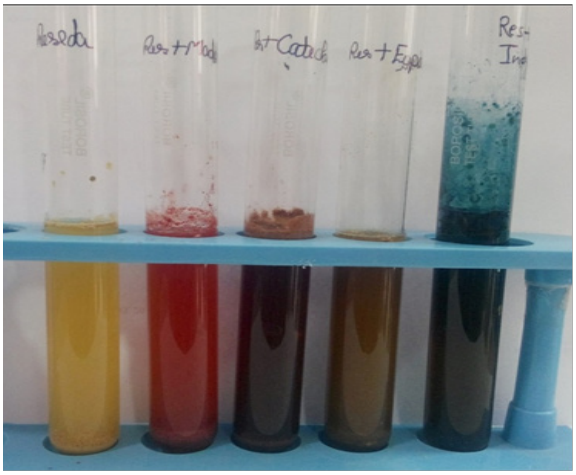

Figure 14 Reseda extract combination with different natural dyes.

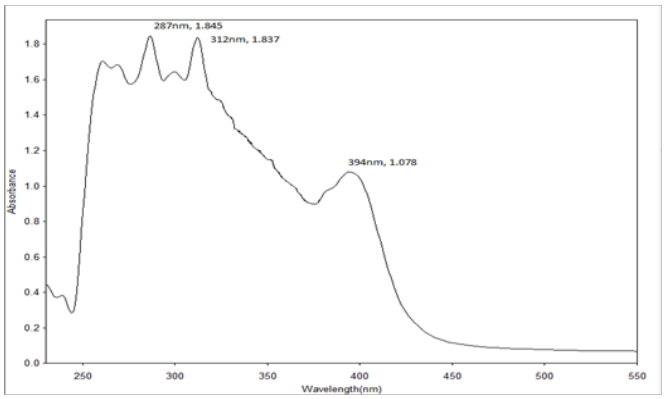

Figure 15 Reseda dye shows two very prominent peaks in the UV region at 287nm (1.84OD), 312 nm (1.83 OD) and a peak at 394-401nm (1.07OD).

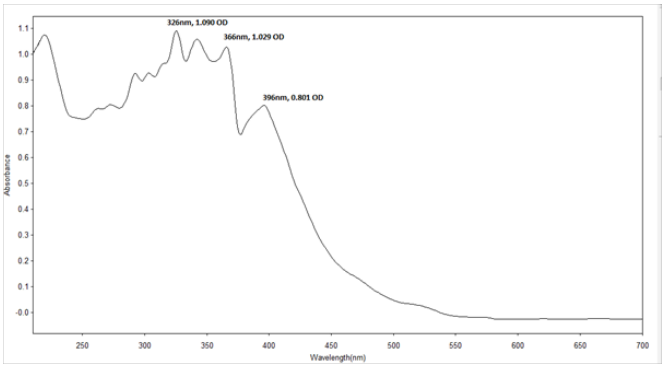

Figure 16 Cosmos dye shows three prominent peaks in the UV Vis region at 326 nm (1.090 OD), 366 nm (1.029 OD) and 396 nm (0.801 OD).

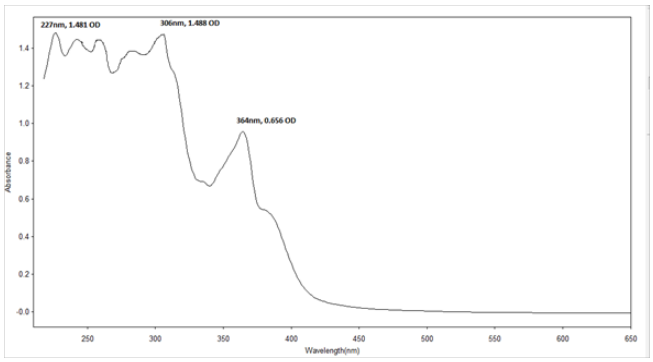

Figure 17 Punica dye shows three prominent peaks in the UV Vis region at 227 nm (1.401 OD), 306 nm (1.488 OD) and 364 nm (0.566OD).

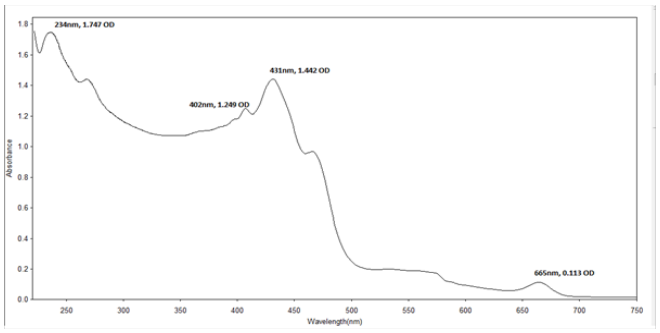

Figure 18 Tegetus.

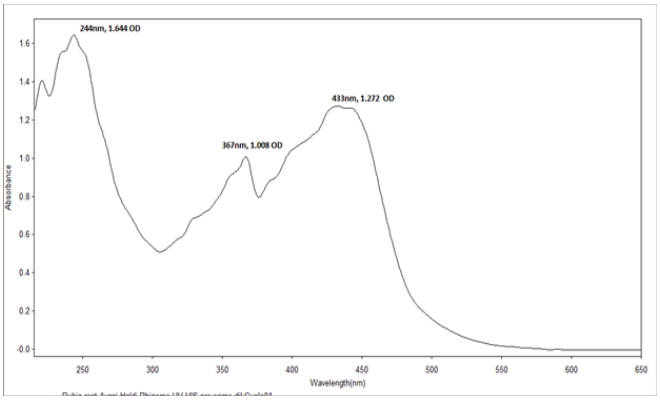

Figure 19 Curcuma.

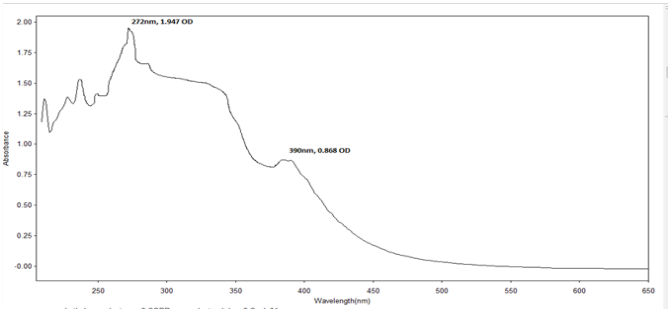

Figure 20 Eucalyptus bark Supercritical extract.

Citation: Vankar PS, Shukla D, Spectrum of colors from reseda luteola and other natural yellow dyes. *J Textile Eng Fashion Technol.* 2018;4(2):106-119. DOI: 10.15406/jteft.2018.04.00127

Table 4 Identification of the dye by this method is through the ultraviolet (UV) region scanning from 200 to 400 nm, and the visible portion is from 400 to 800 nm

Flower extract	Benzoyl band 240-280 nm	Cinnamoyl band 320-385 nm
Reseda	268	348
Curcuma	244	367
Tegetus	234	402
Punica	227	306
Cosmos	274	382
Carthamus		
Cassia	247	398
Gaillardia	272	388
Thevetia	291	308
Eucalyptus bark SCFE	272	390

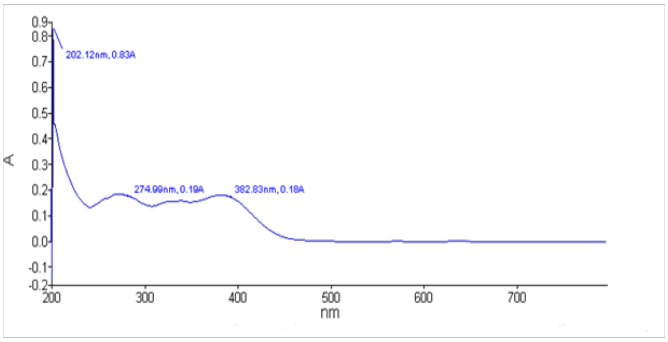

Figure 21 Cosmos.

