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Extraction of Indigo dye from *Couroupita guianensis* and its application on cotton fabric

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Abstract

Couroupita guianensis (Cannon ball tree) flowers and fruits are known to contain indigotin and indirubin. In this study an attempt is made to optimize the period for effective extraction of indigo from *Couroupita guianensis* fruit and application of the crude extract on the cotton fabric. Fermentation of the *Couroupita guianensis* fruit pulp for 5 days gave intense bluer pigment and hence was selected as the optimum period for fermentation. Major colouring components from *Couroupita guianensis* fruit extract were separated by thin layer chromatography using single solvent system, chloroform; which gives three pigments, violet, blue and pink with the R_f values of 0.88, 0.57, and 0.25 respectively. The R_f values, the wavelength of maximum absorption from UV-Visible spectroscopy, Fourier Transform Infrared (FTIR) spectrum and ^{1}H Nuclear magnetic resonance (^{1}H NMR) spectrum of blue pigment matched with that of synthetic indigo standard and that of the pink pigment matched with reported values of indirubin which is an isomer of indigotin. The % purity of indigo dye was found to be 26.46%. Dyeing of cotton fabric was carried out with the crude dye powder which gave comparable fastness properties vis-a-vis synthetic indigo.

Keywords: *Couroupita guianensis*; Extraction; Structural analysis; Indigotin; Indirubin; Dyeing

Introduction

In recent years, there has been a revival of the use of dyes and colours of natural origin for coloring food, pharmaceutical, cosmetic and textile products. This increasing demand for the material of natural origin is because of the toxic nature of many of the synthetic dyes. Amongst the natural dyes which are becoming widely recognized throughout the world, indigo is the one which is oldest known and commercialized natural dye. Indigo is a derivative of colourless glucoside of the enol form of indoxyl e.g. indican (Laitonjam and Wangkheirakpam 2011). Indigo is a dye with a long history and because of the contemporary popularity of indigo dyed denim, it remains an important industrial product (Garcia-Macias and John 2004). Throughout history, indigo was derived from various plants for example Dyer's Woad (*Isatis tinctoria* L.) in Europe (Parmar et al. 1996). Also it is obtained from plant materials such as *Baphicacanthus cusia* Brem., *Indigofera suffruticosa* Mill, *Polygonum tinctorium* Palette, *Isatis indigotica* Fort, *Lonchocarpus cyanescence* (Laitonjam and Wangkheirakpam 2011). Until the commercialization of the synthetic product in the late 19th century, indigo was produced entirely from plants (Garcia-Macias and John 2004). Today, the indigo used in commercial dyeing of denim yarn is no longer of natural origin.

After more than 12 years of research, a method of laboratory synthesis of indigo was discovered by Adolf Von Baeyer in 1880. The first commercial form of indigo based on Von–Baeyers's discovery appeared in the market in 1897 and after the turn of the century, synthetic indigo gradually replaced the natural indigo dye world-wide (Parmar et al. 1996). With increasing concern for sustainability and a demand from consumers for naturally sourced products, there is a revival of interest in natural indigo as an agricultural crop product (Garcia-Macias and John 2004). The current consumption of the dye is enormous due to the popularity of blue jeans, which are dyed with indigo (Vuorema 2008). In this study, an attempt has been made to extract natural indigo dye first time from *Couroupita guianensis* fruit by fermentation technique and to use the obtained dye for colouration of textiles.

Couroupita guianensis possesses antibiotic, antifungal, antiseptic and analgesic qualities. It belongs to the family Lecythidaceae, commonly known as cannon ball tree. This is truly an amazing tree which bears large showy flowers almost through the year, on the trunk and not on branches like most other trees. The tree also produces globular brown woody, indehiscent, amphisarcun (double fleshy) fruits of an astonishing size, almost the size of a human head. Size and weight of a mature fruit is approximately 24 cm in diameter and 1450 gms respectively. It is widely planted in tropical and subtropical botanical gardens as an ornamental tree (Regina and Uma Rajan 2012). The tree gets its common English name *Cannon Ball Tree* from its heavy, brown, spherical fruits resembling cannon balls. Herein the extraction of dye was carried out by the fermentation of *Couroupita guianensis* fruit pulp in water and dried to get crude dye powder indigo. Further, separation and identification of the major components present in the crude dye powder was done.

Also a successful attempt has been made to dye the industrially bleached woven cotton fabric with the obtained crude dye powder. Indigo dye is considered as a vat dye for dyeing textile materials such as cellulose and silk fibres. The dyed fabrics were then evaluated for their colour strength and fastness properties viz., washing, rubbing and light fastness using standard test methods.

Method

Materials and methods

Sodium hydroxide, sodium hydrogen sulphite, chloroform, hexane, methanol were supplied by S.D. Fine Chemicals Ltd., Mumbai. Synthetic indigo blue dye was supplied by Colorband Dyestuff (P). Ltd., Mumbai. Industrially bleached woven cotton fabric (100%) was purchased from Premier Mills, Mumbai. Rota dyer machine from Rossari Lab Tech., Computer Colour Matching System (Spectra Scan 5100+, Data colour International, Lawrenceville, NJ, USA), TECHCOMP UV-Visible Spectrophotometer (UV–VIS 8500), FTIR-8400S, Fourier Transform Infrared Spectrophotometer, Shimadzu, Crock meter from Rossari Lab Tech Pvt. Ltd. (Mumbai, India) and Light Fastness Tester by Q-Sun's Xenon Arch Light Fastness Tester (Cleveland, OH, USA) were used for testing.

Plant material

The fruit of *Couroupita guianensis* was collected in April-June 2011 from Institute of Chemical Technology (ICT) campus, behind Textile Department, Matunga, Mumbai.

Size of a mature fruit was approximately 24 cm in diameter. The fruit weighed approximately 2 kg, outer part being 1 kg and inner part being 1 kg.

Extraction of Indigo Dye

The rind of the cannon ball was separated by breaking the fruit. The fruit pulp was grinded and kept in glass jars with the addition of water (235 gm/150 ml) for fermentation at room temperature for several (a period of 1, 2, 3, 4 and 5 days) days. During the fermentation, the fruit pulp was turned upside down at the end of each day. After fermentation, the blue mass was filtered and the filtrate was centrifuged at 3000 rpm for 20 minutes to get the crude dye paste which was then dried at 50°C to get crude dye powder.

