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GCMS analysis and colour fastness properties of silk yarn dyed with *Acacia nilotica* pods

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Abstract
Natural dyes possessed several advantages over synthetic dyes from the point of health, safety and ecological aspects. It has unique, soft and soothing colour shades with multi-functional properties *viz.*, anti-allergic, anti-microbial and fungal compared to synthetic dyes *Acacia nilotica* is commonly called as Babul, considered as a very important economic plant and traditionally has been used as a source for tannins, gums, timber, fuel, fodder and medicine. The solvent extract of *Acacia nilotica* pods were subjected to GCMS analysis and it was found that the source possessed 12 major compounds with varied area percentage. The area percentage of Pyrogallol was found to be higher (72.62 Per cent) followed by 6 Octadecanoic acid (7.87 Per cent) and 1, 2 Benzenediol, Pyrocatechol (6.66 Per cent). The aqueous extract of optimised *Acacia nilotica* pods dye concentration was used for dyeing pre mordanted mulberry silk yarn with four metallic mordants *viz.*, potash alum, stannous chloride, copper sulphate and ferrous sulphate. The dyed silk yarn was subjected to colour strength, colour co-ordinates and colour fastness properties. Irrespective of mordants, the fastness properties of silk yarn possessed good to excellent colour fastness to dry crocking and sunlight. The sample possessed good colour fastness to acidic perspiration than the alkaline perspiration. Further, the wash fastness of all the mordanted silk yarn exhibited good to excellent fastness properties even after 10th wash. Thus *Acacia nilotica* pods possessed numerous phytochemicals which are suitable for colouration of silk which yields good to excellent fastness properties and the potential source creating new enterprise among the silk dyers and printers.

Keywords: *Acacia nilotica* pods, colour strength, colour co-ordinates, colour fastness, GCMS, metallic mordants

Introduction
Natural colorants find the application in a wide range of colouration like dyeing and printing of textiles, paper, rubber, paints, and plastics. Natural dyes have several advantages over synthetic dyes from the point of health, safety and ecology with uncommon, soothing and soft shades compared to synthetic dyes (Singh, 2000) [6]. Natural dyes have been used to colouring the textiles since time immemorial. People all over the world have been using plant sources *viz.*, roots, stem, bark, leaves and flowers for textile colouration. In recent years, interest has been manifested towards natural dyes because of ecological movement, bio degradability and higher compatibility of natural dyes with environment. Other advantages associated with natural dyes include lower toxicity and allergic reactions in relation to synthetic dyes (Victoria, 2014) [8].

Acacia nilotica is considered as a very important economic plant since early times as it was used as a source in tannins, gums, timber, fuel, fodder and medicine. Gum is widely used in paints, medicines, industrial, food and medicinal purposes. Timber is widely used in building constructions, railway sleepers, mine props, tool handles and carts. The gum is frequently used in calico printing and dyeing as a thickening agent. It is also used as sizing material for silk and cotton (Abhishek, 2015) [1]. The species has an inspiring range of medicinal uses with potential anti-oxidant activity. The methanol extracts of *Acacia nilotica* showed significant inhibition against Gram-positive and Gram-negative bacteria. *Acacia nilotica* dye source absorbs approximately 20-30 Per cent radiations in UV-B region referred as good UV protection which is suitable for use in sun protective clothing (Malviya, S. *et al.*, 2011) [4].

Silk is one of the major textiles that possesses a combined set of aesthetic properties that make it useful for high-fashion luxury textile goods. Silk exihibeted lustrous, translucent, good drapability, crisp hand and pleasing appearance (Mahale G. *et al.*, 2003) [3]. It is highly moisture absorbent and excellent to wrinkle resistance, exhibits fair abrasion resistance and good resistance to pilling,

sensitive to chlorine bleaches and to alkalies and is easily damaged by sunlight. It has a greater affinity for natural and synthetic dyes with better fastness properties (www.Ibef.Org).
Hence the present study was designed with the following objectives, characterization of *Acacia nilotica* pods through GCMS analysis, optimisation of dyeing conditions for colouring the silk and to assess the colour fastness properties of dyed silk yarn.