Method used

The experimental conditions are as following: Column C18, 150×4.6 mm; flow rate: 1.0 ml/min; detection wavelengths 255 nm (band width 16 nm), Solvent System: Methanol: Deionised water (97: 03) in method-I and EtOAc: MeOH (10:90) in method -II has been used, Pump Pressure: 15 MPa, Machine Brand: Waters. Clear observation was made from the analysis of Reseda dye by two different methods as mentioned below:

Method-I

Commercial reseda powder: Solvent System: 97:03 MeOH: H_2O, Run Time: 12 min, Sample prepared in MeOH, Chromatogram taken on 366nm (Figures 22−27).

Cosmos flower: Solvent System: 97:03 MeOH: H_2O, Run Time: 15 min, Sample prepared in MeOH, Chromatogram taken on 255nm (Figures 28) (Figure 29).

Punica granatum: Solvent System: 97:03 MeOH: DW, Run Time: 15 min, Sample prepared in MeOH, Chromatogram taken at 255nm (Figures 30−35).

Carthamus flower: Solvent System: 95:5 MeOH: DW, Run Time: 15 min, Sample prepared in MeOH, Chromatogram taken at 255nm (Figure 36).

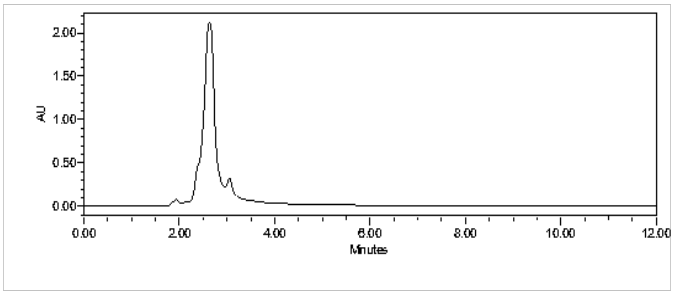

Figure 22 Commercial reseda powder.

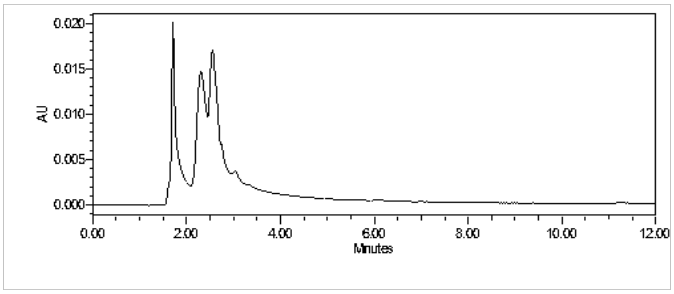

Figure 23 Reseda stamen.

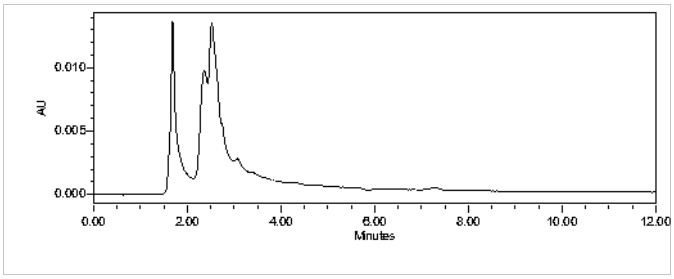

Figure 24 Reseda flower.

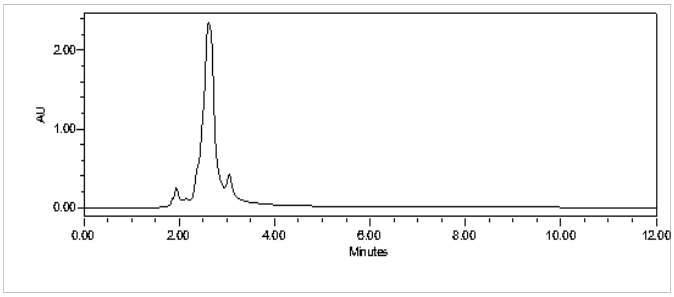

Figure 25 Reseda powder: Chromatogram taken on 268 nm.

Carthamus flower: Solvent System: 95:5 MeOH: DW, Run Time: 15 min, Sample prepared in MeOH, Chromatogram taken at 280nm (Figure 37).

Eucalyptus bark CO_2 extract: Solvent System: 95:5 MeOH: DW, Run Time: 15 min, Sample prepared in MeOH, Chromatogram taken at 255nm (Figure 38).

Eucalyptus bark CO_2 extract: Solvent System: 95:5 MeOH: DW, Run Time: 15 min, Sample prepared in MeOH, Chromatogram taken at 280nm (Figure 39).

Citation: Vankar PS, Shukla D, Spectrum of colors from reseda luteola and other natural yellow dyes. *J Textile Eng Fashion Technol.* 2018;4(2):106-119. DOI: 10.15406/jteft.2018.04.00127

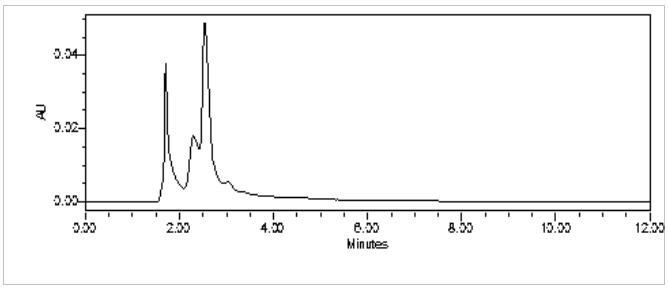

Figure 26 Reseda stamen: chromatogram taken on 268nm.

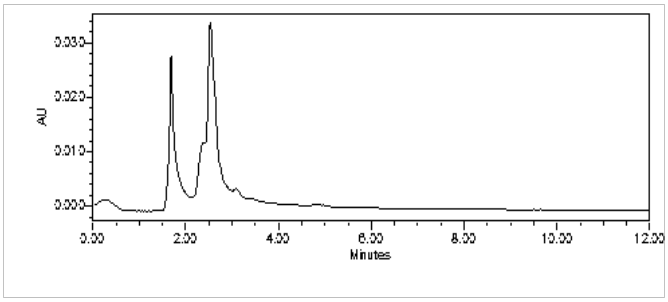

Figure 27 Reseda flower: chromatogram taken on 268nm.

