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Eco-printing of cotton fabric with arecanut slurry using *Asteracantha longifolia* (*Kokilaksha*) seed gum

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Abstract
The present study was undertaken to investigate the possibility to using natural gum extracted from *kokilaksha* seed gum for screen printing on cotton fabric. The Printing of fabric requires a thickening agent, which is used to make the varying thickness of the printing material. The print quality was assessed by visual analysis and by colourfastness test. Eco-printed cotton samples without mordant and with $FeSO_4$ mordant using 0.5 per cent *kokilaksha* gum concentration exhibited higher depth of colour, evenness of print, sharpness of print and overall appearance compared to other concentrations (0.1 – 0.4%). With $FeSO_4$ mordant printed sample exhibited good to very good (3/4) colour fastness properties compared to without mordant printed samples. The experimental outcome exhibited that 0.5 per cent *kokilaksha* seed gum can be successfully used for textile printing on cotton fabrics as natural thickener/gum.

Keywords: colourfastness, cotton, *kokilaksha*, mordant, thickener

Introduction
Surface ornamentation of textile has brought variety, liveliness and interest in clothing. Without designing clothing would be monotonous and boring. The art of dyeing and printing has played a significant role in adding attractiveness to the earth as well as makes an important contribution to fabric beautification. Textile printing is a term which is used to indicate the patterning of cloth by means of printing, dyeing or painting. Printing of fabric requires a thickening agent, which is used to make the varying thickness of the printing material. The viscosity of the printing paste can be increased or decreased by varying the amount of thickener. Printing also requires highly specialized equipments, materials, knowledge and a lot of aesthetic ability in order to achieve the desired effect. A printed fabric can be produced by a wide array of methods like stencil printing, block printing, screen printing, roller printing, resist printing, *etc*.

Recently there has been a revival of interest in the use of natural dyes in textile colouration. This is a result of the stringent environmental standards imposed by many countries in response to the toxic and allergic reactions associated with the use of synthetic dyes. It is well known that problems in dyeing with natural dyes are the low exhaustion of natural colourants and the poor fastness of dyed fabrics. Attempts to overcome these problems have mainly focused on the use of metallic salts as mordants, which are traditionally used to improve fastness properties/exhaustion and to develop different shades with the same dye.

Arecanut (*Areca catechu* L.) belongs to the family *Arecaceae* commonly called as Betel nut, grown in much of the Tropical Pacific, Asia and parts of East Asia. In India arecanut occupies a prominent place among the cultivated crops in Kerala, Karnataka, Assam, Meghalaya, Tamilnadu and West Bengal. Popularly known as arecanut, beetal nut or supari, is extensively used in India by all classes of society as a masticatory and is an essential requisite for several religious and social ceremonies. *Kokilaksha* is an annual herb that grows up to the height of 60 cms. The plant stem is square, hairy and thickened at the nodes. The bark is dark brown in colour while the leaves are elliptic-lanceolate and hispid. The flowers of this herb are violet and somewhat purple-blue. The fruit is oblong, linear, glabrous and about 1cm long containing seeds which are spherical hairy and brown in colour. *Hygrophila auriculata* (L.) belonging to family *Acanthaceae*; is also known as *Asteracantha longifolia* and commonly known as *Neermulli*, *Talmakhana*, *Kokilaksha* and *Iksura* is a common plant growing in marshy and water logged areas. The plant is an important medicinal herb, widely distributed in India and is used for different medicinal purposes. With water the seeds develop a large amount of tenacious mucilage (Thankamma, 1999; Nigam *et al*., 2015) [6].

There is no evidence of the use of these seeds in printing. Therefore an effort was made to standardize the printing recipe using arecanut slurry and *Kokilaksha* gum assessed the colourfastness properties of printed samples.

Material and Methods

Desizing of cotton: Plain cotton fabric was purchased from local market. Desizing was done in order to remove the impurities from the fabric. The fabric was treated in 5 gpl detergent solution with MLR ratio of 1:40 for 45 min. Fabric was squeezed as well as rinsed in running water and dried.

