Int.J.Curr.Microbiol.App.Sci (2019) *8*(5): 872-882

EXCELLENT PUBLISHERS

International Journal of Current Microbiology and Applied Sciences
ISSN: 2319-7706 **Volume 8 Number 05 (2019)**
Journal homepage: http://www.ijcmas.com

Original Research Article https://doi.org/10.20546/ijcmas.2019.805.102

Eucalyptus Bark as a Source of Natural Dye for Cotton Fabric

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Keywords

Eucalyptus, Bark, Natural dye, Cotton, pre-mordant

Article Info

Accepted:
10 April 2019
Available Online:
10 May 2019

A B S T R A C T

Natural dye extracted from *Eucalyptus* bark was applied to cotton fabrics by conventional method of dyeing. Alum, Vinegar, Myrobolan, Betel leaves, Copper sulphate ($CuSO_4$), Alum + $CuSO_4$, Vinegar + $CuSO_4$, Myrobalan + $CuSO_4$, and Betel leaves + $CuSO_4$ were the treatments used for mordants. The dyeing was carried out with and without mordants by pre-mordanting method. The colour of each dyed material was investigated in terms of CIELAB (L*, a* and b*) and K/S values by perimer color scan. The colour fastness to washing, rubbing (dry and wet), light and perspiration of aqueous dyed fabrics were tested according to ISO standards. Results showed that cotton dyed fabrics without mordant showed a shade of reddish brown, while other mordants exhibited light to dark brown shades. The colour fastness to washing and perspiration were good except unmordanted fabric which showed moderate to good fastness to washing, whereas colour fastness light and dry rubbing were excellent for all fabrics; wet rubbing was good for all nine fabrics except unmordanted fabric which showed moderate to good fastness.

Introduction

Since time immemorial the usage of natural dyes have been an integral part of the human life and society to colour different textiles materials. In the nineteenth century, synthetic dyes in view of their low cost, large variety of shades, superior colour fastness, high dye ability and greater reproducibility over took the usage of natural dyes (Samantha and Agarwal, 2009; Yususf *et al.*, 2013; Mohd shabbir *et al.,* 2016). Nevertheless, some of these dyes have the potential to generate toxic effluents that adversely affect the aquatic ecosystem and may have mutagenic, carcinogenic, toxicological properties and despite the low incidence, some are associated with contact skin problems or diseases (Bulut and Akar, 2012; Rossi *et al.*, 2017).

Textile industry in particular textile dyeing industry is known to be not a sustainable activity due to high demand of resources such as water, energy and excessive consumption of chemicals, contributing to the increasing environmental degradation. And hence textile industry in searching of new cleaner methodologies in order to minimize the use of natural resources, as well as by continually improving the availability techniques towards a sustainable activity of zero emissions (Teresa Linhares and Maria Teresa, 2017).

As an alternative to new dye design and synthesis, to replace the synthetic dyes either derived from toxic precursors or prone to forming toxic metabolites, the return of natural dyes has increasingly been contemplated because of their biodegradability, cheap availability of raw material, low incidence of allergic reactions and low toxicity (Shahid *et al.,* 2013; Rossi *et al.,* 2017). This approach is aligned with the growing movement in our society towards sustainability, green and environmentally friendly products “green chemistry” as an alternative or co-partner to synthetic dyes. Naturally the plants and animals are having full of exquisite colours, fascinating and attracting human being towards a vast portfolio of possibilities and have been identified for extraction of colour and their diversified use in textile dyeing and other disciplines *viz.,* medicinal textiles, green fashion textiles and Ayurvastra.

