IJCRT.ORG ISSN : 2320-2882

INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)

An International Open Access, Peer-reviewed, Refereed Journal

Dyeing properties of natural dye extracted from *Tamarindus Indica* bark on cotton fabric

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1. ABSTRACT

This paper investigates the application of dye extracted from *Tamarindus Indica* (bark) in an aqueous solution and used to dye cotton fabric. Application of colours on textiles was achieved conventionally by using natural dyes which are environment friendly. A revival of commercial interest in natural dyes has opened several research avenues. In the present study, the natural dye were extracted from the bark of *Tamarindus Indica* on cotton fabric has been studied and the study was been carried out to optimize dye extraction prepared in aqueous medium and dyeing conditions for cotton fabric. The effect of variables on extraction and dyeing in terms of pH (4-10), temperature (30ºC -100ºC) and duration (30-90 minutes) were studied.

The dyed cotton samples were subjected to fastness tests and colour strength. Colour fastness property investigations included wash, rubbing, perspiration, light fastness performance and antimicrobial activity. The dyed fabric showed good wash, rubbing, perspiration and moderate light fastness properties. K/S values of the dyed fabrics were also evaluated and the effect of mordant type with different mordanting methods on dyeing was studied and mordanted samples showed better K/S values improved with gallnut compared to alum. Dyed samples showed enhanced colour values, fastness properties and antimicrobial activity with mordants, this outcome specifies dye extracs from the bark of *Tamarindus Indica* it has potential for use in the textile industry.

Keywords: cotton, optimization, mordant, *Tamarindus Indica.*

2. INTRODUCTION

Plants play a vital role in maintaining human health and contribute towards the improvement of human life. These plant materials are a source of dye yielding material, may be applied on natural fabrics to provide value-added products. Natural dyes have many advantages; they are eco-friendly, biodegradable and renewable; after extraction of dyes, the residual vegetal matter left is used as manure. Natural dye is a UV protector, exhibit flame-retardant properties have antibacterial and antiseptic properties.

Plant materials like myrobalan fruits, turmeric, manjishtha root, Arjuna (*Terminalia arjuna*) bark, and safflower florets have curative properties and are used in various traditional medicinal systems. Natural dye possesses healing properties by absorbing therapeutic compounds through the skin; therefore, they are skin-friendly (Gulrajani, 2001). They are important components of medicines, cosmetics, dyes and beverages etc. (Khare, 2004). Although hundreds of plant species were tested for antimicrobial properties

(Nair and Bruke 1990). The application of natural antimicrobial agents on textiles dates back to ancient times (Rani, 2015).

Tamarindus indicia L., belongs to the Dicotyledonous family Leguminosae Sub family Caesalpiniaceae, which is the third largest family of flowering plants with a total of 727 genera and 19, 327 species (Lewis et al., 2005). Almost all tree parts find some use or the other in food, chemical, pharmaceutical, textile industries and as fodder, timber, and fuel. The macerated fresh bark of the young twigs was used both as a purgative and to relieve abdominal pain. (Nagarajan et al., 2014).

3. Materials & Methods:

3.1 Materials

3.1.1 Source: The bark of *Tamarindus Indica* was collected from the agriculture field at K G Lakkenahalli (Karnataka) and dried at room temperature.

3.1.2 Substrate: The plain woven cotton fabric was purchased from Rana's silk store, Jumma Masjid Road, Bengaluru.

3.1.3 Chemicals: Sodium carbonate, Acetic acid, Alum were supplied by Polysales, Chickpet, Bengaluru.

3.1.4 Equipment's used:Weighing balance, Water bath, Hot air Oven, Colorimeter, Hot Plate.

3.2 Method

Crude extraction method was used for extracting the dye.

3.2.1 Preparation of raw material:

The bark of *Tamarindus Indica* was coarsely powdered and then dye was extracted by aqueous crude extraction method and the resultant liquid was then filtered. The extracted dye solutions were then concentrated and then dried in vacum oven at 50-60°C till dry material was obtained. The dry material was then removed from the trays and weighed to ascertain the yield.