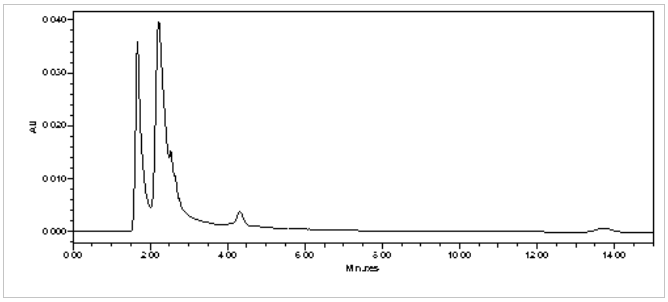

Figure 28 Cosmos flower: chromatogram taken on 255nm.

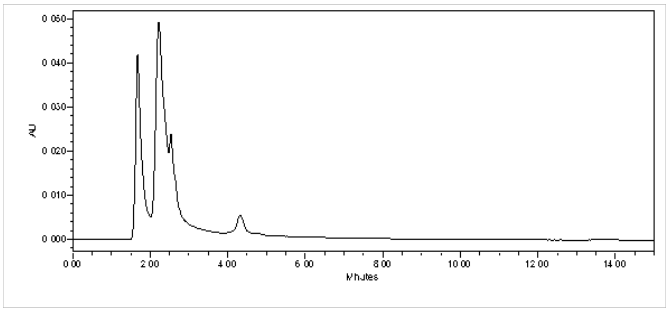

Figure 29 Cosmos flower: chromatogram taken on 280nm.

Punica: Solvent System: 95:5 MeOH: DW, Run Time: 15 min, Sample prepared in MeOH, Chromatogram taken at 255nm (Figure 40).

Punica: Solvent System: 95:5 MeOH: DW, Run Time: 15 min, Sample prepared in MeOH, Chromatogram taken at 280nm (Figure 41).

Curcuma powder: Solvent System: 95:5 MeOH: H_2O, Run Time: 15 min, Sample prepared in MeOH, Chromatogram taken at 255nm (Figure 42).

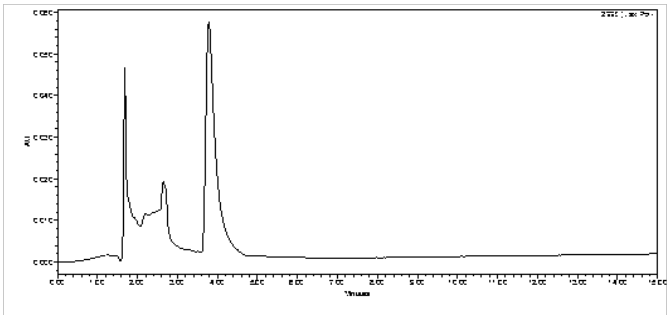

Figure 30 Punica granatum: chromatogram taken at 255nm.

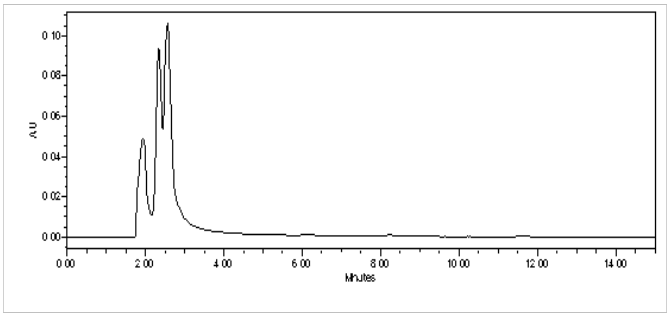

Figure 31 Thevetia (kaner flower): chromatogram taken at 255nm.

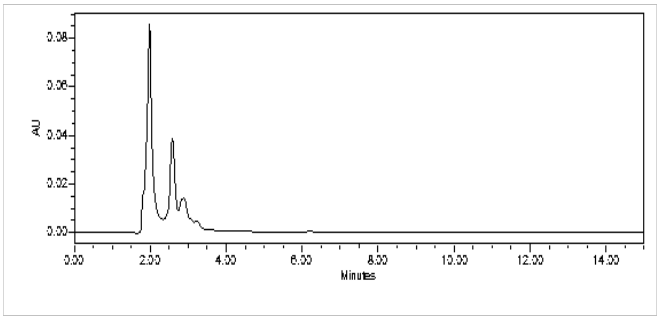

Figure 32 Gaillardia flower: chromatogram taken at 255nm.

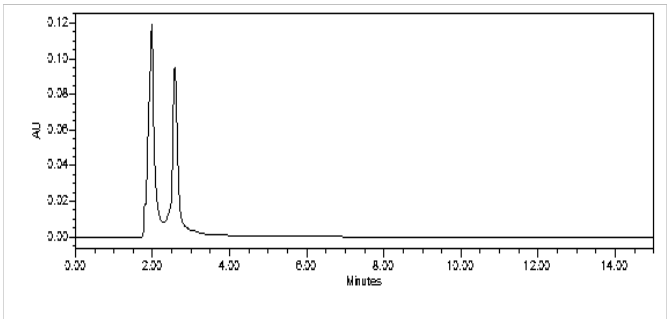

Figure 33 Gaillardia flower: chromatogram taken on 280nm.

Reseda powder: Solvent System: 97:3 MeOH: H_2O, Time: 15 min, Sample prepared in MeOH, Chromatogram taken on 255nm (Figure 43).

Reseda powder: Solvent System: 97:3 MeOH: H_2O, Time: 15 min, Sample prepared in MeOH, Chromatogram taken on 280nm (Figure 44).

Citation: Vankar PS, Shukla D, Spectrum of colors from reseda luteola and other natural yellow dyes. *J Textile Eng Fashion Technol.* 2018;4(2):106-119. DOI: 10.15406/jteft.2018.04.00127

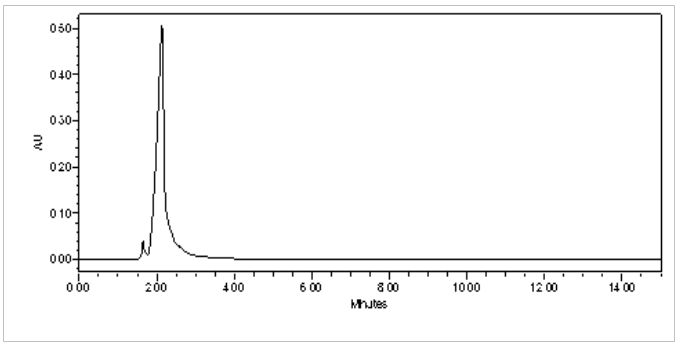

Figure 34 Cassia fistula (Amaltas flower): chromatogram taken on 255nm.

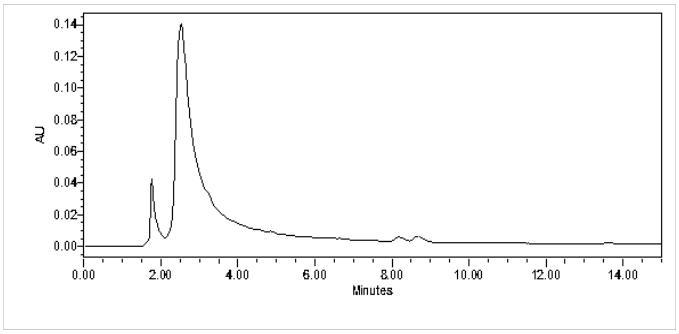

Figure 35 Tegetus flower: chromatogram taken at 255nm.