Eucalyptus grows on wide variety of soils and distributed throughout the India. This species is exotic and mostly colonized in India because of the presence of wide variety of climatic regions. Eucalyptus is fast growing and can be harvested in 3-4 years after planting; it is one of the most important sources of natural dye, yielding several yellowish-brown colourant. Currently Eucalyptus is one of the important tree which is grown outside the forests contributing nearly >8X10^{6} ha of plantation with the productive potential of five tonnes of biomass ha^{-1}yr^{-1} and an average production is 2.5 tonnes/ha/yr. The bark content (per cent of stem volume) is about 17.4 % and yields 55.7 m^{3} ha^{-1} and weighs of 25 tonnes ha^{-1} (Padam *et al.,* 2014). Eucalyptus is a prime raw material for the paper and pulp industries across the world particularly in southern India. The huge quantity of bark is being generated from Eucalyptus utilization sectors or industries mainly pulp and paper industries, where bark contains ample natural tannins and polyphenols ranging from 10 per cent to 12 per cent (Ali *et al.,* 2007), mainly disposed as waste or used as fuel. Hence this work is concerned with the extraction of dye from Eucalyptus bark and its application on cotton fabric in an endeavour to investigate application conditions to attain the desirable fastness properties.

Materials and Methods

Plant material

The bark was collected from trees grown at Forest College and Research Institute, Mettupalayam; from each tree 0.5 kilograms of bark was collected by making rectangular shape at breast height (1.37m) and below by using the sharp knives and hammer. After the collection of bark was washed thoroughly with water to remove the impurities present on surface of the bark and soaked in 1% ascorbic acid. Then bark was made into small pieces of 25mm to 50mm size and shade dried for 14 days until the moisture content reduced to less than 10 to 11 per cent. Later the bark was pulverized by using the mixer grinder and then sieved by using 0.6 mm sieve and stored in air tight glass containers. The cotton fabric (124 GSM), plain weaved and 100% ready for dyeing type was purchased from Premier Mills, Mumbai, India. Copper sulphate of analytical grade was purchased from S.D. fine chemicals, Mumbai. From Central Drug House (P) Ltd. Alum analytical grade was purchased. Myrobalan powder, Vinegar, and Betel leaves were purchased from local market. Non-ionic surfactant was purchased from Kusmo chemicals, Thane, Maharastra.

Aqueous dye extraction

The extraction of natural dye from *Eucalyptus* bark was done with normal RO purified water. One litre of water was taken and heated up to 70ºC and then 50g of powdered bark was added to the heated water and stirred well and left boiling for 60 minutes till the solution become approximately 500ml. Then the solution was cooled and filtered by using muslin cloth and used for dyeing.

Dyeing of cotton fabric

Scouring

The 100% cotton fabric was cut into 30.0 cm X 30.0 cm and then treated with non-ionic surfactant solution containing 2g/L each of soap and soda ash at 60^{o}C for 30 minutes to remove starch and other stiffening agents.

After scouring the fabric was pre-mordanted with the mordants and their combinations *viz.*, Alum (T_1), Vinegar (T_2), Myrobalan (T_3), Betel leaves (T_4), Copper sulphate ($CuSO_4$) (T_5), Alum + $CuSO_4$ (T_6), Vinegar + $CuSO_4$ (T_7), Myrobalan + $CuSO_4$ (T_8), and Betel leaves + $CuSO_4$ (T_9). With one per cent concentration of each mordant on weight of fabric (o.w.f) with 20 percent shade of dye bath and compared with control (T_{10} no mordant). The dyeing was carried out at 80^{o}C in a dye bath with the Material to Liquor ratio (M:L) of 1:40 for 1 hour. After 1 hour, the dyed samples were subsequently washed and dried at room temperature.

Colour fastness evaluation

Colour fastness to wash: ISO 105 c03

The dyed fabric of size 40 mm x 100 mm was attached to a piece of the multi-fibre adjacent fabric containing wool, acrylic, polyester, nylon, cotton and acetate, also measuring 40 mm x 100 mm, by sewing along one of the shorter sides with the multifibre fabric next to the face of the dyed fabric. The sewed specimen was placed in the container and the necessary amount of soap solution (5 g of soap and 2 g of anhydrous sodium carbonate per litre) was added and heated upto 60^{o}C±2^{o}C in a liquor ratio of 50:1 for 30 minutes. After that stipulated time, the sewed specimen was removed, rinsed twice in cold water and running tap water for 10 minutes, squeezed and dried. The change in colour of the specimen and the staining of the adjacent fabrics were assessed with grey scales (ISO, 1989).