Pre-treatment agent: In the present study dye powder and myrobolan powder were combined in 50:50 and 75:25 proportions.

Printing recipe

Arecanut: myrobolan ratio - 100:0, 50:50, 75:25
Gum - *Kokilaksha* seed gum
Gum concentration - 0.1%, 0.2%, 0.3%, 0.4% and 0.5%
Mordant - Ferrous sulphate mordant

According to 100:0, 50:50 and 75:25 ratios, arecanut dye powder and myrobolan soaked for 8 hours in 100 ml water. The solutions were boiled for 30 mins for extraction of dye. The extracted dye solution was made into concentrated form by boiling it until the volume of the dye liquor reduced to 10 ml.

Preparation of mucilage: For the present experiment, 0.1-0.5 per cent *kokilaksha* seed gum was directly added to the condensed dye solution.

Mordanting: For printing experiments 0.4 per cent of ferrous sulphate (1%) was mixed in 5 ml of water and then added to the printing paste (10ml).

Printing procedure: Once the printing paste was ready, printing process was carried out on cotton fabrics using screen printing. After printing the fabrics were shade dried for 24 hrs.

Steaming and curing: Steaming is very important process for dye fixation. Steaming of the printed samples were done by laboratory steamer at 125 ^{o}C for 30 min.

Visual evaluation of printed samples: Printed samples were assessed through visual analysis by thirty respondents using questionnaire with five point rating scale for depth of colour, evenness of print, sharpness of print and overall appearance of the print by weighted mean score. Based on the outcome of the survey, 0.5 percent gum concentration was selected for colourfastness test.

Washing: After steaming the printed samples were treated with 5 per cent common salt for 30 minutes at room temperature. The fabrics were rinsed thoroughly in running water to remove all the vegetative matter deposited on the print area, rinsed squeezed and dried under shade.

Assessment of colourfastness properties of printed samples

Colour fastness of the printed fabrics to light, wash, perspiration (acidic & alkaline) and rubbing were assessed by standard test procedures *viz*., IS: 2454 – 1985, IS: 971 – 1983 and IS: 687 – 1979, respectively. Light fastness ISO 120-40 (1-8) *viz*., 1-poor, 2-low, 3-average, 4-rather good, 5-good, 6-very good, 7-extreamly good and 8-excellent ratings were given as per the blue wool standards. Samples for washing, rubbing and perspiration fastness were assigned ratings for colour change and colour staining on standard fabric with help of grey scales *viz.*, 1-5, where 1 - poor, 2 - fair, 3 - good, 4 - very good and 5 - excellent.

Results and Discussion

Records presented in Table 1 that visual evaluation of cotton samples screen printed with different concentrations of arecanut: myrobolan (without mordant) for optimization of the *kokilaksha* seed gum.

Table 1: Visual evaluation (WMS) of cotton samples screen printed with different concentrations of arecanut: myrobolan pigment (with mordant) for optimization of the *kokilaksha* gum concentration

Gum Conc. (%)	100% Arecanut					Arecanut: Myrobolan (50:50)					Arecanut: Myrobolan (75:25)				
	Weighted mean score (WMS)														
	DC	EP	SP	OA	Average	DC	EP	SP	OA	Average	DC	EP	SP	OA	Average
0.1	3.00	2.50	2.53	2.56	2.64	3.93	2.13	2.40	2.60	2.76	2.80	2.93	3.30	3.20	3.05
0.2	3.46	2.50	2.53	2.60	2.77	3.76	2.66	2.83	2.60	2.96	2.93	3.33	3.60	3.40	3.31
0.3	3.46	2.56	3.00	3.46	3.12	3.86	2.76	3.40	3.00	3.25	3.00	3.73	3.73	3.46	3.48
0.4	3.80	2.86	3.40	3.53	3.39	4.00	3.60	3.80	3.16	3.64	3.00	3.93	3.93	4.06	3.73
0.5	3.86	3.30	3.80	4.26	3.80	4.60	3.86	3.93	3.93	4.08	3.46	4.13	4.20	4.06	3.96