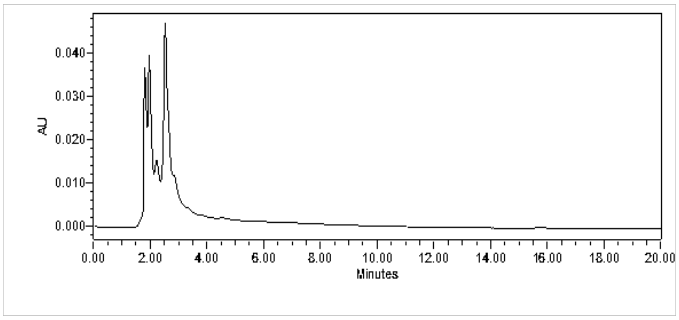

Figure 36 Carthamus flower: chromatogram taken at 255nm.

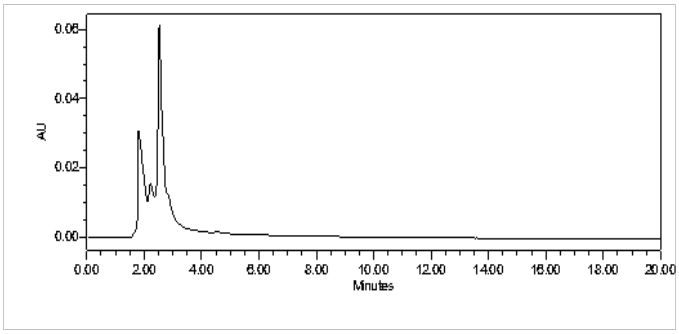

Figure 37 Carthamus flower: chromatogram taken at 280nm.

Reseda powder: Solvent System: 90:10 MeOH: EtOAc, Time: 10 min, Sample prepared in MeOH, Chromatogram taken on 255nm (Figure 45).

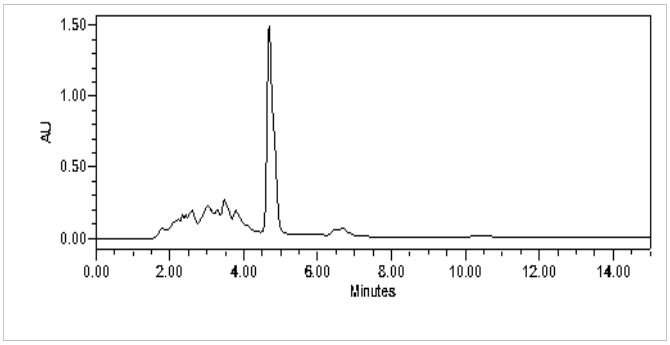

Figure 38 Eucalyptus bark CO2 extract: chromatogram taken at 255nm.

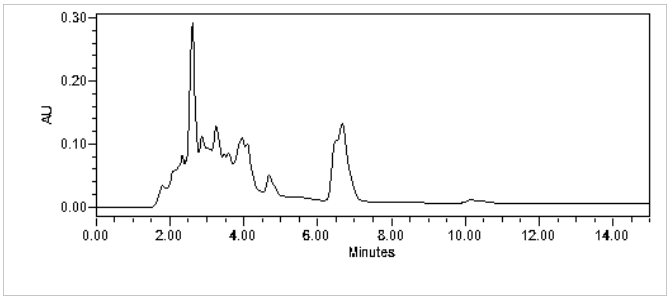

Figure 39 Eucalyptus bark CO2 extract: chromatogram taken at 280nm.

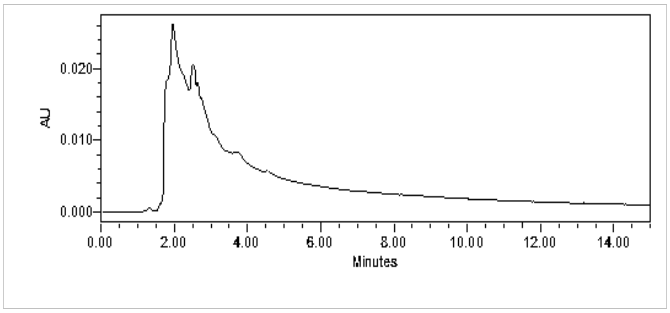

Figure 40 Punica: chromatogram taken at 255nm.

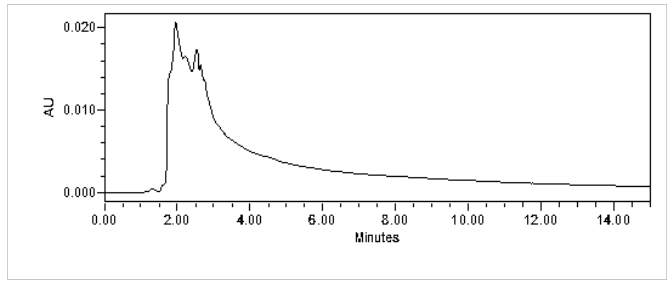

Figure 41 Punica: chromatogram taken at 280nm.

Reseda powder: Solvent System: 90:10 MeOH: EtOAc, Time: 10 min, Sample prepared in MeOH, Chromatogram taken on 280nm (Figure 46).

Solvent System: 97:03MeOH: H_2O, Run Time: 15 min, Sample prepared in MeOH, Chromatogram taken at 255nm (Figure 47).

Method-II: Solvent System: 10:90 EtOAc: MeOH, Run Time: 8 min,

Citation: Vankar PS, Shukla D, Spectrum of colors from reseda luteola and other natural yellow dyes. *J Textile Eng Fashion Technol.* 2018;4(2):106-119. DOI: 10.15406/jteft.2018.04.00127

Chromatogram taken at 255nm, Sample prepared in EtOAc (Figure 48).

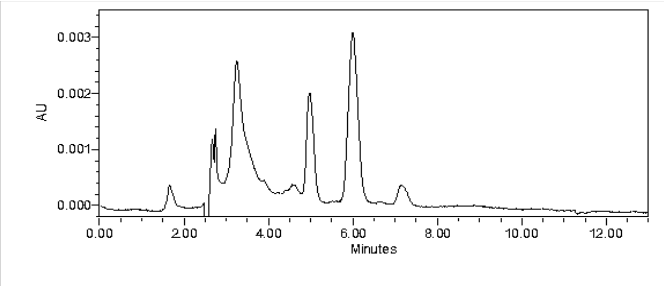

Figure 42 Curcuma powder: chromatogram taken at 255nm

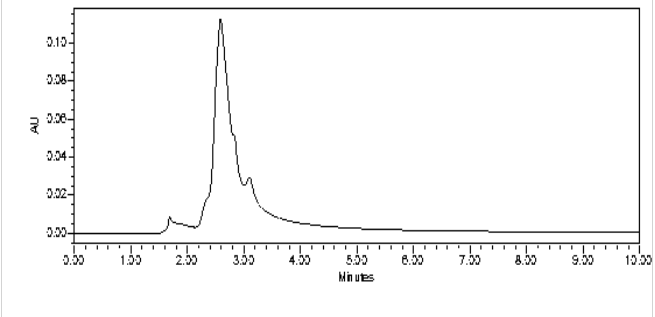

Figure 43 Reseda powder: chromatogram taken on 255nm.