Colour fastness to rubbing: ISO 105 x12

Dry rubbing

The natural dyed specimen was mounted to the holding clamp on the baseboard of the crockmeter. A dry rubbing cloth was mounted flat over the end of the peg on the crockmeter and fixed by means of the spring clip provided. The specimen was rubbed back and forth over a straight track 100 mm ± 8 mm long for 10 complete cycles (10 times back and forth) @ one cycle per second. Staining of the rubbing cloth was assessed with grey scale (ISO, 2001).

Wet rubbing

A rubbing cloth was wetted with distilled water and squeezed between blotting papers and the tests were carried out as the procedure for dry rubbing. The tested rubbing cloth was allowed to dry at room temperature. Staining of the rubbing cloth was assessed with grey scale (ISO, 2001).

Colour fastness to light: ISO 105 b02

The colour fastness to light was assessed with an artificial light source, namely xenon arc lamp (as it is resembles the natural daylight). Here the paper cards were cut to the size of 4.5cm x 13.5cm and fit to the specimen holder. Each fabric specimen was measured with the size of 4.5cm × 1.0cm and attached to the paper card prepared. The specimens were exposed until a contrast (change in colour) corresponding to grey scale grade 4 and later to a grey scale 3 (ISO, 2013).

Colour fastness to perspiration: ISO 105 e04

The specimens of the natural dyed cotton fabric in contact with the standard multifibre fabric (for colour transfer) was immersed in simulated alkaline and acid solution, drained and placed between two plates under a specific pressure, temperature and time in a testing device (perspirometer). Any change in colour of the specimens and staining of the multifibre was then assessed with the corresponding grey scale rating (ISO, 2014).

Assessment of the results

Grey scale rating

The grey scale was used for assessing the degree of change in shade caused to a dyed cotton fabric material and the degree of staining on the adjacent fabric caused by a dyed fabric material in the colour fastness tests. The scale consists of nine pairs of standard grey colour chips each representing a visual difference and contrast. It has 9 possible values, i.e. 5, 4-5, 4, 3-4, 3, 2-3, 2, 1-2, 1.

Evaluation of colour strength

Dyed cotton fabric was evaluated in terms of CIELAB colour space (L*, a* and b*) values using the Perimer Colorscan. L* corresponding to the brightness (100= white, 0 = black), a* to the red–green coordinate (+ve= red, -ve= green) and b* to the yellow–blue coordinate (+ve = yellow, -ve =blue). The L*, a* and b* values helped to understand the tonal variations between the samples.

Results and Discussion

Colour fastness to washing

The fastness ratings of dyed cotton fabric with and without mordants are presented in Table 1. Results showed that all pre-modranted fabrics exhibited colour change of in 4 grey scale rating except non mordanted fabric moderate to good (3-4), and staining on multifibre fabric resulted in 4-5 rating for wool, acrylic, polyester and acetate. Whereas, for cotton and nylon it is 4.

The results are similar to the literature of Anjali and Ela, (2016) in *Acacia arabica* (Excellent); Pisitsak *et al.*, (2016) in *Xylocarpus granatum* bark (very good to excellent); Lodrick *et al.*, (2015) *Mangifera indica* bark (excellent); Gulzar *et al.,* (2014) in *Acacia nilotica* bark (good to excellent); Anshu sharma and Ekta grover, (2011) in *Juglas regia* Linn. (4-5 i.e. very good to excellent). A probable explanation for good fastness property is that *Eucalyptus* bark is rich with tannins and flavonoids and can form metal chilates with mordants (metallic and non-metallic) i.e. formation of covalent bond between fabric and dye. Hence after mordanting the tannins and flavonoids are insoluble in water, which improves the washing fastness.