Paired t-test

Arecanut: myrobolan ratio	Mean ± SD	Mean ± SD	t-value
100:0 and 50:50	3.16 ± 0.29	3.33 ± 0.41	0.84^{NS}
100:0 and 75:25	3.16 ± 0.29	3.52 ± 0.51	0.20 ^{NS}
50:50 and 75:25	3.33 ± 0.41	3.52 ± 0.31	0.31 ^{NS}

DC-Depth of colour, EP-Evenness of print, SP-Sharpness of print, OA- Overall appearance
WMS – weighted mean score
Significant at 0.05 level of significance
NS- Non-significant
N - Significant

Depth of colour: Cotton samples screen printed with 100 per cent arecanut dye using 0.5 per cent *kokilaksha* gum concentration imparted greater weighted mean score (3.86) followed by 0.4 per cent gum concentration sample (3.80). Whereas screen printed cotton sample using 50:50 arecanut: myrobolan dye proportion with 0.5 per cent *kokilaksha* gum

concentration exhibited highest weighted mean score (4.60) followed by 0.4 per cent gum concentration sample (4.00) towards depth of colour. Similarly 0.5 per cent *kokilaksha* gum concentration with 50:50 arecanut and myrobolan dye concentration of printed cotton sample showed maximum weighted mean score (3.46) followed by 0.4 per cent gum concentration sample (3.00) with respect to depth of colour.

Evenness of print: Cotton samples which were screen printed using 100 per cent arecanut dye using 0.5 per cent *kokilaksha* gum concentration without mordant reported maximum weighted mean score (3.30) followed by 0.4 per cent gum concentration sample (2.86). On the other hand cotton printed samples with 0.5 per cent *kokilaksha* gum concentration using 50:50 arecanut and myrobolan dye concentration showed greater weighted mean score (3.86) followed by 0.4 per cent gum concentration sample (3.60) with respect to evenness of print. Similarly samples printed with 75:25 arecanut: myrobolan dye concentration of 0.5 per cent *kokilaksha* gum concentration showed maximum mean score (4.13) followed by 0.4 per cent gum concentration sample (3.93).

Sharpness of print: It was observed from the Table 1 that cotton samples printed with 100 per cent arecanut dye using 0.5 per cent *kokilaksha* gum concentration recorded highest weighted mean score (3.80) followed by 0.4 per cent gum concentration sample (3.40). On the other hand cotton printed samples using 50:50 arecanut: myrobolan dye proportion with 0.5 per cent gum concentration showed maximum weighted mean score (3.93) followed by 0.4 per cent gum concentration sample (3.80) towards sharpness of print. Similarly 75:25 arecanut and myrobolan dye concentration using 0.5 per cent *kokilaksha* gum concentration printed samples reported greater weighted mean score value (4.13) followed by 0.4 per cent gum concentration sample (3.93) with respect to sharpness of print.

Overall appearance: Overall appearance of cotton sample screen printed with using 100 per cent arecanut dye of 0.5 per cent *kokilaksha* seed gum concentration exhibited highest weighted means (4.26) followed by 0.4 per cent gum concentration (3.53). In the same way 50:50 arecanut: myrobolan ratio using 0.5 per cent gum concentration of screen printed cotton samples showed maximum weighted mean score value 3.93 followed by 0.4 per cent gum concentration sample (3.16) with respect to overall appearance. Similarly 0.5 per cent and 0.4 per cent *kokilaksha* gum concentration of 75:25 arecanut: myrobolan proportion printed samples reported highest weighted mean score values 4.06 and 4.00 respectively towards overall appearance. Statistically it was observed that 100 per cent, 75:25 and 50:50 arecanut: myrobolan ratio printed samples using kokilaksha seed gum reported non-significant difference.