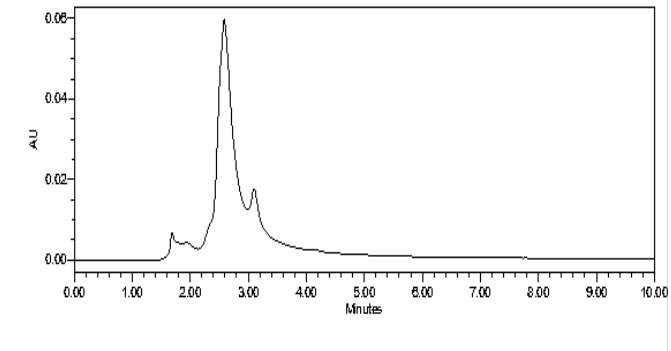

Figure 44 Reseda powder: chromatogram taken on 280nm.

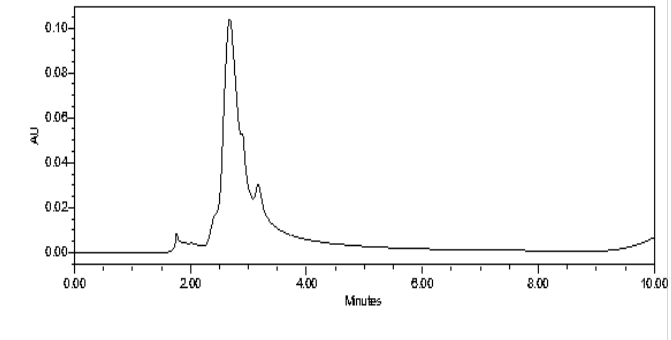

Figure 45 Reseda powder: chromatogram taken on 255nm.

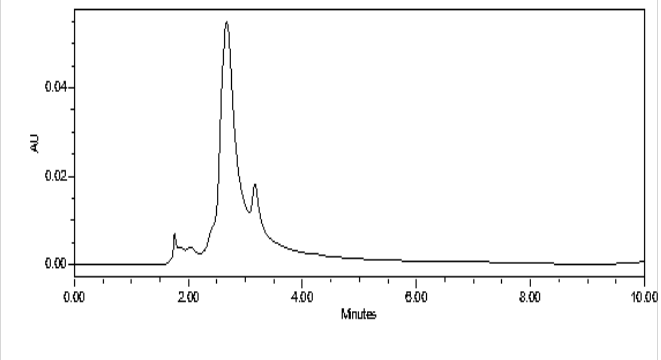

Figure 46 Reseda powder: chromatogram taken on 280nm.

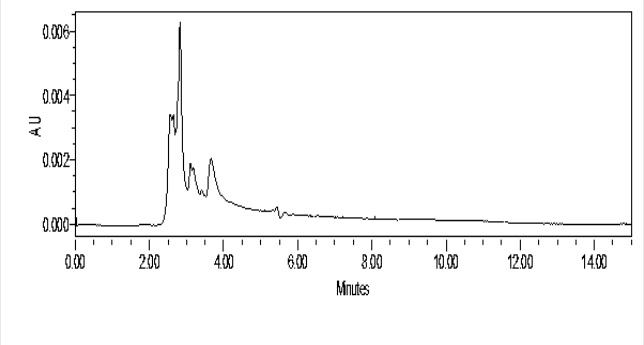

Figure 47 Solvent system: 97:03MeOH: H_2O, run time: 15 min, sample prepared in MeOH, chromatogram taken at 255nm.

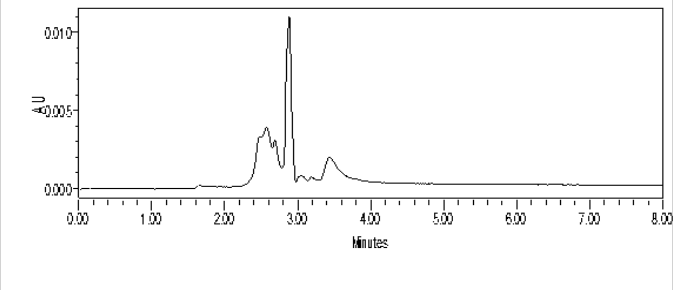

Figure 48 Solvent system: 10:90 EtOAc: MeOH, run time: 8 min, chromatogram taken at 255nm, sample prepared in EtOAc.

Cold dyeing experiments on silk and cotton with fresh *reseda* extract

Dyeing method for silk and cotton: In the present study Silk and Cotton fabrics were dyed with *Reseda* extract from stem, flower and leaves using alum and protease enzyme for silk as pretreatment, while for cotton only alum mordant was used.

Experimental materials

Pure Silk-The munga silk of GSM-45 fabric was scoured with solution containing 0.5 g/L sodium carbonate and 2 g/L non-ionic detergent (Labolene) solution at 40-45°C for 30 min, keeping the material to liquor ratio at 1:50. The scoured material was thoroughly

Citation: Vankar PS, Shukla D, Spectrum of colors from reseda luteola and other natural yellow dyes. *J Textile Eng Fashion Technol.* 2018;4(2):106-119. DOI: 10.15406/jteft.2018.04.00127

washed with tap water and air dried at room temperature. The scoured material was soaked in clean water prior to dyeing or mordanting. Cotton fabric was also scoured by standard process similar to the process mentioned herein Enzymes (Protease) was procured from TFF Speciality Chemicals, Kanpur. Alum as Metal mordant was procured from SD Fine Chemicals, Kanpur.

Dye material

The extract was prepared by using dry matter powder of stem .leaves and flowers (100g) were soaked in sufficient water 300 mL at 70-75˚C. The pH of the dyeing solution was 6.99.

Two step processes with premordanting followed by dyeing was carried out. Dyeing was carried by a stepwise dyeing process using either alum or enzyme- Protease, 1% w/w of the fabric). Similarly experiments were carried out for metal mordant--alum, and comparison of dyed swatches with enzyme treated swatches in the case of Silk fabric and only with alum 2% in the case of Cotton fabric.

The enzyme pre-treated silk fabric was used for dyeing with Reseda extract (10 %, w/w with respect to the wt. of the fabric). The dyeing time was 3 hours at a temperature of 30-40˚C.[19] Dyeing was also carried out for metal mordanted silk piece (in the ratio of 2 % mordant, w/w with respect to the fabric) in a similar way keeping the dyeing time and temperature same.

Similarly premordanting with alum followed by dyeing under same conditions was carried out for cotton fabric as well.

Results and discussion

Samples of Silk and cotton:

a. Sample 0: Silk control

b. Sample 1: Silk pretreated with Alum

c. Sample 2: Silk Pretreated with protease enzyme

d. Sample 3: Cotton control

e. Sample 4: Cotton pretreated with Alum (Figure 49) (Table 5).

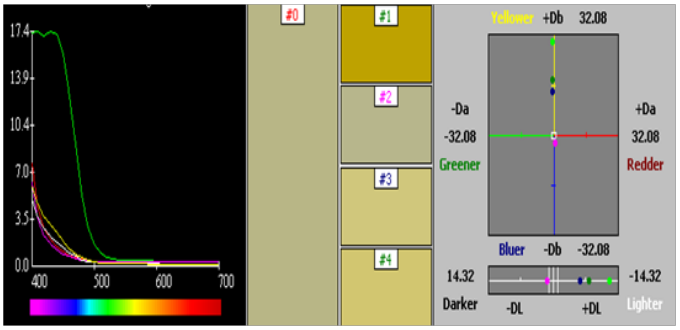

Figure 49 Samples of silk and cotton.