Colour fastness to rubbing

The colour fastness to rubbing was carried out in dry and wet conditions. Dry rubbing of cotton fabric on adjacent cotton exhibited a grey scale rating of 4-5 irrespective of mordanted and non mordanted fabrics.

Whereas, under wet condition, staining on adjacent cotton was found to have grey scale rating of 4 for mordanted fabrics except unmordanted fabric (3-4 grey scale rating) as presented in Table 2.

Results are in line with Kuchekar *et al.,* (2018) in *Thespesia populnea* (good fastness for both dry and wet rubbing); Gulzar *et al.,* (2015) in *Acacia nilotica* (4-5 grey scale rating under dry and wet rubbing. A probable explanation for low fastness under wet condition, was due to poor complexes formed between dye bath and fabric leads to staining on periphery of the fabric (Burkinshaw and Kumar *et al.,* 2008), which can be improved by pre or post or simultaneous addition of metallic mordnats to fabric (Tera *et al.,* 2012).

Colour fastness to light

With respect to fastness to light, mordanted and un-mordanted fabrics exhibited the same grey scale rating of 5 (excellent) for all the treatments (Table 3), which can be attributed to high photostability of oxidative polyphenolic compounds in dye bath (Rattanphol *et al.,* 2013.), structure and concentration of dye (Gokhan *et al.,* 2014). The results are similar to the literatures such as Gulzar *et al.,* (2014) in *Acacia nilotica* (3-unmordanted fabric, 4 - premordanted fabric with 7% tannic acid and 5% $CuSO_4$); Anjali and Ela, (2016) in *Acacia arabica* (fair - good fastness); in *Xylocarpus granatum* Pisitsak *et al.,* (2016) good (4) fastness; Anshu and Ekta, (2011) in walnut (*Juglas regia* Linn.) excellent fastness; in *Mangifera indica* as reported by Lodrick *et al.,* (2015); dyed cotton fabric found to have good to excellent (4-5) light fastness.

Colour fastness to perspiration

Fastness to perspiration in acidic and alkaline conditions dyed fabric exhibited a good colour fastness property with a grey scale rating of 4. Staining on multifibre fabric exhibited 4-5 grey scale rating for wool, acrylic, polyester and acetate. Whereas, staining on nylon and cotton fabric is 4 grey scale rating in both acid and alkaline conditions given in Table 4. Overall the fastness to perspiration of dyed fabric mainly depend on structure of dye and mordant, concentration.

Similar findings were reported by Wan *et al.,* (2011) in *Gluta aptera* (wood extract dyed to cotton fabric shown good fastness to perspiration), Anshu and Ekta (2011) in walnut (bark dye 3-4 under alkaline and acidic conditions), Padma *et al.,* (2011) in *Artocarpus heterophyllus* (3 for unmoranted fabrics under alkaline and acidic conditions, 4 - pre-moranted fabrics under alkaline and acidic conditions), and Kumaresan (2013) in *Achras sapota* (stems - excellent) and *Cordia sebestena* (flowers) very good to excellent fastness to perspiration under acidic and alkaline conditions.

Colour strength and colour coordinates of dyed samples

The colour strength (k/s) values and colour co-ordinates are presented in Table 5. Results showed that ten different brown shades of mordanted fabrics and un mordanted fabric (control) varied with different K/S values at 400 nm. Highest K/S value was observed in T_7 ($CuSO_4$+vinegar) followed by T_3 (Myrobalan), T_6 ($CuSO_4$+Alum) T_8 ($CuSO_4$+Myrobalan), T_9 ($CuSO_4$+Betel leaves), T_5 ($CuSO_4$), T_4 (Betel leaves), T_1 (Alum), T_2 (Vinegar) and T_{10} (Control) with the values of 44.03, 40.28, 40.24, 38.39, 37.61, 37.51, 36.61, 35.85, 34.85, and 34.51 respectively (Table 5). This confirms that addition of mordant will help in higher binding and colour strength to the fabric as represented in Figure 1.