Fig 1: Acacia nalotica pods

Material and Methods

Fresh *Acacia nilotica* pods were collected from local forest of Karnataka State (Fig 1). The collected fresh pods were shade dried and crushed into fine powder by traditional pounding technique and multivoltine yellow race degummed mulberry silk was used for dyeing with *Acacia nilotica* pods. These pods were subjected to GCMS analysis to estimate the compounds present in the source using standard test procedures.

GCMS spectral analysis of solvent extract of *Acacia nilotica* pods

Gas Chromatography Mass Spectrometry (GCMS) is a powerful technique to provide the identification of compounds with low detection limits and potential for quantitative analysis. The *Acacia nilotica* pods extract was analysed by GCMS as per the test condition.

Column :	RT x 5 MS (30m x 0.25 mm x 0.25 µm)
Oven Temperature:	60^0C (3 min)@10^0C/min., 150^0C @ 5^0C/min
Injector Temperature:	280^0C
Detector Temperature:	280^0C

Dyeing conditions

The powdered *Acacia nilotica* (5, 10, 15 and 20g) was soaked overnight in different M.L.R (1:20, 1:30 and 1:40) to optimize the dye-concentration and was extracted by aqueous method. The dyeing variables are influenced on reflectanced and colour strength of the dye source. The variables like, method of extraction, time of extraction (30, 45, 60 min), dyeing time (30, 45, 60 min) were optimized based on the spectral value at 430 (λ) wavelength of the dye source. Based on the reflectance and K/S value, 10g dye-concentration, 1:30 M.L.R, 30 min extraction-time were optimised for dye extraction and further used for silk dyeing.
The natural mordant myrobolan and four metallic salts *viz.,* potash alum (5, 10 and 15%), copper sulphate and ferrous sulphate (1, 2 and 3%) and stannous chloride (0.5, 1 and 1.5%) in pre, simultaneous and post mordanting methods were used for the study and based on the colour strength and colour co-ordinates the pre mordanted method was used for dyeing. The myrobolan treated mulberry silk yarn dyed with *Acacia nilotica* pods extract in optimised dye bath (1:40) M.L.R at 30 min dyeing time. Irrespective of mordants, mordant concentration and mordanting methods, pre-mordanted dyed silk yarn with alum (15%), stannous (0.5%), copper and ferrous (3%) shows increased K/S value & colour co-ordinates hence these mordanted samples were used for assessing the colour fastness tests using the following BIS standards.

- Colour fastness to rubbing (IS 766-1988)
- Colour fastness to perspiration (IS 971-1983)
- Colour fastness to sunlight (ISO: 105B)
- Colour fastness to washing (IS: 3361-1979)

Results and Discussion

GCMS analysis

The analysis and extraction of plant material play an important role in the development, modernization and quality control of herbal formulations. Hence the present study was aimed to find out the bioactive compounds present in the solvent extract of *Acacia nilotica* by using Gas chromatography and Mass spectroscopy. The active compounds with their peak number, concentration (peak area %), and retention time (RT) are presented in Table 1, Fig 2 and Fig 3 which showed the presence of 12 bioactive phytochemical compounds in the solvent extract of *Acacia nilotica* pods (Seema Bai *et al.,* 2014) [5].
Based on the spectral value it was found that the area percentage of Pyrogallol was found to be 72.76 per cent at retention time 14.877 with base peak m/z 126.10 followed by 6 Octadecanoic acid 7.87 per cent, 26.749 retention time with base peak of 55.05 m/z, 1,2 Benzenediol, Pyrocatechol (6.66 per cent) at 11.785 retention time and 110.05 base peak, Pentadecanoic acid (5.59 per cent) at retention time 23.590 with the base peak of 73.05 respectively. Further, the source exhibited 2.17 area percentage of Octa decanoic acid, Dibutyl phthalate (1.65 %) and Benzene (1.48 %) respectively and compounds present in the powder ranged from 0.10 - 0.50 per cent *viz.,* Palmitoyl chloride (0.51 %), 1,2-Benzenedicarboxylic acid (0.38 %), Squalene (0.36), Hexadecanoic acid (0.36) at different retention time and base peak m/z. The major phenols present in the source were pyrocatechol and pyrogallol. Pyrocatechol exhibits potential anxiolytic, antinociceptive and antimicrobial properties and is used in pharmaceuticals. Pyrogallol has anticeptic properties and is used in synthetic drugs, medicine and laboratory reagents.
Due to the presence of above mentioned compounds in the solvent extract of *Acacia nilotica* pods it can be stated that the pods have numerous phytochemicals which can be a good bio resource for textile colourants, pharmaceutical and industrial applications.