Results recorded in Table 1 summerizes that cotton samples screen printed with 75:25 arecanut: myrobolan ratio using 0.5 per cent *kokilaksha* gum without mordant exhibited higher weighted mean score than the 100 per cent areanut and 50:50 arecanut: myrobolan printed samples. *Kokilaksha* seed gum was found to be rich in carbohydrates, starch and free radicals which help to bind the gum molecules and dye molecules for proper consistency, and viscosity of the *kokilaksha* gum was higher than sodium alginate. *Kokilaksha* seed gum contains high starch content that is naturally occurring polymer, biodegradable, inexpensive and are abundantly available. These polymers when soaked in water produces viscose paste and widely distributed in the form of tiny granules, that is suitable for printing.

Records presented in Table 2 that visual evaluation of cotton samples screen printed with different concentrations of arecanut: myrobolan (with $FeSO_4$) for optimization of the *kokilaksha* seed gum.

Table 2: Visual evaluation (WMS) of cotton samples screen printed with different concentrations of arecanut:myrobolan pigment (With $FeSO_4$ mordant) for optimization of the *kokilaksha* gum concentration N = 30

Gum Conc. (%)	100% Arecanut					Arecanut: Myrobolan (50:50)					Arecanut: Myrobolan (75:25)				
	Weighted mean score (WMS)														
	DC	EP	SP	OA	Average	DC	EP	SP	OA	Average	DC	EP	SP	OA	Average
0.1	2.93	2.93	3.30	3.20	3.09	2.30	2.93	3.33	3.20	2.94	4.20	3.53	3.40	4.07	3.80
0.2	2.80	3.73	3.93	4.06	3.63	2.80	2.80	3.93	4.06	3.39	3.00	3.33	3.40	3.53	3.23
0.3	3.00	3.33	3.60	3.46	3.34	3.00	3.33	3.46	3.46	3.31	3.00	3.33	3.80	3.47	3.48
0.4	3.00	3.93	3.73	3.80	3.61	3.00	2.87	3.73	3.80	3.61	3.80	4.07	4.00	4.27	4.03
0.5	3.46	4.13	4.20	4.06	3.96	3.46	4.13	4.20	4.06	4.00	3.87	4.00	4.20	4.33	4.10

Paired t-test

Arecanut: myrobolan ratio	Mean ± SD	Mean ± SD	t-value
100:0 and 50:50	3.52 ± 0.32	3.45 ± 0.39	1.46 ^{NS}
100:0 and 75:25	3.52 ± 0.32	3.72 ± 0.36	1.09 ^{NS}
50:50 and 75:25	3.45 ± 0.39	3.73 ± 0.36	0.34 ^{NS}

DC-Depth of colour, EP-Evenness of print, SP-Sharpness of print, OA- Overall appearance
WMS – weighted mean score
Significant at 0.05 level of significance
NS- Non-significant
N – Significant

Depth of colour: Cotton samples screen printed with pure arecanut slurry and 50:50 proportion of arecanut: myrobolan concentration with ferrous sulphate mordant imparted highest (3.46) weighted mean score followed by 0.4 and 0.3 per cent gum concentrations, whereas highest (4.20) weighted mean score was observed for cotton samples printed with 75:25 arecanut: myrobolan pigment which had 0.5 per cent *kokilaksha* gum concentration with ferrous sulphate mordant followed by 0.4 per cent gum (3.87) concentration towards depth of colour.

Evenness of print: Samples screen printed with 100 per cent arecanut slurry using 0.5 per cent *kokilaksha* gum with ferrous sulphate exhibited highest (4.13) weighted mean score followed by 0.4 per cent gum concentration printed sample (3.93) towards evenness of print. Similarly cotton samples screen printed with 50:50 arecanut: myrobolan pigment using 0.5 per cent *kokilaksha* gum concentration imparted highest (4.13) value than 0.4 per cent gum concentration (3.93) sample. Cotton sample screen printed with 75:25 arecanut and myrobolan proportion of 0.5 per cent *kokilaksha* gum concentration using ferrous sulphate depicted highest (4.07) value followed by 0.4 per cent gum concentration for evenness of print.