Table.1 Response of Eucalyptus bark dyed cotton fabrics to colour fastness to washing

Treatment Mordants	Change in colour	Staining on adjacent cotton					
		Wool	Acrylic	Polyester	Nylon	Cotton	Acetate
T_1 – Alum	4	4-5	4-5	4-5	4	4	4-5
T_2 - Vinegar	4	4-5	4-5	4-5	4	4	4-5
T_3 – Myrobalan	4	4-5	4-5	4-5	4	4	4-5
T_4 - Betel leaves	4	4-5	4-5	4-5	4	4	4-5
T_5 - $CuSO_4$	4	4-5	4-5	4-5	4	4	4-5
T_6 - $CuSO_4$ + Alum	4	4-5	4-5	4-5	4	4	4-5
T_7 - $CuSO_4$ + Vinegar	4	4-5	4-5	4-5	4	4	4-5
T_8 - $CuSO_4$ + Myrobalan	4	4-5	4-5	4-5	4	4	4-5
T_9 - $CuSO_4$ + Betel leaves	4	4-5	4-5	4-5	4	4	4-5
T_{10} - Control	3-4	4	4-5	4-5	4	4	4-5

Table.2 Response of Eucalyptus bark dyed cotton fabrics to colour fastness to rubbing

Treatment Mordants	Staining on adjacent cotton	
	Dry	Wet
T_1 - Alum	4-5	4
T_2 - Vinegar	4-5	4
T_3 - Myrobalan	4-5	4
T_4 - Betel leaves	4-5	4
T_5 - $CuSO_4$	4-5	4
T_6 - $CuSO_4$ + Alum	4-5	4
T_7 - $CuSO_4$ + Vinegar	4-5	4
T_8 - $CuSO_4$ + Myrobalan	4-5	4
T_9 - $CuSO_4$ + Betel leaves	4-5	4
T_{10} - Control	4-5	3-4

Table.3 Response of Eucalyptus dyed cotton fabrics to colour fastness to light

Treatment Mordants	Staining on adjacent cotton
T_1 - Alum	5
T_2 - Vinegar	5
T_3 - Myrobalan	5
T_4 - Betel leaves	5
T_5 - $CuSO_4$	5
T_6 - $CuSO_4$ + Alum	5
T_7 - $CuSO_4$ + Vinegar	5
T - $CuSO_4$ + Myrobalan	5
T_9 - $CuSO_4$ + Betel leaves	5
T_{10} - Control	5