Separation of major components by thin layer chromatography

The crude dye powder was dissolved in chloroform and plotted on Thin Layer Chromatography plate (Silica Gel $60F_{254}$). The solvent used for eluting the plate was chloroform. No developing reagent was used as the components of the crude dye were coloured and the separated bands were analyzed visually.

Separation of major components by column chromatography

Column chromatography is a commonly used purification technique in laboratories. Done right, it can simply and quickly isolate desired components from a crude product. A glass column of 36 cm × 2.5 cm containing 25-30 gm of silica gel (Silica gel 60–120 Mesh LR) saturated with chloroform was used to separate the major components. The column was eluted with chloroform and the fractions obtained were checked for their purity by performing thin layer chromatography. The fractions obtained were then taken for further studies of chemical structure analysis i.e. UV-Visible spectrophotometery, FTIR and ^{1}H NMR.

Characterization studies

Structural analysis of the major components

The fractions collected from the column chromatography were further taken for the UV-Visible spectrophotometery and FTIR spectroscopy for their functional group detection and ^{1}H NMR for the structure elucidation.

UV-visible spectroscopy

The pure fractions obtained in chloroform from column chromatography were analyzed for their maximum wavelength i.e. λ_{max}. The UV-Visible spectra were recorded in the range of 400–700 nm on TECHCOMP UV-Visible Spectrophotometer (UV–VIS 8500).

FT-IR spectroscopy

The Infrared Spectra were obtained on FTIR-8400S, Fourier Transform Infrared Spectrophotometer, Shimadzu, to find the functional groups of these purified pigments.

NMR spectroscopy

Chemical structure analysis of the purified pigments was done by performing ^{1}H NMR spectroscopy. The data of the ^{1}H NMR for blue and pink pigments using $CDCl_3$ as a

solvent and proton (s2pul) as pulse sequence were collected on Balaji-vnmrs in Tata Institute of Fundamental Research, Mumbai.

Percentage purity of indigo dye

In order to calculate the % purity of indigo dye, testing method was followed as given by Parmar et al. 1996. The method is as follows:

Initially, 0.5gm of indigo dye (crude indigo dye) along with the addition of 20 ml of conc. H_2SO_4 and 3gm of white quartz was taken in a flask to prevent the bumping of reaction. The flask was kept in water bath at 70-75°C for 1 h. After 1 h, 300 ml of distilled water and 10 ml of 10% $BaCl_2$ solution was added and the total solution was made upto 500 ml. After 30 mins the $BaSO_4$ formed and the impurities present in the dye settle down at bottom of the flask. 20 ml of supernatant solution and 150 ml of distilled water was taken in the conical flask and this solution was titrated with 0.02 N $KMnO_4$ solution. Knowing the volume of $KMnO_4$ required to reach the end point (appearance of pale yellow) the % purity was calculated.

Dyeing of Cotton Fabric

Indigo being a vat dye, is insoluble in aqueous medium. To make it soluble, indigo is converted into its soluble form leuco-indigo by providing alkaline conditions (Pathak and Madamwar 2010). The soluble indigo, viz., leuco-indigo, was prepared by reducing crude indigo dye powder viz., 50% (12.5 gm), 100% (25 gm) and 150% (37.5 gm) with 3 ml of 40% (w/v) sodium hydroxide and 1gm sodium hydrosulphite. The total liquor volume was made-up by using blank vat solution (2 g l^{-1} sodium hydrosulphite and 2 g l^{-1}sodium hydroxide) until the solution was completely clear having light yellow-green colour. Dyeing was carried out at room temperature for 1 h keeping 1: 25 material-to-liquor ratio. After dyeing, the samples were air oxidized, rinsed and dried at room temperature and taken for further study.

Colour strength measurement

Estimation of the colour strength of dyed fabrics was carried out by determining K/S values using a Computer Colour Matching System (Spectra Scan 5100+, Data colour International, U.S.A). Kubelka – Munk K/S function is given by:

$$\frac{K}{S} = \frac{(1-R)^2}{2R}$$

where "R" is the reflectance at complete opacity, "K" is absorption coefficient and "S" is the scattering coefficient.

Extraction of colouring material from dyed fabric and its analysis by Thin Layer Chromatography

Colouring material from the dyed fabric was extracted with chloroform. Absorbance of the extracted solution was measured by TECHCOMP UV-Visible Spectrophotometer (UV–VIS 8500). Extracted colouring component from the dyed fabric sample in chloroform was spotted on Thin Layer Chromatography plate along with standard synthetic

Table 1 The characteristic of crude indigo paste in different periods of maceration in water

Plant material	Period of fermentation (days)	Colour of fermented solution	Colour of paste	Dye content from 100 gm of fermented mass (gm)
Couroupita guianensis	1	Grey	Grey-black	0.228
	2	Grey blue	Grey-black	0.230
	3	Grey blue	Grey-black	0.228
	4	Blue	Blue black	0.230
	5	Intense Blue	Blue black	0.230
	6	Bluish violet with fungus	Blue black	–

indigo. The plate was eluted with single solvent system, chloroform, in a glass vessel and was examined visually.

Fastness properties

The dyed samples were tested according to ISO standard test methods. Colourfastness to washing was assessed as per ISO 105-CO2:1989, Colourfastness to rubbing on Crock meter as per ISO 105-X12:2001 and Colourfastness to Light on Q-Sun's Xenon Arc Light Fastness Tester as per ISO 105-B02:1994.

Results and discussion

Optimization of dye extraction period

Although the yield of crude dye obtained for different fermentation period was practically same as shown in Table 1, intense bluer pigment was obtained only when the fruit pulp was fermented for 5 days. Beyond 5 days of fermentation, fungal growth was observed hence, the period of 5 days fermentation was taken as optimum. The yield of crude indigo dye obtained from *Couroupita guianensis* fruit was found to be 0.236% and that of the Indigofera Tinctoria, traditional source of natural indigo, is reported to be 0.4% (Bhattacharrya 2010).