Sharpness of print: Cotton samples screen printed with pure arecanut slurry using 0.5 per cent *kokilaksha* gum concentration with ferrous sulphate mordanted sample exhibited highest (4.20) weighted mean score followed by 0.4 per cent gum (3.90) concentration. The sample screen printed with 50:50 arecanut and myrobolan pigment using 0.5 per cent *kokilaksha* gum with ferrous sulphate imparted greater (4.20) mean values followed by 0.4 per cent gum (3.93) concentration towards sharpness of print. Whereas cotton samples printed with 75:25 arecanut: myrobolan pigment using 0.5 per cent and 0.4 per cent gum concentrations with ferrous sulphate mordant exhibited highest (4.20) and (4.00) weighted mean scores respectively for sharpness of print.

Overall appearance: It was observed from Table 2 that cotton samples screen printed with pure arecanut slurry using 0.5 per cent and 0.4 per cent *kokilaksha* gum concentrations depicted highest (4.06) weighted mean scores towards overall appearance of the print. Whereas maximum mean values (4.06) was observed for cotton samples printed with 50:50 arecanut: myrobolan pigment using 0.5 per cent *kokilaksha* gum concentration followed by 0.4 per cent gum concentration (3.96) sample. However 75:25 arecanut: myrobolan concentration with ferrous sulphate printed sample scored highest (4.33), (4.27) and 4.07 values towards overall appearance of the print. Further statistically it was observed that there is no significant difference for 100 per cent arecanut, 50:50 arecanut: myrobolan and 75:25 arecanut and myrobolan proportions.

All the mordanted samples scored higher ranks for visual evaluation, it may be due to the reason that, with the use of mordant there was variation in shade and hence the overall appearance of design appeared better (Yadav *et al.*, 2016). Table 2 revealed that samples printed with 75:25 arecanut: myrobolan of 0.5 per cent gum with $FeSO_4$ mordanted were accepted by the majority of the respondents than the samples printed with 100 per cent arecanut and 50:50 arecanut: myrobolan printed samples. Starch powders / gums readily dissolve in water; whereas mucilage forms slimy masses most of these molecules are smaller in size, which in turn suggests more absorbency of the dye source by the textile material during printing.

Mordants helps to fix the dye molecules on the surface of fabric and maximum amount of bonding takes place with the help of mordants in dyeing and printing process. Success of printing process is established by the sharpness mark, levelness, correct of colour, good hand, which are directly influenced by the rheology of the thickener (Asaduzzaman *et al.*, 2016) [2].

Table 3: Colour fastness properties of cotton samples screen printed with arecanut: myrobolan pigment using *kokilaksha* seed gum (0.50%), after treated with 5 gpl salt solution

Concentration (Arecanut: Myrobolan)	Colour fastness grades											
	Light	Washing			Perspiration						Rubbing	
					Acid			Alkali			Dry	Wet
		CC	SC	SW	CC	SC	SW	CC	SC	SW		
Without mordant(control)												
50:50	2/3	3	2/3	2/3	3/4	3	2	3	3	2/3	3	2/3
75:25	3/4	3	2/3	3	3	2/3	2/3	3	3	3/4	3	2/3
With ferrous sulphate ($FeSO_4$)												
50:50	4	3/4	3	3/4	3/4	3	3/4	3/4	3/4	3/4	3/4	2/3
75:25	4	3/4	3	3/4	3/4	3	2/3	3/4	3/4	3	3/4	3
CC –Change in colour SC - Stain on cotton SW – stain on wool												

Ratings: 1-5, where 1 - poor, 2 - fair, 3 - good, 4 - very good and 5 - excellent
Ratings: 1-8, where 1 - poor, 2 – low, 3 - average, 4 - rather average, 5 - good, 6 – very good, 7 – extremely good and 8 – excellent

Colour fastness properties of cotton samples screen printed with arecanut: myrobolan pigment (0.50%) *kokilaksha* gum after treated with 5 gpl salt solution

Data presented in Table 3 reveals the light fastness properties of cotton samples printed with arecanut: myrobolan dye using *kokilaksha* gum, after treated with 5 gpl salt solution.