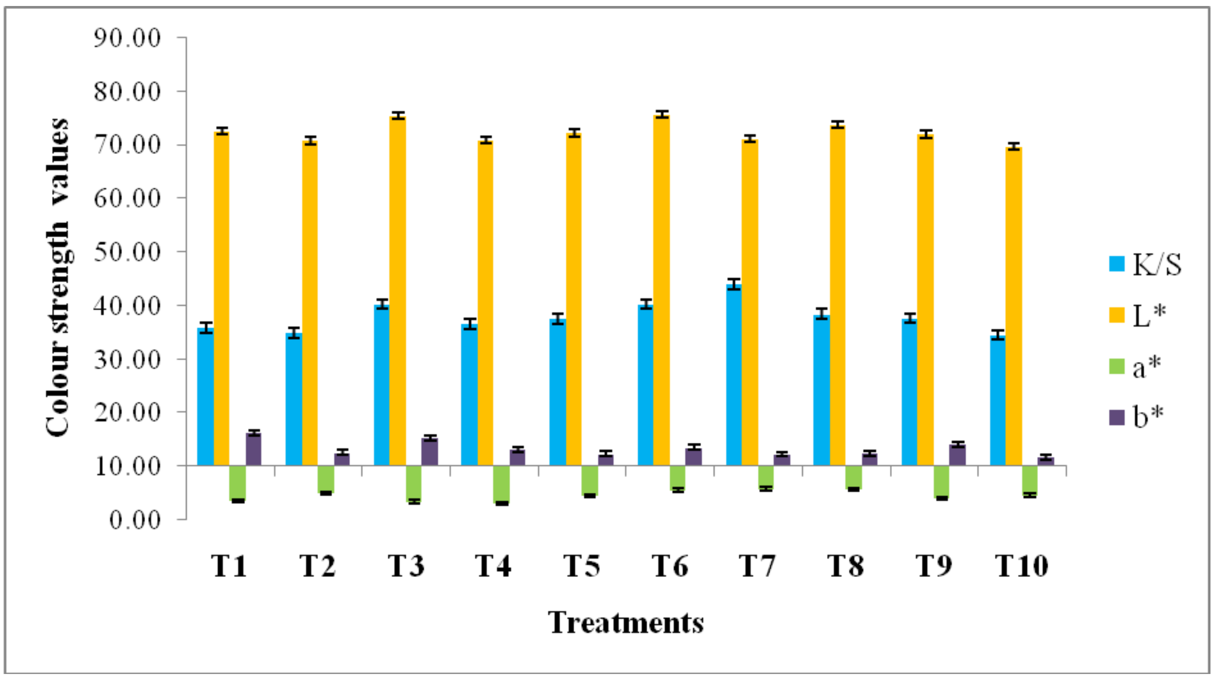
Table.4 Response of Eucalyptus dyed cotton fabrics to colour fastness to perspiration

Treatment Mordants		Change in colour	Staining on adjacent cotton					
			Wool	Acrylic	Polyester	Nylon	Cotton	Acetate
T_1 - Alum	Acidic	4	4-5	4-5	4-5	4	4	4-5
	Alkaline	4	4-5	4-5	4-5	4	4	4-5
T_2 - Vinegar	Acidic	4	4-5	4-5	4-5	4	4	4-5
	Alkaline	4	4-5	4-5	4-5	4	4	4-5
T_3 - Myrobalan	Acidic	4	4-5	4-5	4-5	4	4	4-5
	Alkaline	4	4-5	4-5	4-5	4	4	4-5
T_4 - Betel leaves	Acidic	4	4-5	4-5	4-5	4	4	4-5
	Alkaline	4	4-5	4-5	4-5	4	4	4-5
T_5 - $CuSO_4$	Acidic	4	4-5	4-5	4-5	4-5	4	4-5
	Alkaline	4	4-5	4-5	4-5	4-5	4	4-5
T_6 - $CuSO_4$ + Alum	Acidic	4	4-5	4-5	4-5	4-5	4	4-5
	Alkaline	4	4-5	4-5	4-5	4	4	4-5
T_7 - $CuSO_4$ + Vinegar	Acidic	4	4-5	4-5	4-5	4-5	4	4-5
	Alkaline	4	4-5	4-5	4-5	4	4	4-5
T_8 - $CuSO_4$ + Myrobalan	Acidic	4	4-5	4-5	4-5	4-5	4	4-5
	Alkaline	4	4-5	4-5	4-5	4	4	4-5
T_9 - $CuSO_4$ + Betel leaves	Acidic	4	4-5	4-5	4-5	4-5	4	4-5
	Alkaline	4	4-5	4-5	4-5	4	4	4-5
T_{10} - Control	Acidic	4	4-5	4-5	4-5	4-5	4	4-5
	Alkaline	4	4-5	4-5	4-5	4	4	4-5