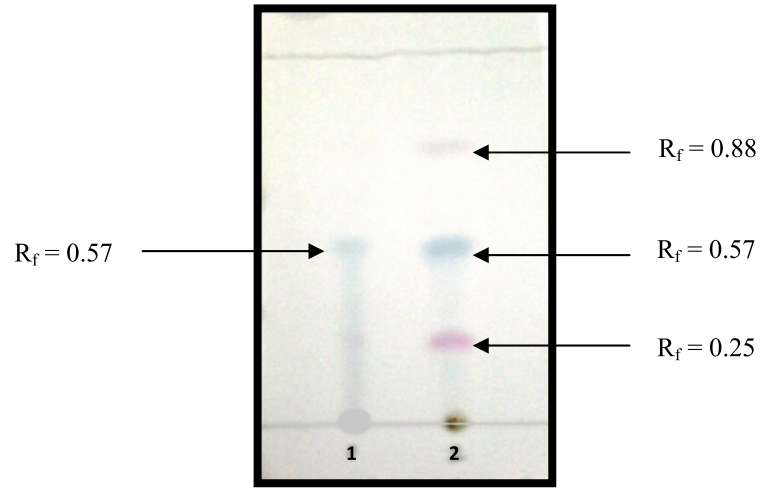

Figure 1 TLC of standard indigo dye and the crude extract from *Couroupita guianensis fruit*. Spot 1: standard synthetic indigo. Spot 2: crude indigo extract from *Couroupita guianensis* fruit.

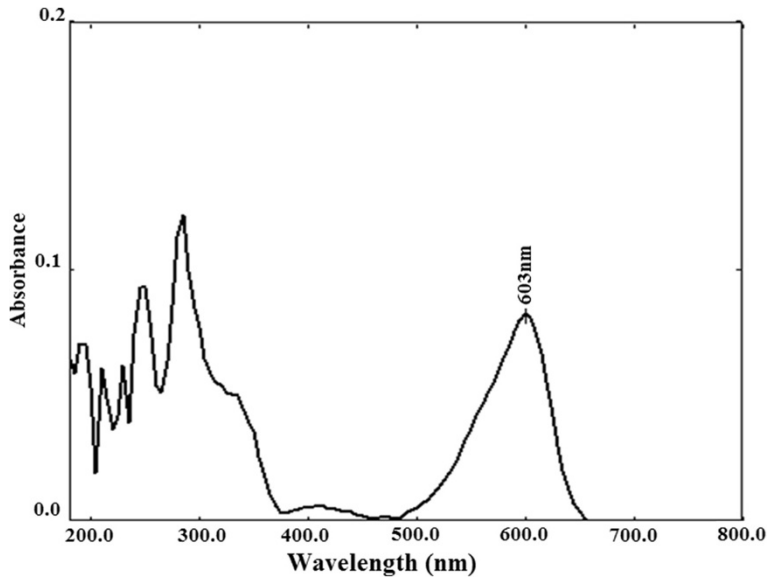

Figure 2 UV visible spectrum of purified blue pigment showing maximum absorbance at 603 nm.

Extraction, purification and identification of major components from crude dye powder

Identification of pigments by Thin layer chromatography

The separation of major components from crude dye powder by thin layer chromatography using chloroform as single solvent system gave three spots viz., violet, blue, pink with the R_f values, 0.88, 0.57, and 0.25 respectively as shown in Figure 1. Also from figure

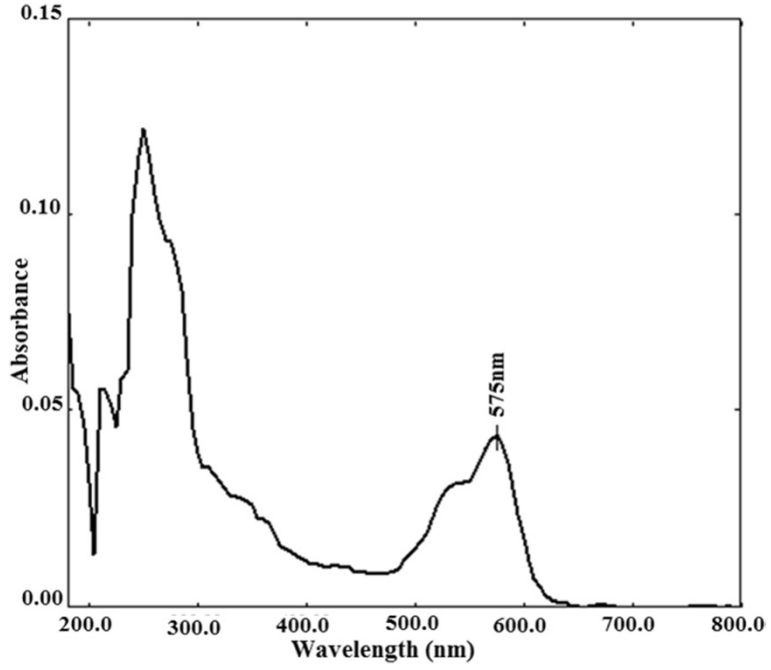

Figure 3 UV visible spectrum of purified violet pigment showing maximum absorbance at 575 nm.

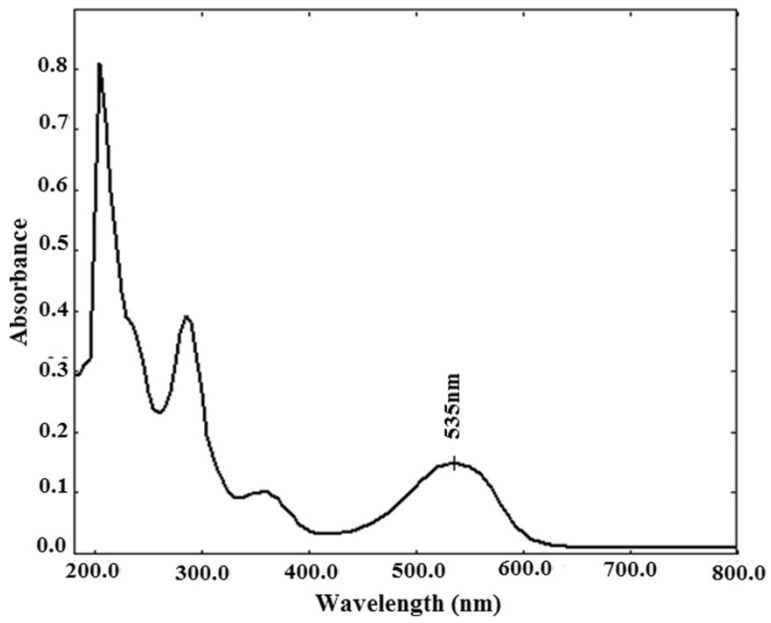

Figure 4 UV visible spectrum of purified pink pigment showing maximum absorbance at 535 nm.

it is seen that the R_f values of the blue component (blue pigment) of crude product matches exactly with that of synthetic indigo which was taken as a standard.