Table 1: GCMS spectral analysis of Solvent extracts of *Acacia nilotica* pods

S. No.	R. Time	Area%	Base peak m/z	Name of the compounds	Formula
1	4.216	1.48	112.00	Benzene, chloro-chlorobenzene, Monochlorobenzene MCB, Phenyl chloride, Benzene chloride	C_6H_6
2	11.785	6.66	110.05	1,2-Benzenediol, Pyrocatechol, o-Benzenediol, o-Dihydroxybenzene, o-Dioxybenze	$C_6H_6O_2$
3	14.877	72.76	126.10	1,2,3-Benzenetriol, Pyrogallol, C.I. Oxidation Base 32, C.I. 76515, Fouramine Brown AP, Fourrine	$C_6H_6O_3$
4	22.515	0.36	74.05	Hexaadecanoic acid, methyl ester, palmitic acid, methyl ester, n-Hexadecanoic acid methyl ester	$C_{16}H_{32}O_2$
5	23.385	1.65	149.05	Dibutyl phthalate, 1,2-Benzenedicarboxylic acid, dibutyl ester, Phthalic acid, dibutyl ester	$C_{16}H_{22}O_4$
6	23.590	5.59	73.05	Pentadecanoic acid, Pentadecyclic acid, n-Pentadecanoic acid, n-Pentadecyclic acid	$C_{15}H_{30}O_2$
7	25.596	0.20	67.05	9,12-Octadecadienoic acid(Z,Z)-, methyl ester, Linoleic acid, methyl ester, Methyl cis, cis-9, 12-octadecadien	$C_{18}H_{32}O_2$
8	26.749	7.87	55.05	6,Octadecenoic acid, (Z)- (6Z)-Octadecenoic acid	$C_{18}H_{34}O_2$
9	27.091	2.17	73.05	Octadecanoic acid, Stearic acid, n-Octadecanoic acid, Humko Industrene R, Hydrofol Acid 150,	$C_{18}H_{36}O_2$
10	32.178	0.51	98.10	Palmitoyl chloride, Hexadecanoyl-chloride- Palmitic acid chloride	$C_{16}H_{31}ClO$
11	33.032	0.38	149.05	1,2-Benzenedicarboxylic acid, mono(2-ethylhexyl) ester, Mono (2-ehylhexyl), phthalate, Phthalic acid	$C_8H_6O_4$
12	36.147	0.36	69.10	Squalene 2,6,10,14,18,22-Tetracosahexaene, 2,6,10,15,19,23-hexamethyl-Skvalen Spinacene	$C_{30}H_{50}$