Light fastness: Cotton samples screen printed with arecanut: myrobolan dye (75:25 and 50:50) each and 0.5 per cent *kokilaksha* as natural gum substance in absence of mordant resulted low to average (2/3) and average to rather average (3/4) range of fastness ratings properties on exposure to sunlight. It was evident from the Table 3 that light fastness ratings of samples printed with ferrous sulphate mordant with arecanut: myrobolan concentration and 5 gpl salt after treatment showed rather average (4) ratings. Fading in sunlight is partly due to ultraviolet radiation that initiates chemical degradation of loosely held electrons of chromophores (Babel and Gupta, 2016) [4]. Light fastness of printed fabric was influenced by chemical, physical state, concentration of dye, nature of fibres and mordant type (Cristea and vilarem, 2006) [5].

Wash fastness: Wash fastness of cotton screen printed fabric samples depicted good (3) for change in colour and fair to good (2/3) for colour staining with respect to both the ratios of dye concentration (50:50 and 75:25, arecanut: myrobolan) with 0.5 per cent *kokilaksha* gum in absence of mordant. Whereas, in presence of ferrous sulphate mordant, good to very good (3/4) fastness for change in colour was obtained with 50:50 and 75:25 arecanut: myrobolan dye concentration of 0.5 per cent *kokilaksha* gum with curing, steaming and salt

treatment (5gpl). Moreover samples printed with 50:50 and 75:25 concentration of arecanut: myrobolan with mordant samples recorded good to very good (3/4) wash fastness properties than the samples printed without mordant that may be due to the hydrolyses of dye molecules in the absence of the mordant.

Perspiration: Acidic perspiration fastness of cotton screen printed fabric with 50:50 arecanut: myrobolan dye concentration of 0.5 per cent *kokilaksha* gum without and with $FeSO_4$ mordant showed change in colour as good to very good (3/4). Nevertheless, good to very good (3/4) in presence of ferrous sulphate mordant of both dye concentration was noted. In alkaline perspiration, change in colour was observed good to very good (3/4) in both dye concentrations for both mordanted and unmordanted samples. However, rating of staining on cotton was good to very good (3/4) in presence of ferrous sulphate mordant at both the dye concentration. There was slight colour staining on both samples on both sides with agents. The good colourfastness properties might be attributed to the benzene ring and conjugated system present in dye which makes fair bonding with modified fabric upon exposure to agents like heat and rubbing resistance (Babel and Gupta, 2016) [4].

Rubbing fastness: Dry rubbing fastness of 50:50 and 75:25 arecanut:myrobolan of 0.5 per cent *kokilaksha* gum with mordant and without mordant exhibited good (3) and good to very good (3/4) respectively. However, on the other hand, irrespective of the dye concentration and in absence of mordant good to very good (3/4) result was observed in wet rubbing fastness. Further, in presence of ferrous sulphate mordant fair to good (2/3) and fair (2) rating was observed with 50:50 and 75:25 per cent dye concentrations. Rubbing fastness grades of samples printed with mordant exhibited good to very good (3/4) rubbing fastness compared to samples printed without mordant that exhibited fair to good (2/3) rubbing fastness. Rubbing fastness depends on gum properties adhesion on the textile materials and even dispersing of the dye in the printing paste (Yaman *et al.*, 2012) [8]. The excess deposition of the dye held on the fabric surface by adsorption get disturbed and reorientation causes the fading of colour due to rubbing action. Agarwal *et al.* (2007) [1] concluded the same results as use of mordants in printing improved the fastness properties of printed samples.

Conclusion: Eco-printed cotton samples without mordant and with $FeSO_4$ mordant using 0.5 per cent *kokilaksha* gum concentration exhibited higher depth of colour, evenness of print, sharpness of print and overall appearance compared to other concentrations (0.1 – 0.4 per cent). With $FeSO_4$ mordant printed samples exhibited good to very good (3/4) colour fastness properties for wash, perspiration and rubbing compared to without mordant printed samples. There was slight colour change in all samples printed without mordant. Samples printed with 50:50 and 75:25 arecanut: myrobolan exhibited fair to good (2/3) wash, perspiration and rubbing fastness properties in without mordanted samples.

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