UV-Visible absorption spectrum

Figure 2 shows the UV visible spectra of the blue pigment. The λ_{max} of the blue pigment was found to be 603 nm. The λ_{max} of additional violet and pink pigments were found to be 575 nm and 535 nm respectively as shown in Figure 3 and Figure 4.

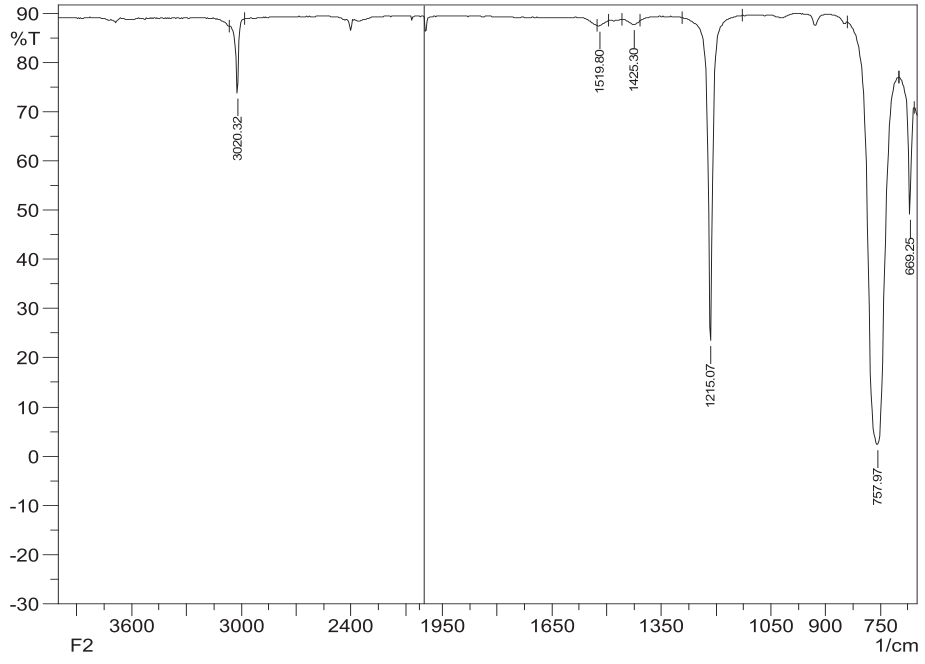

Figure 5 FTIR spectrum of purified blue pigment from *Couroupita guianensis fruit.*

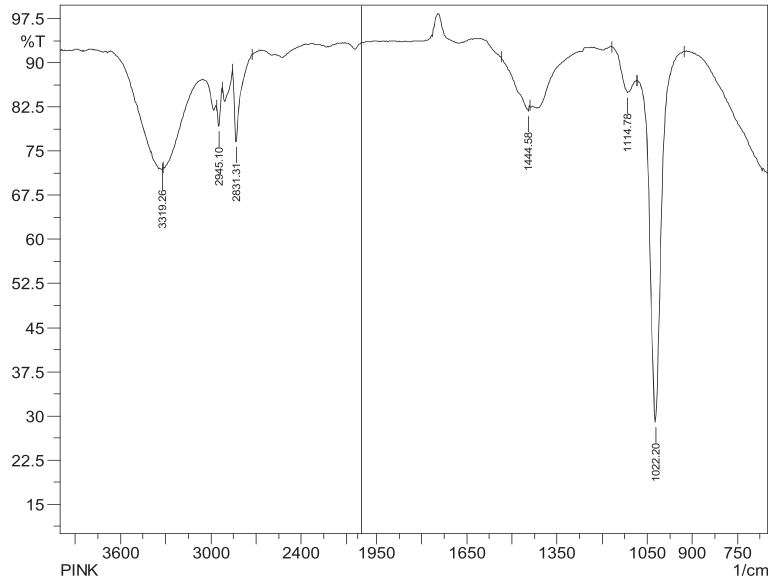

Figure 6 FTIR spectrum of purified pink pigment from *Couroupita guianensis fruit.*

Functional group determination by FTIR

Complete structural analysis of only blue and pink pigment was done as they were of relevance to this study, as commercially available natural indigo shows presence of pink pigment along with blue pigment (Garcia-Macias and John 2004; Chanayath et al. 2002). Also further studies of dye on fibre have shown no presence of pink and violet pigment on the fabric.

Figures 5, 6 & 7 shows the FTIR spectrum of the purified blue and purified pink pigment and standard synthetic indigo whereas Tables 2 & 3 shows their respective functional groups. The functional groups observed in FTIR spectrum of the purified blue pigment, Figure 5, are aromatic C-H stretch (3018.39 cm^{-1}), carboxylic acids O-H

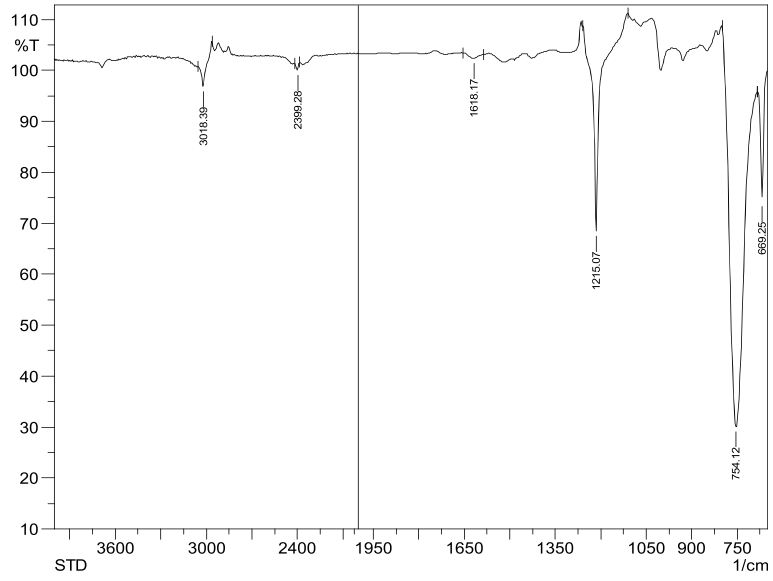

Figure 7 FTIR spectrum of standard synthetic indigo dye.