3.2.2 Extraction of crude dye material:

Maintaining material-to-liquor (MLR) ratio at 1:40, the bark of Tamarindus Indica was coarsely powdered and then the dye was extracted by aqueous crude extraction method and the resultant liquid was then filtered. The extracted dye solutions were then concentrated and then dried in vacum oven at 50-60°C till dry material was obtained. The dry material was then removed from the trays and weighed to ascertain the yield and the absorbance of the dye solution was recorded before and after dyeing process with colorimeter for silk fabric.

3.3.3 Gas Chromatography-Mass Spectroscopy Analysis

Chromatographic Equipment and Analytical Conditions: GC-MS analysis was carried.

The mass analyser used in the analysis was Quadrupole and the software adopted to handle mass spectra and chromatogram was Xcalibur. The peak obtained were identified using Data analysis, AMDIS and NIST 2011 software.

3.2.4 Dyeing of Silk fabrics

Plain woven cotton fabric with a yarn density of 108 EPI and 106 PPI were selected for dyeing. Dyeing was carried out at optimum conditions for pH ranging from 5 to 10, temperature varying from 30^{o}C to 100^{o}C , duration 30 to 90 minutes and dye concentration ranging 5 to 50%. The dyed samples were subsequently washed in 2 gpl non-ionic detergent at 60^{o}C for 10 minutes and dried at room temperature (Narayanswamy, 2013).

3.2.5 Mordanting method

The substrates were mordanted using 10% (owf) solution each of potassium aluminum sulfate a metallic mordant and Gallnut a bio mordant employed with MLR 1:20 for 30 minutes at 60^{o}C. The samples were later rinsed and dried. The silk fabric dyed without mordant constituted the control. Mordanting methods employed like Pre mordanting was carried out before dyeing in aqueous solution of the mordant. All the mordanted fabrics were then separately dyed. In the Simultaneous mordanting method (i.e., dyeing with mordants) the fabrics were immersed in a bath containing a mordant and dye extract and the temperature was maintained at 90^{o}C throughout dyeing cycle. The fabrics were rinsed, soaped with non-ionic detergent at 60^{o}C washed with water squeezed and dried. The post mordanting method dyeing was carried out in the absence of a mordant, followed by mordanting in a separate bath containing a mordant. The fabrics was rinsed, soaped with non-ionic detergent at 60^{o}C washed with water squeezed and dried.

3.2.6 Absorbance and Colour Strength Measurements

Dye solutions (5,10,15,20,30,40,50%) were prepared from the crude extract by maintaining material-to-liquor (MLR) ratio at 1:40. The absorbance of the dye solution was recorded before and after dyeing process with colorimeter for silk fabrics. The percentage of dye uptake was calculated using the following formula.

$$\% \text{ Dye uptake} = \frac{\text{Absorbance before dyeing} - \text{Absorbance after dyeing}}{\text{Absorbance before dyeing}} \times 100$$

3.2.7 Measurement of surface colour strength and Colour values

The colour strength (K/S values) and reflectance of the samples was measured using Spectrophotometer at λmax of the dyed samples was determined using the Kubelka- Munk equation.

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

Where k is the absorption coefficient, ‘s’ is the scattering coefficient and ‘R’ max is the reflectance value of the fabrics at the wavelengths of maximum absorbance. Colour was been evaluated by means of K/S and CIELAB colour difference values with illuminant D 65/10 observer on Greatag Macbeth Color Eye 7000 A Spectro-photometer. Five measurements were made for each sample and the variation in percentage reflectance values over a range of 350–750 nm was recorded.

Where, R is the observed reflectance, K, the absorption co-efficient and S, the light scattering coefficient.

3.2.8 Assessment of colour strength and fastness properties

Colour fastness properties of the dyed fabric such as to light fastness- light: IS 2454: 2016, wash fastness- washing: IS/ ISO C 10-105 and perspiration- IS 1971:R2004, rubbing (ISO: 105x-12) were assessed as per ISO standard testing methods.