Table 5 Showing the CIE Lab and K/S values for the dyed silk and cotton fabric

CIE lab	Control silk	Silk +alum	Silk+protease	Cotton control	Cotton+alum
L*	73.226	85.545	71.876	80.487	90.197
a*	-7.092	-7.578	-6.517	-5.447	-7.904
b*	24.081	54.163	21.532	19.764	43.014
C*	25.104	54.691	22.497	20.501	43.734
H*	106.44	97.998	106.869	105.438	100.444
dE	---	32.51	2.941	---	25.316
K/S	12.796	102.663	11.341	6.208	53.968

The dyeing results of Silk and cotton show the following: While dyeing silk fabric with *Reseda* dye extract, alum mordanting is superior to use of Protease enzyme in terms of dE* values and K/S values. For cotton fabric with *Reseda* dye extract alum mordanting shows better K/S value as well as Chroma C* and Hue color H* higher than control sample (unmordanted cotton fabric). Thus through this experiment it can be concluded that for *Reseda* dye extract alum mordanting is most suited for both silk and cotton fabrics and use of enzyme did not give expected results. Thus we started exploring other metal mordants with Reseda dye extract and the results are given below.

Dyeing experiments on silk and cotton with *reseda* samples with different metallic mordants

In the present study Silk and Cotton fabrics were dyed with *Reseda* extract derived from stem, leaves and flowers using alum, copper sulphate, ferrous sulphate, potassium dichromate and stannous chloride as mordant in two step dyeing process. Silk and Cotton fabrics were also scoured by standard process similar to the process mentioned earlier. Metal mordants were procured from SD Fine Chemicals, Kanpur.

Dye material

The extract was prepared by using dry matter powder of stem leaves and flowers (100g) was soaked in sufficient water 300 mL at 70-75˚C. The pH of the dyeing solution was 6.99.

Two step processes with premordanting followed by dyeing was carried out. Dyeing was carried by a stepwise dyeing process using metal mordants mentioned above in 2 %. The pre-mordanted silk fabric was used for dyeing with Reseda extract. The dyeing time was 3 hours at a temperature of 30-40˚C. Dyeing was also carried out for metal mordanted cotton in a similar way keeping the dyeing time and temperature same (Figures 50−53) (Table 6) (Table 7).

Citation: Vankar PS, Shukla D, Spectrum of colors from reseda luteola and other natural yellow dyes. *J Textile Eng Fashion Technol.* 2018;4(2):106-119. DOI: 10.15406/jteft.2018.04.00127

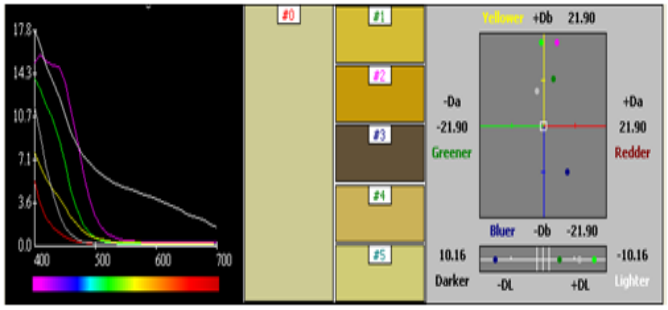

Figure 50 Samples of Silk and cotton: Standard 0: Control cotton Unmordanted, Sample 1: Alum mordanted, Sample 2: Copper sulphate mordanted, Sample 3: Ferrous Sulphate mordanted, Sample 4: Potassium dichromate mordanted, Sample 5: Stannous chloride mordanted.

Figure 51 Dyeing cotton with Reseda luteola.

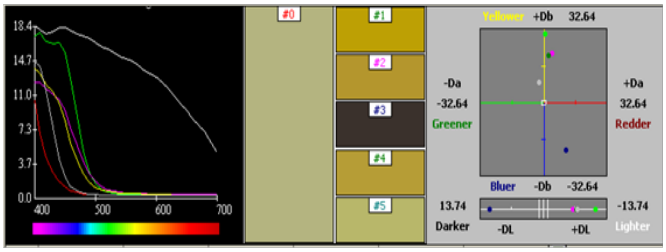

Figure 52 Samples of Silk and cotton: Standard 0: Control Silk Unmordanted, Sample 1: Alum mordanted, Sample 2: Copper sulphate mordanted, Sample 3: Ferrous Sulphate mordanted, Sample 4: Potassium dichromate mordanted, Sample 5: Stannous chloride mordanted.

The dyeing results of Silk and cotton show the following: Dyeing silk and cotton fabrics with *Reseda* dye extract different mordanting shows highest K/S for ferrous sulphate mordanted fabrics. For cotton fabric with *Reseda* dye extract the order of effective dyeing in terms of K/S values is Ferrous sulphate > >Copper> Alum> Potassium dichromate> Stannous chloride. With silk fabric with *Reseda* dye extract the order of effective dyeing in terms of K/S values is Ferrous sulphate > > Alum> Copper> Potassium dichromate> Stannous chloride. We did more experiments with different surfactants and Reseda dye.

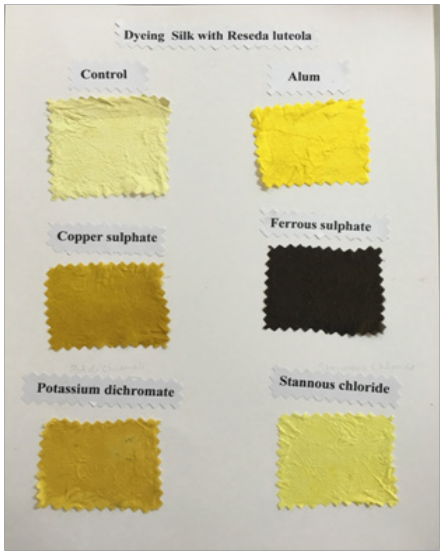

Figure 53 Dyeing cotton with Reseda luteola.

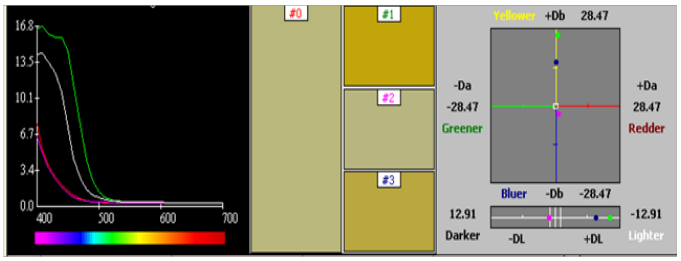

Figure 54 Silk and Alum, SDBS and CTAB Surfactants.

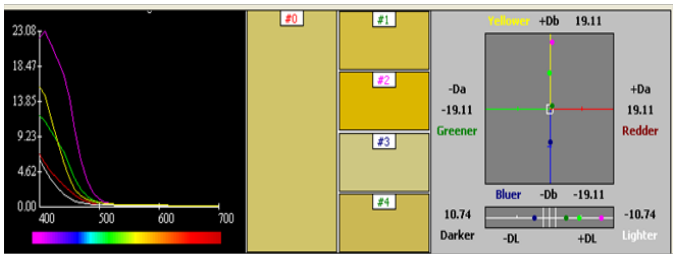

Figure 55 Cotton and Knitted and Alum, SDBS and CTAB Surfactants.