Table 2 Functional groups of purified blue pigment from FTIR Spectra

Wave number (cm^{-1})	Functional group with mol. motion	Std. synthetic indigo	Purified blue pigment
3020-3000	Aromatics C-H Stretch	3018.39	3020.32
3400-2400	Carboxylic acids O-H Stretch	2399.28	2399.00
1640-1500	Amines N-H bend	1618.17	1519.80
1430-1290	C-H in-plane bend	1425.00	1425.30
1320-1210	Carboxylic acids (C-O stretch)	1215.07	1215.07
860-680 (s)	Aromatic C-H bending	754.12	757.97
730-550	C-Cl stretch, acid chloride	669.25	669.25

stretch (2399 cm^{-1}), N-H bend amines (1519.80 cm^{-1}), C-H in-plane bend (1425.30 cm^{-1}), C-O stretch carboxylic acids (1215.07 cm^{-1}), C-H bending aromatics (757.97 cm^{-1}) and C-Cl stretch acid chloride (669.25 cm^{-1}) corresponding to the peak of chloroform ($CHCl_3$) which was used for elution of purified blue pigment from column chromatography. As can be seen from Table 2 the functional groups of the purified blue pigment matches exactly with that of the functional groups of standard synthetic indigo.

The functional groups of purified pink pigment are shown in Table 3. The functional groups observed in FTIR spectrum of the purified pink pigment, Figure 6, are N-H stretch (1 per N-H bond) amines (3319.26 cm^{-1}), C-H stretch alkanes (2945.10 cm^{-1}, 2831.31 cm^{-1}), O-H bend carboxylic acids (1444.58 cm^{-1}) and C-N stretch (alkyl) amines (1114.78 cm^{-1}, 1022.20 cm^{-1}). Majority of the functional groups observed for pink pigment matches with the reported values in literature of indirubin which is an isomer of indigotin (Chanayath et al. 2002).

Structural determination by ^1H NMR

The purified blue and pink pigments obtained were analyzed for ^1H NMR. The assignment of ^1H NMR spectrum for blue pigment is shown in Figure 8 (a,b) and for pink pigment it is shown in Figure 9.

The ^1H NMR spectrum of the purified blue pigment Figure 8 (a,b) gave peaks at δ 8.486, δ 8.473 ppm, δ 7.485 ppm, δ 7.025 ppm and δ 6.535 ppm corresponding to aromatic ring protons, while at δ 4.054 ppm corresponding to aromatic C-NH. The ^1H NMR spectrum of the purified pink pigment Figure 10 (a,b) gave peaks at δ 8.916, δ 8.903 ppm, δ 7.458 ppm, δ 7.338 ppm, δ 7.145 ppm, δ 7.025 ppm, δ 6.966, ppm and δ 6.884 ppm corresponding to aromatic ring protons, while at δ 4.054 ppm corresponding to aromatic C-NH and δ 8.083 ppm is due to the secondary amide.

Table 3 Functional groups of purified pink pigment from FTIR Spectra

Wave number (cm^{-1})	Functional group with mol. motion	Purified pink pigment
3500-3300	Amines N-H stretch (1 per N-H bond)	3319.26
2950-2800	Alkanes C-H stretch	2945.10
2950-2800	Alkanes C-H stretch	2831.31
1440-1400	Carboxylic acids (O-H bend)	1444.58
1200-1025	C-N stretch (alkyl) Amines	1114.78
1200-1025	C-N stretch (alkyl) Amines	1022.20

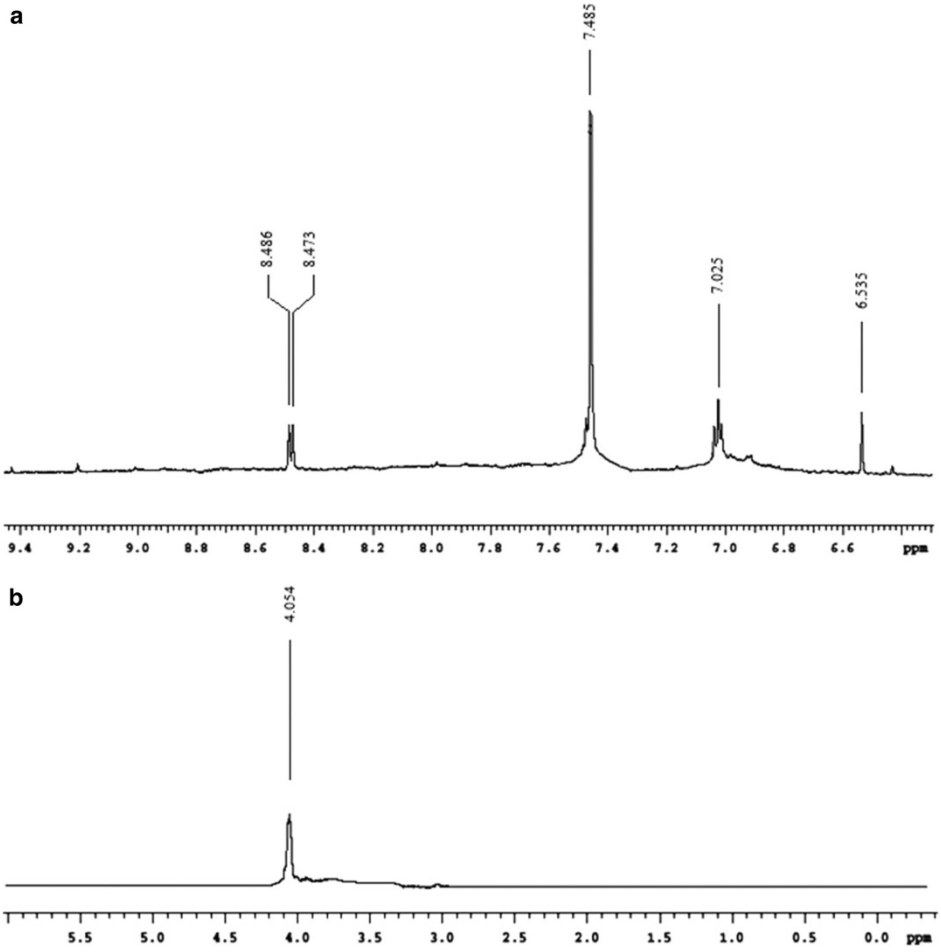

Figure 8 (a,b) ^{1}H NMR spectrum of purified blue pigment extracted from *Couroupita guianensis fruit*.