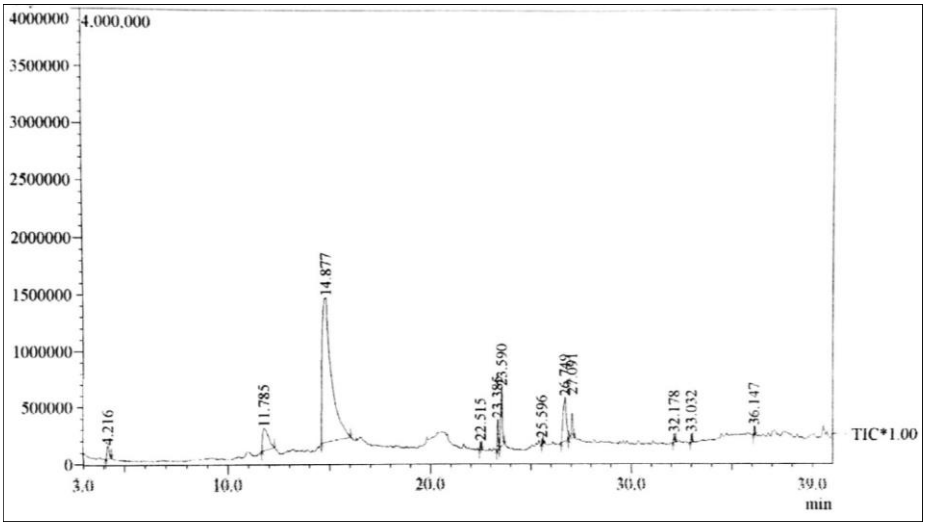


Fig 2: GCMS spectral graph of solvent extracts of *Acacia nilotica* pods

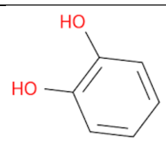
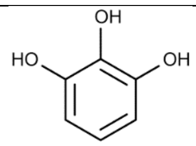
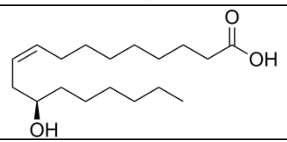
Name of the Compound	Formula	Structure
Pyrogallol	$C_6H_6O_3$	
Pyrocatechol	$C_6H_6O_2$	
6,Octadecenoic acid,	$C_{18}H_{34}O_2$	

Fig 3: Chemical Structure of major compound present in *Acacia nilotica* pods

Colour strength and Colour co-ordinates of mulberry silk yarn mordanted with metallic mordants

The pre mordanted silk yarn with ferrous sulphate exhibited significantly greater colour strength (141.54) followed by copper sulphate (30.98), stannous chloride (29.72) and potash alum (16.96) respectively. The samples treated with potash alum exhibited lighter shades with less redder and yellower compared to silk yarn mordanted with stannous chloride and copper sulphate. Colour co-ordinates of silk yarn treated with ferrous sulphate showed darker shades, more redder and yellower than the other samples. This may be due to iron salts, such as ferrous sulphate as transition metal mordant form a large number of complexes with the dye molecules, mostly octahedral ones with coordination number 6. As a result, some coordination sites remain unoccupied when they interact with the fiber and at that time functional groups such as amino and carboxylic groups on the silk fiber can occupy these unoccupied sites. Thus, ferrous sulphate salts can form a ternary complex on one site with the fibre and in the other site with the dye resulted in to higher dye uptake as well as darker shades (Uddin, 2014) [7].

Table 2: Influence of colour strength and colour co-ordinates of pretreated silk yarn mordanted with metallic mordants.

S. No.	Mordants	Optimised mordant concentrations	K/S	L*	a*	b*
1	Potash Alum	15%	16.96	69.98	2.69	27.97
2	Stannous Chloride	0.5%	29.72	63.12	5.75	29.72
3	Copper Sulphate	3%	30.98	60.47	5.78	24.34
4	Ferrous Sulphate	3%	141.54	27.56	5.39	3.94

S. No.	Mordants	K/S		L*		a*		b*	
		CD 5%	CV	CD 5%	CV	CD 5%	CV	CD 5%	CV
1	Potash Alum	1*	0.13	0.40*	0.08	0.71*	0.01	0.04*	0.01
2	Stannous Chloride	1*	0.19	-0.31*	-0.06	0.71*	0.02	-0.55*	0.07
3	Copper Sulphate	1*	0.13	0.05*	0.03	0.27*	0.01	-0.79*	0.07
4	Ferrous Sulphate	1*	0.26	-0.93*	-0.01	0.06*	0.01	-0.98*	-0.01