3.2.9 Antimicrobial Screening testTest Microorganisms

The bacterial strains used for the test were *Escherichia coli* (MTCC 443), *Staphylococcus aureus* (MTCC 3160) were the pathogens used in the present study. All stock cultures were obtained from the Microbiology Dept., Bangalore University. Bacterial strains were cultured using Nutrient agar (Himedia). Antibacterial activitywas evaluated by qualitative test methods for dye powder assessment was made by disc diffusion method (AATCC 147-2001) and for treated and untreated fabric samples parallel streak method (AATCC 147-2001), Antifungal activity (AATCC- 30).

4 Results and discussions

4.1 The GC-MS screening indicates chemical compounds belongs to a category like Silyl ester, Silanol, Dicarboxylic acids, Sugars/carbohydrates, Flavonoids, Triterpenol, Ketones, Triterpenoid. 13 metabolites extracted from the plant *Tamarindus indica* (bark) were identified (Table 4.32) and two specific metabolites like amyrin and lupenone were identified.

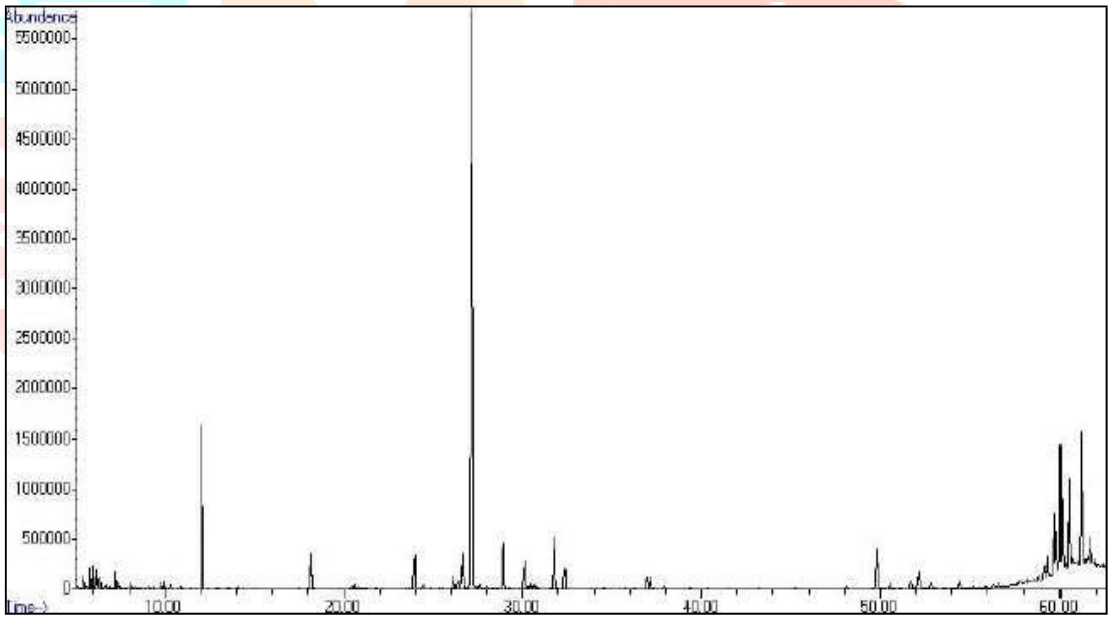
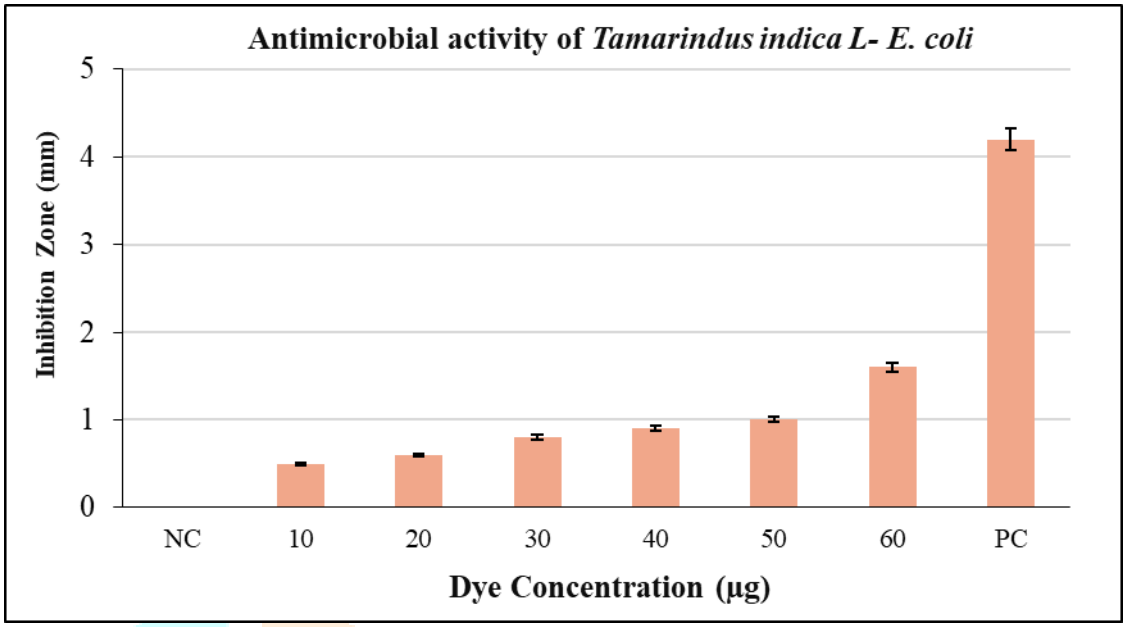
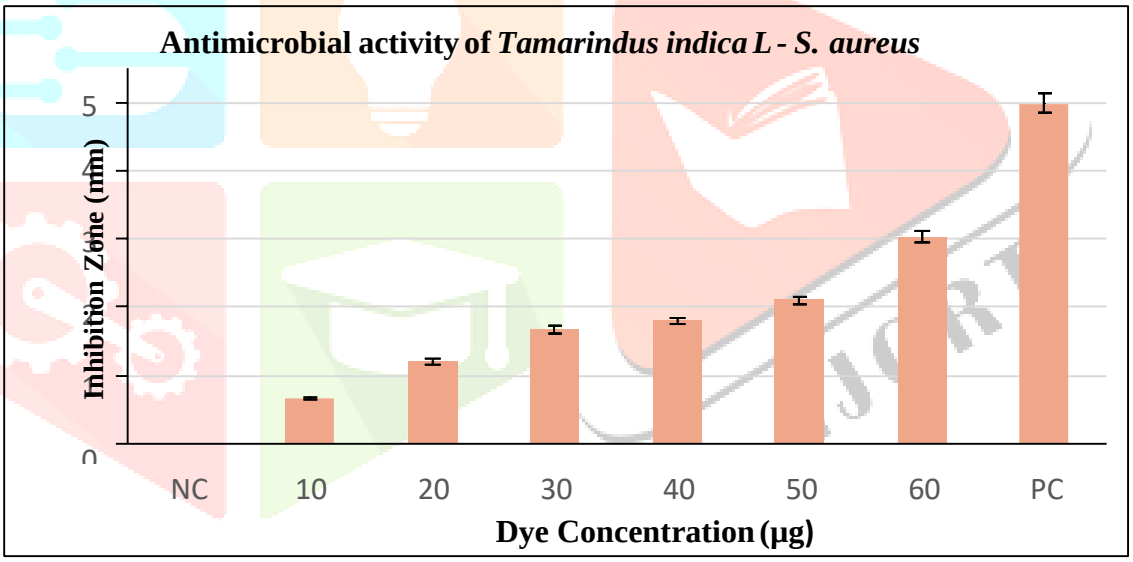