The chemical shifts for blue and pink pigment are shown in Table 4 and Table 5 respectively. It could be concluded from these results that the blue pigment has chemical formula of $C_{16}H_{10}N_2O_2$ and chemical structure as indigotin as shown in Figure 10 (a,b) whereas the pink pigment has chemical formula of $C_{16}H_{10}N_2O_2$ and chemical structure as indirubin as shown in Figure 11.

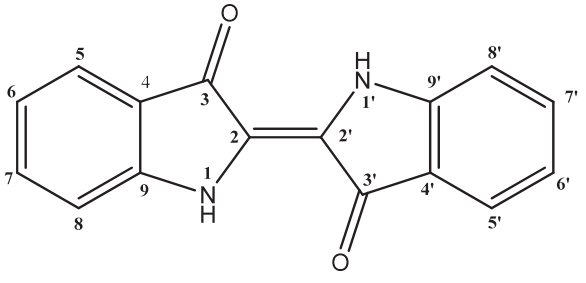

Figure 9 Chemical structure of purified blue pigment extracted from *Couroupita guianensis fruit* as indigotin.

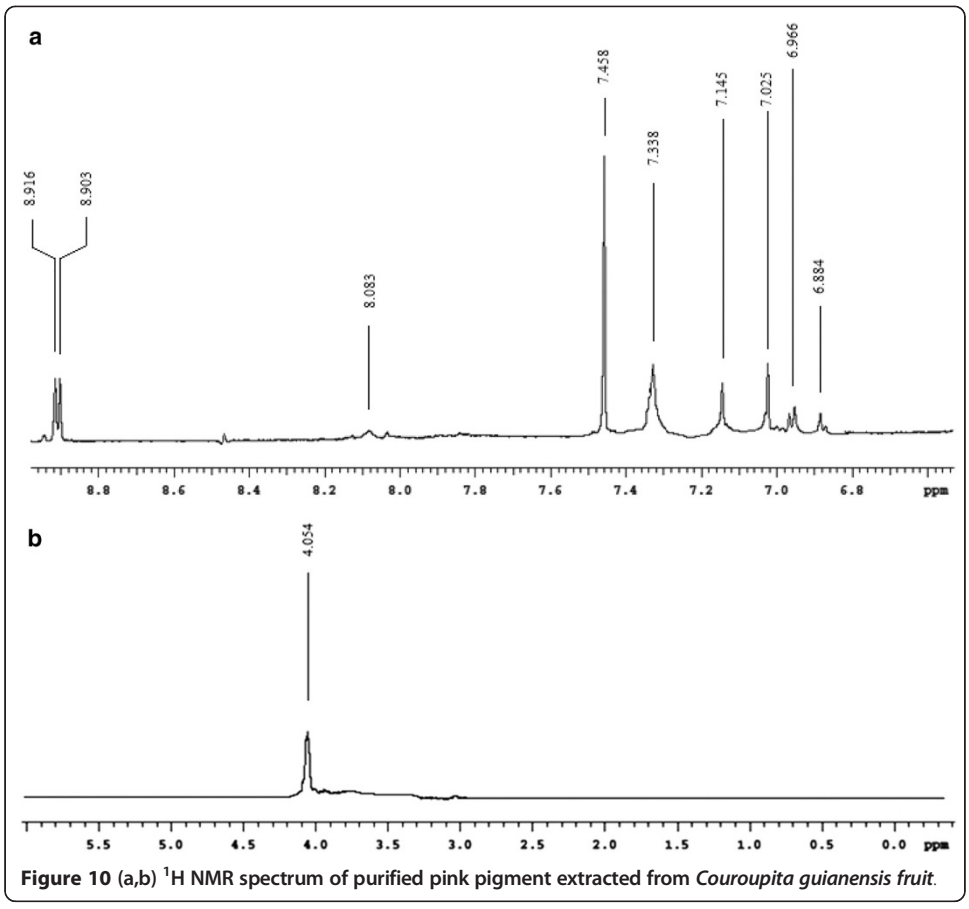

Figure 10 (a,b) ^{1}H NMR spectrum of purified pink pigment extracted from *Couroupita guianensis fruit*.

Percentage purity of indigo dye

The experimentation is shown in Table 6. The % purity of indigo dye was calculated by using the following formula:

$$1 \text{ ml of } 0.02\text{N } KMnO_4 = 0.00147 \text{ gm of Indigo}$$

$$\%\, purity = \frac{Vol.of\ KMnO_4 \times 0.00147 \times 500 \times 100}{Vol.of\ sample \times weight\ of\ the\ dye}$$

Table 4 ^{1}H-NMR chemical shift (ppm) of the purified Blue pigment

Peak no.	Position of ^{1}H	Chemical shift (ppm)	Comment
1	H-1	4.054	Aromatic C-NH
2	H-5	8.486	1-benzene C-H
3	H-6	7.025	1-benzene C-H
4	H-7	7.458	1-benzene C-H
5	H-7′	7.458	1-benzene C-H
6	H-6′	7.025	1-benzene C-H
7	H-5′	8.473	1-benzene C-H
8	H-1′	4.054	Aromatic C-NH

Table 5 ^{1}H-NMR chemical shift (ppm) of the purified pink pigment

Peak no.	Position of ^{1}H	Chemical shift (ppm)	Comment
1	H-1	8.083	Sec. amide
2	H-6	6.966	1- benzene
3	H-7	7.338	1- benzene
4	H-8	7.145	1- benzene
5	H-9	8.916	1- benzene
6	H-5′	8.903	1- benzene
7	H-6′	7.025	1- benzene
8	H-7′	7.458	1- benzene
9	H-8′	6.884	1- benzene
10	H-1′	4.054	Aromatic C-NH

Knowing the volume of $KMnO_4$ (3.6 ml), the volume of sample (20 ml) and the weight of dye (0.5 gm) the purity of indigo dye was found to be 26.46%.