Table.5 Colour strength and colour coordinates of dyed samples

Treatment Mordants	K/S at 400 (nm)	L*	a*	b*
T_1 - Alum	35.85	72.58	3.55	16.22
T_2 - Vinegar	34.85	70.78	4.95	12.56
T_3 – Myrobalan	40.28	75.39	3.46	15.31
T_4 - Betel leaves	36.61	70.93	3.03	13.07
T_5 - $CuSO_4$	37.51	72.20	4.43	12.28
T_6 - $CuSO_4$ + Alum	40.24	75.58	5.53	13.46
T_7 - $CuSO_4$ + Vinegar	44.03	71.04	5.82	12.24
T_8 - $CuSO_4$ + Myrobalan	38.39	73.72	5.63	12.37
T_9 - $CuSO_4$ + Betel leaves	37.61	71.93	4.03	14.07
T_{10} - Control	34.51	69.68	4.59	11.63

Fig.1 Colour strength and colour coordinates of dyed samples



The results are confirmed by literatures *viz.*, Keka Sinha *et al.*, (2016) found that *Terminalia arjuna* bark dyed cotton fabric showed better CIE L* a* and b* values in improvement of colour quality of natural polymers treated by tannic acid+alum mordant (L* 43.73, a*19.05, and b* 32.06); Ozan avinc *et al.*, 2013 found that dyed cotton fabric from *Pinus brutia* bark with alum (L* 52.9, a*10.3, b* 17.0) oak ash (L*66.9, a 7.1*, and b* 8.4) mordants. The enhancement in the colour depth was due to the presence of copper sulphate a metal mordant which forms insoluble metal tannates with eucalyptus bark tannin, polyphenolic hydroxyl group and further metal tannates present on pre-mordanted fabric forms an insoluble lake with reactive group dye molecules and lead to higher fixation of natural colourant on fabric (Prabhu and Teli, 2014). The colour coordinates L* a* and b* values results obtained for cotton fabric pre-mordanted with alum, vinegar, myrobolan, betel leaves, copper sulphate, $CuSO_4$+alum, $CuSO_4$+vinegar, $CuSO_4$+myrobolan, $CuSO_4$+betel leaves and bark dye (without mordant) are given Table 5. In all pre-mordanted and naturally dyed fabrics, the brightness or L* values decreased highly resulting in deepening of shades as compared to only naturally dyed fabrics. From a* and b* values, the incorporation of bark dye (without mordant) and in combination with mordants produced good improvement in shades and their values were positive and this showed shits in their tones resulting in beautiful colours compared with only dyed cotton fabrics and colours obtained are presented in Table 5. Results are in line with reports such as Pisitsak *et al.,* (2016) in *Xylocarpus granatum* without mordant (L* 64.66, a* 11.52, and b* 15.13) and with mordant (L*43.33, a* 16.32, and b* 4.45); Geom and Kyung (2011) in Pinux^{TM} for without mordant (L* 56.02, a* 8.19, b*16.45) and with (L* 52.58, a* 12.17 and b* 22.47).

In conclusion, it has been found that waste i.e. bark from pulp and paper processing industries and, harvesting sites of eucalyptus wood is a potential source of natural dye for dyeing cotton and other fabrics (silk, wool, and nylon). In this regard, results from colouristic assessments indicate that light to dark shades having good (washing, perspiration, wet rubbing) and excellent (light and dry rubbing) fastness properties can be readily obtained. This characteristic *Eucalyptus* dyed cotton fabric can be used in manufacturing of inner and kids wears, curing textiles, dye fabric shows consistent with shades such as yellowish-brown, brown and beige are frequently used in all colour charts of all collections of apparel brands, because it is the basic colour of that segment. Utilization of bark from *Eucalyptus* processing industries will lead to transforming a residue into a useful colouring matter for textile industry. Also, the natural dye from *Eucalyptus* bark apart from meets cleaner and eco-friendly textiles manufacturing in cheaper and reliable manner, it also becomes an additional source of income for the farmers who cultivate *Eucalyptus* spp.

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How to cite this article:

Gajendra, C.V. and Kumaran, K. 2019. *Eucalyptus* Bark as a Source of Natural Dye for Cotton Fabric. *Int.J.Curr.Microbiol.App.Sci.* 8(05): 872-882.
doi: https://doi.org/10.20546/ijcmas.2019.805.102