*Significant @ 5% level
K/S: Colour strength
L: The lightness/darkness co-ordinate
a*: The red/green co-ordinate with +a* indicating red -a* indicating green
b*: The yellow/blue co-ordinate with +b* indicating yellow and -b* indicating blue

Colour fastness properties of mulberry silk yarn mordanted with optimised mordant concentrations

Colour fastness to crocking

The colourfastness to dry and wet crocking assessed in terms of change in colour as well as colour staining. Irrespective of mordant concentrations, mordanting methods, the mordanted samples showed better colourfastness than the control (Table 3). Colourfastness to dry crocking was found to be better compared to wet crocking in all the samples mordanted with optimised concentration. Of the four mordants, the dry crocking samples found good to excellent (4/5 to 5) fastness grades in colour staining and excellent grades (5) in colour change than the control sample (4 to 4/5). However, wet crocking samples exhibited fair to good (3/4 to 4/5) grades in both colour change and colour staining.

On the whole, the moranted silk yarn dyed with *Acacia nilotica* pods exhibited good to excellent (4/5 to 5) colour fastness to dry crocking and fair to good colour fastness to wet crocking due to solublation of a part of dye and its migration to the surface of the coloured specimen. Breakage of dye metal complexes into simple particles during wet crocking and release of superficially deposited dye molecules from dry substance to wet, thus resulting into slightly stain on the material.

Table 3: Effect of crocking on colour fastness (ratings) of dyed silk with optimised mordant concentrations and mordanting methods

S. No.	Mordants	Mordant concentrations (%)	Mordanting methods	Rubbing			
				Dry		Wet	
				CC	CS	CC	CS
1	Control	-	Without mordant	4/5	4	4	3/4
2	Potash Alum	15%	Pre mordanting	5	4/5	4	4/5
3	Stannous Chloride	3%		5	5	4/5	4/5
4	Copper Sulphate	0.5%		5	4/5	4/5	3/4
5	Ferrous Sulphate	3%		5	4/5	4	4/5

Note: CC- Colour change, CS- Colour staining
Ratings: 1-Very poor, 2-Poor, 3-Fair, 4-Good, 5-Excellent

Colour fastness to perspiration

Table 4 illustrates, the effect of acidic and alkaline perspiration of *Acacia nilotica* dyed samples on change in colour and stain on cotton and silk materials. The mordanted samples exhibited better colourfastness (4-5) than the control sample which may be due to the combined effect of mordants-fibre-dye interaction. Irrespective of mordants and mordant concentrations the colourfastness to acidic perspiration showed good, good to excellent (3-4, 4-5) than the alkaline perspiration due to the stable electronic configuration of silk yarn in acidic condition (Gohl and Vilensky, 1987) [2]. Further, the stain on cotton in acidic perspiration was found to be relatively less than alkaline, due to synergistic combined effect of metal and heat on dye emulsion which causes fading of dye at selective points whereas no stain was observed on silk material. Of the four mordants, the silk yarn dyed with ferrous sulphate mordant exhibited excellent fastness to acidic (5) and alkaline (4-5) perspiration than the samples treated with other mordants.

Table 4: Effect of perspiration on colour fastness (ratings) of dyed silk with optimised mordant concentration and mordanting methods

S. No	Morants	Mordant concentrations (%)	Mordanting methods	Perspiration					
				Acid			Alkali		
				CC	CS		CC	CS	
					S	C		S	C
1	Control	-	Without mordant	4	4/5	3/4	3/4	3/4	3/4
2	Potash Alum	15%	Pre mordanting	4/5	5	5	4	4/5	4
3	Stannous Chloride	3%		4/5	4/5	4/5	4	4/5	3/4
4	Copper Sulphate	0.5%		4/5	4/5	4/5	4/5	3/4	4/5
5	Ferrous Sulphate	3%		5	5	5	4/5	5	5