Dyeing mechanism and colour strength value

Dyeing mechanism

The indigo plants contain glucoside indican which is hydrolyzed by enzyme to indoxyl and then oxidized to form indigo blue by air oxidation. The enzyme could come from the indigo plants and maceration of fresh indigo plants would release the glycolytic enzyme from the plant cells to hydrolyze glucoside indicant or glycan which gives indoxyl which on air oxidation gives indigo blue (Chanayath et al. 2002). Indigo blue is water insoluble pigment. In the insoluble form indigo is of little use as a dye, as it cannot be taken up by the fibres, but in the reduced form it is absorbed by the fibres (Gilbert and Cooke 2001). So, during the dyeing process, both an alkaline and reducing medium are required to convert the insoluble indigo to its soluble leuco-indigo form, thus permitting it to enter into the fibre (Poulin 2007). After being taken out and exposed to the air, the indigo white upon and within the fibre rapidly takes up oxygen from the atmosphere and is reversed to insoluble indigo forming a permanent blue colour as shown in

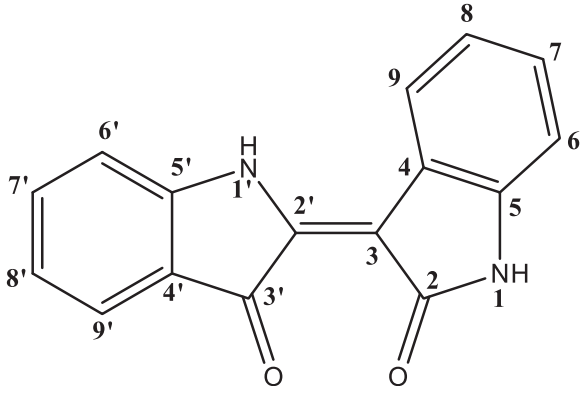

Figure 11 Chemical structure of purified pink pigment extracted from *Couroupita guianensis fruit* as indirubin.

Table 6 Experimentation on % purity of indigo dye

Trials	Vol. of sample solution (ml)	Burette reading (Vol. of $KMnO_4$) ml
1	20	3.5
2	20	3.6
3	20	3.6

Constant burette reading (Vol. of $KMnO_4$) : 3.6 ml.
End point indication: appearance of pale yellow.

Figure 12 (Gilbert and Cooke 2001; Kongkachuichay et al. 2010). On re-oxidation in the air, the insoluble indigo particles are retained in the fibres (Chanayath et al. 2002).

Table 7 shows the colour coordinate values viz., L, a, b, c, H for cotton fabric dyed with crude indigo powder with their varying concentration by using Computer Colour Matching System. The L value indicates perceived lightness or darkness where value of 0 indicates black and 100 indicates white. The values of a and b indicates red (+a) and green (−a) while b value indicates yellow (+b) and blue (−b). The values of C indicates the chroma coordinates i.e. the perpendicular distance from the lightness axis whereas H indicates the hue angle expressed in degrees, with 0° being a location on the + a axis, then continuing to 90° for the + b axis, 180° for −a, 270° for −b and back to 360° = 0° (Tayade and Adivarekar 2013). It is also seen that as the percent shade of dyeing increases the colour strength value also increases which is quite obvious.

The dyeings obtained with natural crude indigo dye by varying their concentration were compared with the dyeings obtained with standard synthetic indigo dye and the shades obtained are shown in Figure 13. The shades obtained were evaluated for their colour coordinate values viz., L, a, b, c, H using Computer Colour Matching System (Spectra Scan 5100+, Data colour International, U.S.A) as shown in Table 8. The table

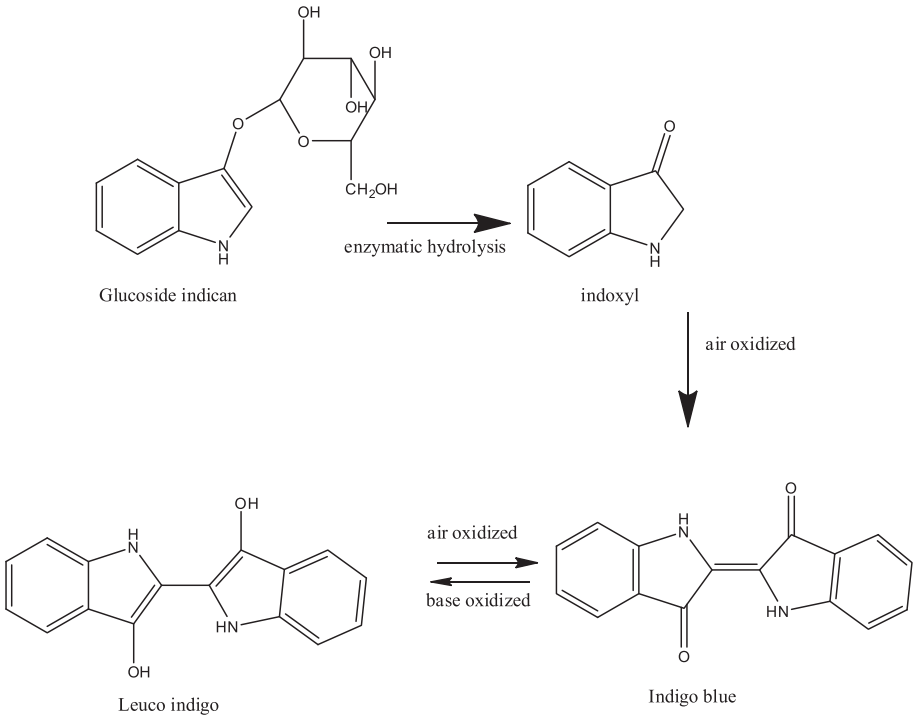

Figure 12 Diagram showing the chemical change of indigo from plant material.

Table 7 Colour strength value of the fabric dyed with crude indigo dye

Samples	L	a	b	C	H	dE	K/S	Colour strength (%)
50%	68.251	−4.089	−11.677	12.372	250.673	———————	0.909	100
100%	59.132	−4.701	−16.404	17.064	253.979	10.290	1.9941	219.382
150%	49.322	−4.762	−18.285	18.895	255.372	20.061	4.0122	441.397

shows the colour coordinate values of the natural indigo dyed samples which are to a greater extent nearer to the synthetic indigo dyed sample.