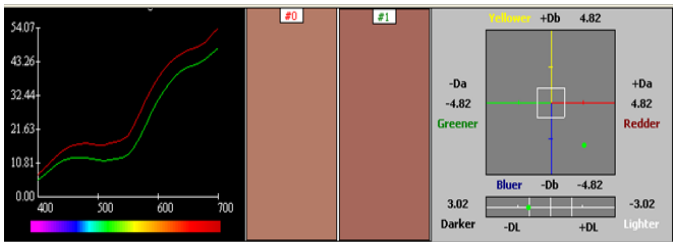

Figure 56 Standard 0: Control cotton Unmordanted Rubia+Reseda, Sample 1: Alum mordanted Rubia+ Reseda.

Dyeing experiments on silk and cotton with *reseda* samples with alum metallic mordant and surfactant

In the present study Silk and Cotton fabrics were dyed with *Reseda* extract using alum, as well as surfactants such as Sodium

Citation: Vankar PS, Shukla D, Spectrum of colors from reseda luteola and other natural yellow dyes. *J Textile Eng Fashion Technol.* 2018;4(2):106-119. DOI: 10.15406/jteft.2018.04.00127

salt of Dodecylbenzenesulphonic acid (SDBS) and Cetyl trimethyl ammonium bromide (CTAB) as pretreatment have been used in two step dyeing process.

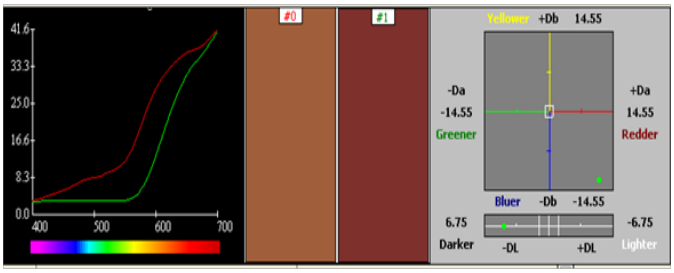

Figure 57 Standard 0: Control Silk Unmordanted Rubia+Reseda, Sample 1: Alum mordanted Rubia+ Reseda.

Silk and Cotton along with knitted variety D100% fabrics were also scoured by standard process similar to the process mentioned earlier. Metal mordant and Surfactants were procured from SD Fine Chemicals, Kanpur.

Pretreatment with surfactant

Aqueous solution of the surfactant (0.02 gm/ml) was prepared in 1:30 M:L. Silk fabric was dipped in 25 ml of surfactant solution and heated on water bath at 60-70˚C for 30-45 mins. Cotton was heated for 40 mins at 80-90˚C. Then the fabrics were left for aerial oxidation.

Dyeing

After this Silk and Cotton were dipped in 25 ml of 10 % solution of Reseda dye extract using 0.5 % NaOH separately at 1:30 MLR. Two step processes with premordanting followed by dyeing was carried out. Dyeing was carried by a stepwise dyeing process using metal mordants as well as surfactants SDBS and CTAB mentioned above. The pre-mordanted silk fabric was used for dyeing with Reseda extract. The dyeing time was 3 hours at a temperature of 40°C. Dyeing was also carried out for metal mordanted cotton and surfactant pre treated in a similar way keeping the dyeing time and temperature same. .Dyed fabrics were washed with water, followed by mild soap and finally with water (Figure 54) (Figure 55) (Table 8) (Table 9).

Table 6 Cotton - Metal mordanting

CIE lab	Control	$KAL(SO_4)_2$	$CuSO_4$	$FeSO_4$	$K_2Cr_2O_7$	$SnCl_2$
L*	80.546	88.706	86.344	72.851	83.227	86.421
a*	-7.598	-8.289	-2.85	0.804	-4.11	-9.836
b*	27.214	47.114	47.026	16.243	38.423	35.451
C*	28.255	47.838	47.112	16.263	38.642	36.79
H*	105.629	100.01	93.503	87.131	96.139	105.537
dE	--	21.519	21.182	15.817	12.041	10.362
K/S	8.564	51.288	92.946	124.162	26.535	19.267

Table 7 Silk- Metal mordanting

CIE lab	Control	$KAL(SO_4)_2$	$CuSO_4$	$FeSO_4$	$K_2Cr_2O_7$	$SnCl_2$
L*	72.148	83.523	78.589	60.407	79.303	79.547
a*	-8.014	-7.114	-3.629	3.577	-5.525	-10.586
b*	26.161	56.8	48.369	5.036	47.214	35.318
C*	27.361	57.244	48.505	6.177	47.536	36.87
H*	107.061	97.172	94.325	54.592	96.708	106.715
dE	--	32.695	23.535	26.804	22.374	12.05
K/S	16.516	100.694	68.27	268.484	62.21	31.919

Table 8 Silk and alum, SDBS and CTAB surfactants

CIE lab	Control	$KAL(SO_4)_2$	SDBS	CTAB
L*	73.305	84.219	71.99	81.373
a*	-8.397	-7.482	-7.039	-8.363
b*	30.219	56.691	27.278	46.341
C*	31.364	57.183	28.172	47.09
H*	105.559	97.552	104.55	100.262
dE	--	28.648	3.496	18.028
K/S	15.9552	93.24	14.84	59.032

Citation: Vankar PS, Shukla D, Spectrum of colors from reseda luteola and other natural yellow dyes. *J Textile Eng Fashion Technol.* 2018;4(2):106-119. DOI: 10.15406/jteft.2018.04.00127

Table 9 Cotton and knitted and alum, SDBS and CTAB surfactants

CIE lab	Control cotton	$KAL(SO_4)_2$	Knitted cotton alum	SDBS	CTAB
L*	78.323	83.267	87.066	75.761	81.118
a*	-7.418	-7.493	-6.786	-7.23	-6.723
b*	45.826	55.103	62.934	37.493	46.547
C*	46.423	55.61	63.299	38.184	47.03
H*	99.227	97.777	96.188	100.947	98.252
dE	--	10.512	19.223	8.72	2.969
K/S	18.712	42.248	98.436	12.615	38.63

Table 10 Cotton alum reseda+ rubia dyes

	Standard 1	Batch 1
L*	56.86	55.841
a*	20.381	22.888
b*	19.29	16.474
c*	28.062	28.2
H*	43.407	35.731
dE*	----	3.906
DL*	----	-1.019
Da*	----	2.507
Db*	----	-2.816
DC*	----	0.138
DH*	----	-3.768
KIS	29.2786	43.568
RFL	10.705	8.508
%	100	148.805

Table 11 Silk alum reseda+ rubia dyes

	Standard	Batch 1
L*	46.994	42.242
a*	23.245	34.59
b*	31.817	19.271
c*	39.404	39.596
H*	53.827	29.112
dE*	----	17.57
DL*	----	-4.752
Da*	----	11.345
Db*	----	-12.546
DC*	----	0.192
DH*	----	-16.914
KIS	83.1575	199.9764
RFL	3.958	2.936
%	100	240.479

The dyeing results of Silk and cotton show the following: Cotton fabrics with *Reseda* dye extract along with use of surfactant did not show any improvement, infact alum mordanting still remained the best, and Knitted fabric showed better result that ordinary cotton fabric.

a. While dyeing Silk fabric with *Reseda* dye extract the order of effective dyeing in terms of K/S values Alum> CTAB> SDBS, infact silk fabric showed the order of effective dyeing in terms of K/S values SDBS was even lower than unmordanted fabrics K/S value.

b. We have also used Reseda with Rubia dye to develop different shades of dyed fabric as show below

Dyeing experiments on silk and cotton with *reseda* and rubia dyes samples with alum metallic mordant

In the present study Silk and Cotton fabrics were dyed with ***Reseda*** + **Rubia** dyes (0.35 gm+ 0.65 gm respectively) extract using alum, as pretreatment in two step dyeing process.