Colour fastness to sunlight

The effect of sunlight on colourfastness of *Acacia nilotica* dyed silk yarn is presented in Table 5. The mordanted samples showed good to excellent (7/8) colourfastness to sunlight compared to control (5) this may be due to probably formation of complex with transitional metal which protects the chromophore from photolytic degradation and the photons sorbed by the chromophoric group dissipate their energy by resonating within the six member ring thus forming and protecting the dye and improve the light fastness. It may also be due to the stable arrangement of electrons which is resistant to photo degradation by the UV rays stated by Gohl and Vilensky (1987) [2].

Table 5: Effect of sunlight on colour fastness (ratings) of dyed silk with optimised mordant concentrations and mordanting methods.

S. No.	Mordants	Mordant concentrations (%)	Mordanting methods	Colour fastness to sunlight
1	Control	-	Without mordant	5
2	Potash Alum	15%	Pre mordanting	6
3	Stannous Chloride	3%		6/7
4	Copper Sulphate	0.5%		7/8
5	Ferrous Sulphate	3%		7/8

Ratings:

Very poor light fastness	1
Poor light fastness	2 3
Fair light fastness	4 5
Very good light fastness	6
Excellent light fastness	7 8

Colour fastness to washing

Table 6 discloses the effect of washing on colour fastness with respect to change in colour and colour on stain material for different washing cycle's *viz.*, 5th, 10th, and 15th wash. It was interesting to observed that all the silk samples dyed in *Acacia nilotica* pod extract possessed good wash fastness even after 15th wash. There was no much change observed in all the samples subjected to washing cycles *i.e.*, 5 to 15th indicating that samples are good fastness to washing. The stain on cotton and silk sample found excellent (5) and colour change was good to excellent (4/5) for all the mordanted samples in 5th, 10th and 15th washes than control sample. Irrespective of mordants, the wash fastness of dyed silk showed good to excellent fastness to washing than the control (3/4).

The wash fastness of all the samples *i.e.*, control and mordanted samples showed good to excellent results even after 15th wash. This may be due to, effect of dye fixes, (mordants) dye OH^+ group and silk $-NH^2$ group may combine to form insoluble bigger size complex either inside the fibre or on the surface of the dyed fibres to improve their wash fastness properties.

Table 6: Effect of washing on colour fastness (ratings) of dyed silk with optimised mordant concentration and mordanting methods

Mordents	Mordant concentration (%)	Mordanting method	5th wash			10th wash			15th wash		
			CC	CS		CC	CS		CC	CS	
				S	C		S	C		S	C
Control	-	-	4	5	5	4	5	5	4	5	4/5
PA	15%	Pre	4/5	5	5	4/5	5	5	4/5	5	5
SC	3%	Pre	4/5	5	5	4/5	5	5	4	5	5
CS	0.5%	Pre	4/5	5	5	4/5	5	5	4	5	5
FS	3%	Pre	4/5	5	5	4/5	5	5	4	5	5

Note: CC- Colour change CS- Colour staining, C-Cotton, S-Silk
Ratings: 1-Very poor, 2-Poor, 3-Fair, 4-Good, 5-Excellent

Conclusion

Acacia nilotica pods are traditionally being used as a supplement to poultry rations and fodder for small animals like sheep and cattle. Based on the findings of the study it can be enclosed that the solvent extract of *Acacia nilotica* pods exhibited 12 major compounds which are useful for textile colouration and pharmaceuticals. The mulberry silk yarn dyed with *Acacia nilotica* pods extract in presence of different metallic mordants showed good to excellent colour fastness properties with unique colour shades. Thus, the natural dyeing of *Acacia nilotica* pods with silk yarn is unique and a new venture for the dyers, printers and fashion designers. Rural folk can take up these activities as an enterprise to sustain their livelihood.

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