Negligible deviation among the colour coordinate values indicates excellent shade matching. The highest tolerance limit for colour difference, dE in the industry is generally 1 and hence the samples with the value of $dE \leq 1$ are considered to have PASSED or else it is FAILED (Tayade and Adivarekar 2013). The natural indigo dyed fabric which showed the colour coordinate values much closer to the synthetic indigo dyed fabric was considered to be the best match. It is found that fabric dyed with 150% of crude indigo dye matches with the fabric dyed with 2% shade of synthetic indigo dye which was taken as standard with dE of 0.825. The exceptionally higher % shade was required for matching as commercial synthetic indigo is pure product whit purity over

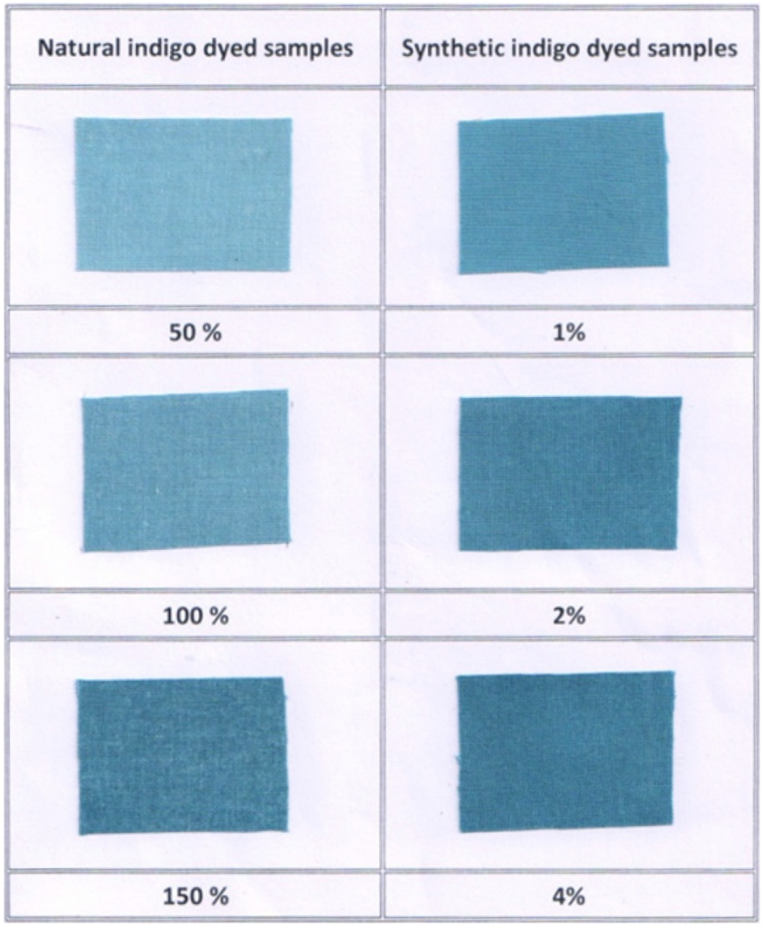

Figure 13 Cotton fabrics dyed with crude indigo dye and synthetic indigo dye.

Table 8 Colour coordinate values of fabrics dyed with synthetic and crude indigo dye

	Standard (4% shade)	Batch (150% shade)
L =	49.124	49.322
a =	−5.205	−4.762
B =	−18.952	−18.285
C =	19.654	18.895
H =	254.613	255.372
dE =	0.825	

Observer: 10 degree.
Colorspace: CIELAB (1976).
Illuminant: D65.
Mode: Reflectance.
Spectro: 5100.
Judgement: PASS.
Standard: Fabric dyed with synthetic indigo dye.
Batch: Fabric dyed with crude indigo dye from *Couroupita guianensis*.

90% (Vuorema 2008) whereas it is not so in case of crude indigo dye which is extracted from natural source.

Fastness properties

The results for fastness properties of fabric dyed with crude dye powder are reported in Table 9. The dyed fabric shows wash fastness and rubbing fastness rating of 3–4 to 4–5 on grey scale implying good to very good fastness property whereas the fabric shows light fastness rating 6–7 to 7–8 on blue wool scale corresponding to good light fastness property. Also it can be seen that the fastness properties obtained for natural indigo dyed fabric are comparable with synthetic indigo dyed samples.

Conclusion

Fermentation of the *Couroupita guianensis* fruit pulp for 5 days gave intense blue pigment and hence was selected as the optimum period for fermentation. The major components from the crude indigo dye extract obtained were blue and pink pigments. The blue pigment was found to be indigotin and the pink pigment was found to be its isomer, indirubin. It was also found from the study that the crude indigo dye extract obtained from *Couroupita guianensis* fruit pulp can be successfully used for dyeing of cotton fabric with good fastness properties. The yield as well as the depth of the dyeing

Table 9 Colour fastness value of the fabric dyed with crude indigo dye and synthetic indigo dye

	Samples	K/S	Rubbing fastness		Washing fastness			Light fastness
					CC[a]	CS[b]		
			Dry	Wet		Cotton	Wool	
Crude indigo dye	50%	0.909	4 – 5	4 – 5	4	4 – 5	4 – 5	6 – 7
	100%	1.9941	4 – 5	4 – 5	4	4 – 5	4 – 5	7/7 – 8
	150%	4.0122	4 – 5	4	3 – 4	4 – 5	4 – 5	6 – 7
Synthetic indigo dye	2%	4.1921	4 – 5/5	4 – 5	4 – 5	4	4 – 5	7/7 – 8
	4%	4.4034	4 – 5/5	4 – 5	3 – 4	4 – 5	4	7 – 8

[a]CC – Colour Change.
[b]CS – Colour Staining.

can be increased with proper experimental procedure and hence can be used as ready to use natural dye for clothing.

Competing interests
The authors declare that they have no competing interests.

Authors' contributions
PBT and RVA collaboratively designed the experiments. PBT performed the experiments, analyzed the data and drafted the manuscript. RVA thoroughly revised the manuscript. Both authors read and approved the final manuscript.

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