Silk and Cotton fabrics were also scoured by standard process similar to the process mentioned earlier. Metal mordant (Alum) was procured from SD Fine Chemicals, Kanpur.

Dye material

The extract was prepared by using powder 1.0g was soaked in sufficient water 150 mL at 70-75˚C. Mass to liquor ratio: 1.0 g in150 mL at 70˚C for 1.5 hrs.

Two step processes with premordanting followed by dyeing was carried out. Dyeing was carried by a stepwise dyeing process using metal mordant alum in 2 %. The pre-mordanted silk fabric was used for dyeing with *Reseda* + Rubia dyes extract. The dyeing time was 3 hours at a temperature of 50˚C. Dyeing was also carried out for metal mordanted cotton pre treated with alum in a similar way keeping the dyeing time and temperature same (Figure 56) (Figure 57) (Table 10) (Table 11).

The dyeing results of Silk and cotton show the following:

i. While dyeing cotton fabrics with *Reseda* + Rubia dyes extract along with use of alum mordanting K/S values showed improvement.

ii. While dyeing Silk fabric with *Reseda*+ Rubia dyes extract, alum mordanting in terms of K/S values very pronounced improvement.

Citation: Vankar PS, Shukla D, Spectrum of colors from reseda luteola and other natural yellow dyes. *J Textile Eng Fashion Technol.* 2018;4(2):106-119. DOI: 10.15406/jteft.2018.04.00127

Conclusion

Among many yellow colored natural dyes Reseda seems to have the most pronounced dyeing ability and is also very good when combined with other dyes. Most of the yellow dye plants contain colorant molecule from the group of hydroxyl flavones, however the combination of components may differ from plant to plant and species to species. It shows vast difference in wash, light fastnesses, solubility and dyeability of the natural extracts. It is very apparent that among 10 different sources, Reseda excels in all the three parameters.

While analyzing Reseda by HPLC, it showed better separation by method-II, however the resolution needs to be further worked out, method –I is not so good. Better separation of peaks can be seen herein.

Dyeing Silk fabric with *Reseda* dye extract the order of effective dyeing in terms of K/S values Alum> CTAB> SDBS, in fact silk fabric showed the order of effective dyeing in terms of K/S values SDBS was not found to be good as pretreatment chemical for Reseda. Reseda extract showed bright yellow color with alum, olive green with copper sulphate and dark brown with ferrous sulphate.

Acknowledgements

None.

Conflict of interest

Authors declare there is no conflict of interest in publishing the article.

References

1. Gaspar H, Moiteiro C, Turkman A, et al. Influence of soil fertility on dye flavonoids production in weld (Reseda luteola L.) accessions from Portugal. *J Sep Sci*. 2009;32(23−24):4234−4240.
2. Sun J, Wu Y, Xiao D, et al. Spectrofluorimetric determination of aluminum ions via complexation with luteolin in absolute ethanol. *Luminescence*. 2014;29(5):456−461.
3. Cerrato A, De Santis D, Moresi M. Production of luteolin extracts from Reseda luteola and assessment of their dyeing properties. *J Sci Food Agric*. 2002;82(10):1189−1199.
4. Sahu R, Saxena J Isolation of Flavonoid derivative from Curcuma longa. *World J Pharm Res*. 2014;3(7): 740−745.
5. Averett JE, Beaman WC. Chemotaxonomy and flavonoids of Gaillardia section Hollandia (Asteraceae-Helenieae). *Plant Systematics & Evolution*. 1975;123(3):237−240.
6. Save SA, Lokhande RS, Choudhary AS. Thevetia peruviana, a Good luck tree. *IPP*. 2015;3(3):586−606.
7. Vankar PS. *Handbook on Natural dyes for Industrial Applications*. NIIR project consultancy services; 2016. 432 p.
8. Vankar PS, Tiwari V, Shankar R, et al. *Carthamus tintorius* (Safflower), a commercially viable Dye for Textile. *Asian Dyers.* 2004;65−69.
9. Yasukawa K, Kasahara Y Effects of Flavonoids from French Marigold (Florets of Tagetes patula L.) on Acute Inflammation Model. *Int J Inflam*. 2013;2013:309493.
10. Zhao X, Yuan Z, Fang Y, et al. Flavonols and Flavones Changes in Pomegranate (Punica granatum L.) Fruit Peel during Fruit Development. *J Agr Sci Tech*. 2014;16(suppl):1649−1659.
11. Luximon-Ramma A, Bahorun T, Soobrattee MA, et al. Antioxidant Activities of Phenolic, Proanthocyanidin, and Flavonoid Components in Extracts of *Cassia fistula*. *J Agri Food Chem*. 2002;50(18):5042−5047.
12. Kurkin VA, Kharisova AV. Flavonoids of Carthamus tinctorius flowers. *Chemistry of Natural compounds*. 2014;50(3):446−448.
13. Santos SAO, Villaverde JJ, Silva CM, et al. Supercritical fluid extraction of phenolic compounds from *Eucalyptus globulus* Labill bark. *J of Supercritical Fluids*. 2012;71:71−79.
14. Vankar PS, Tiwari V, Ghorpade, B. Supercritical Fluid Extraction of Natural dye from Eucalyptus Bark for cotton dyeing in microwave and sonicator. *Canadian Textile J*. 2002;119(5):32−35.
15. Smith GJ, Thomsen SJ, Markham KR, et al. The photostabilities of naturally occurring 5-hydroxy flavones, flavanols and their glycosideand their Aluminium complexes. *J Photochem. Photobiol A*. 2000;136(1−2):87−91.
16. Amat A, Clementi C, Miliani C, et al. Complexation of apigenin and luteolin in weld lake: a DFT/TDDFT investigation. *Phys Chem Phys*. 2010;12(25):6672−6684.
17. Kaiser R Quantitative Analyses of Flavonoides in Yellow dye plant species Weld (*Reseda luteola L*) and Sawwort (*Serratula tinctoria L*.). *Angew Bot*. 1993;67(3−4):128−131.
18. Ren J, Meng S, Lekka CE, et al. Complexation of Flavonoids with Iron: Structure and Optical Signature. *J Physical Chemistry*. 2008;112(6):1845−1850.
19. Vankar PS, Shukla D, Wijayapala S. Innovative Silk dyeing using Enzyme and Rubia cordifolia extract at room temperature. *Pigment & Resin Technol*. 2017;46(4):296−302.