Figure 4.1 Total Ion Chromatogram obtained from the GC-MS study with theextract of *Tamarindus indica L*

4.2 Antimicrobial activity behaviour of dye powder bark of *Tamarindus indica*



Graph 4.1– Antimicrobial activity on dye concentration of *Tamarindus indica L E.coli*



Graph 4.2– Antimicrobial activity on dye concentration of *Tamarindus indica L S. aureus*

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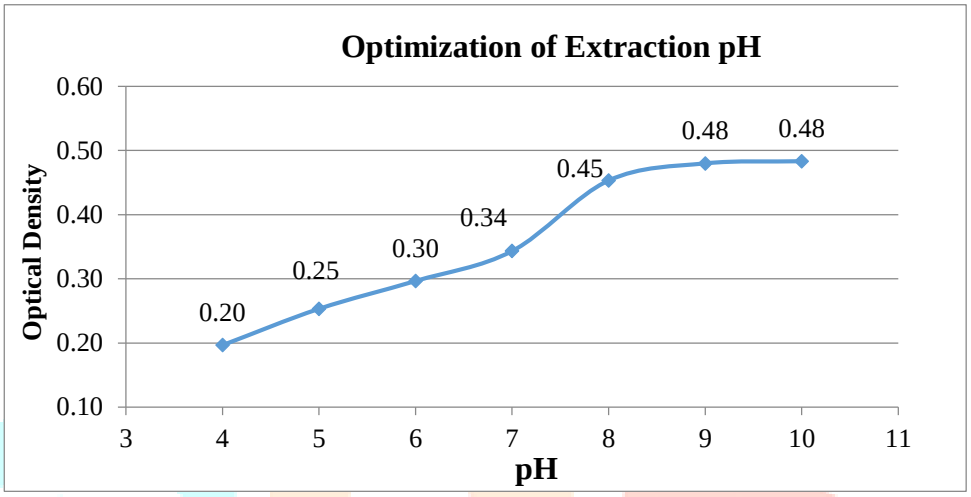
The aqueous extracts of bark of *Tamarindus indica* were subjected to a preliminary screening for antimicrobial activity against bacteria *E. coli, S. aureus* are given. It showed a gradual increase in the zone of inhibition with the increase in dyeconcentration. It was evident that results of 10µg to 60µg dye concentrations against the Gram-positive bacteria exhibited antimicrobial activity against *S. aureus* (0.50 to1.60 mm), which was found to be good activity against the Gram-positive organism *E. coli* (0.67 to 3.03 mm). From the above bar graph results, it is evident that dye powder of the bark of *Tamarindus indica* when subjected to antimicrobial activity by disc diffusion method showed average resistance to bacteria like *E. coli, S. Aureus* (Graph 4.1-4.2).

4.3 Effect of extraction conditions

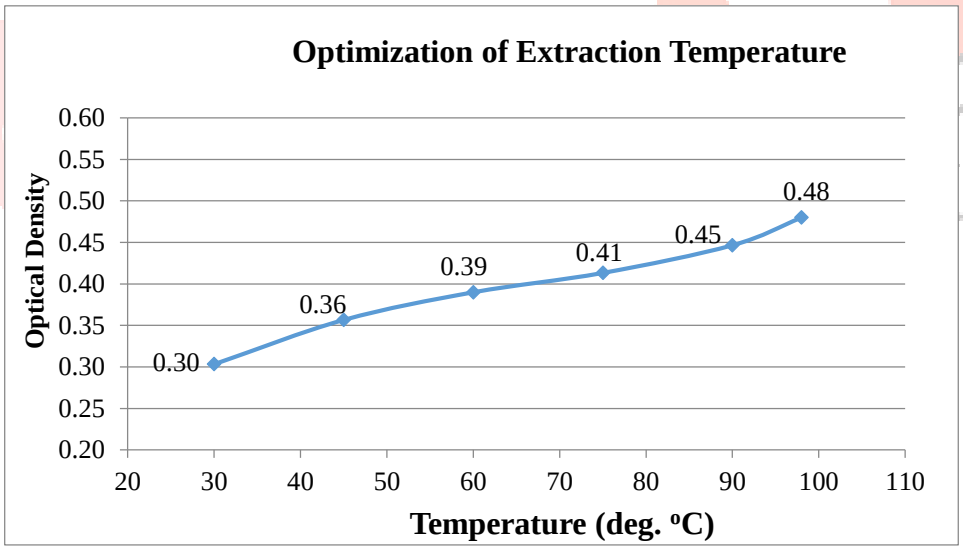
Optimum conditions for dye extraction obtained at pH 9, temperature 100°C and duration 90 minutes. In each case the optical density or absorbance value at a particular (maximum) absorbance wavelength for the aqueous extract of the natural dye material was estimated using colorimeter. Dyes extracted at optimum conditions were filtered and evaporated using oven at 40°C and obtained dye powder was used for dyeing experiments (Graph 4.3-4.5).

Optimization of pH



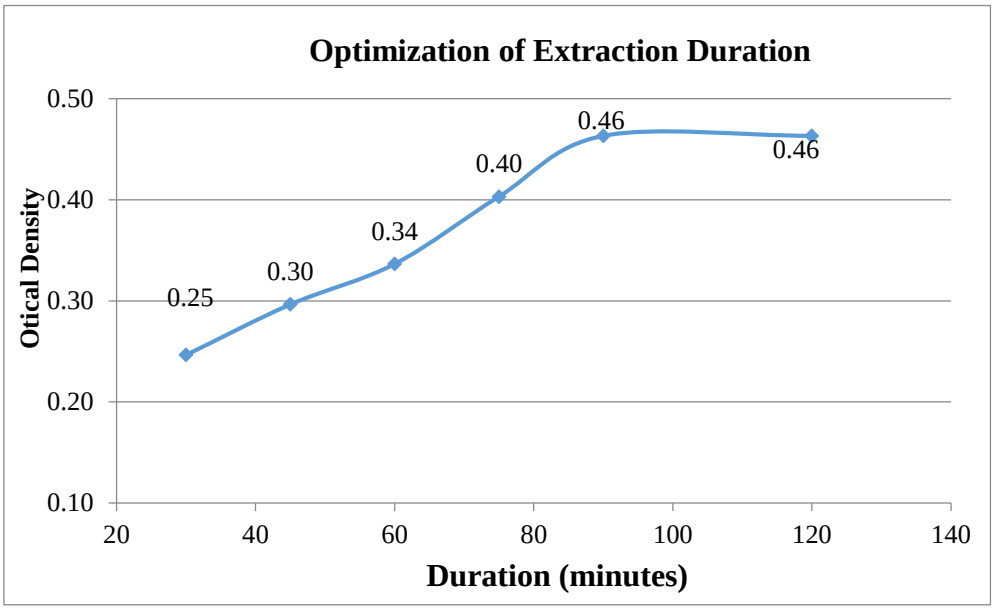
Graph 4.3- Effect of pH on dye extraction

Optimization of Temperature



Graph 4.4 – Effect of temperature on dye extraction

Optimization of Duration

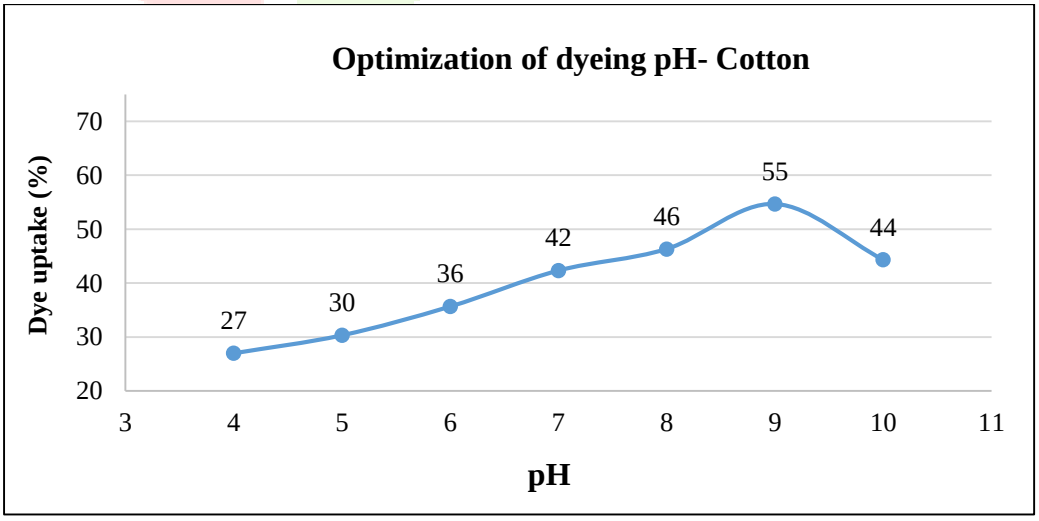


Graph 4.5– Effect of duration on dye extraction

4.4 Effect of Dyeing conditions

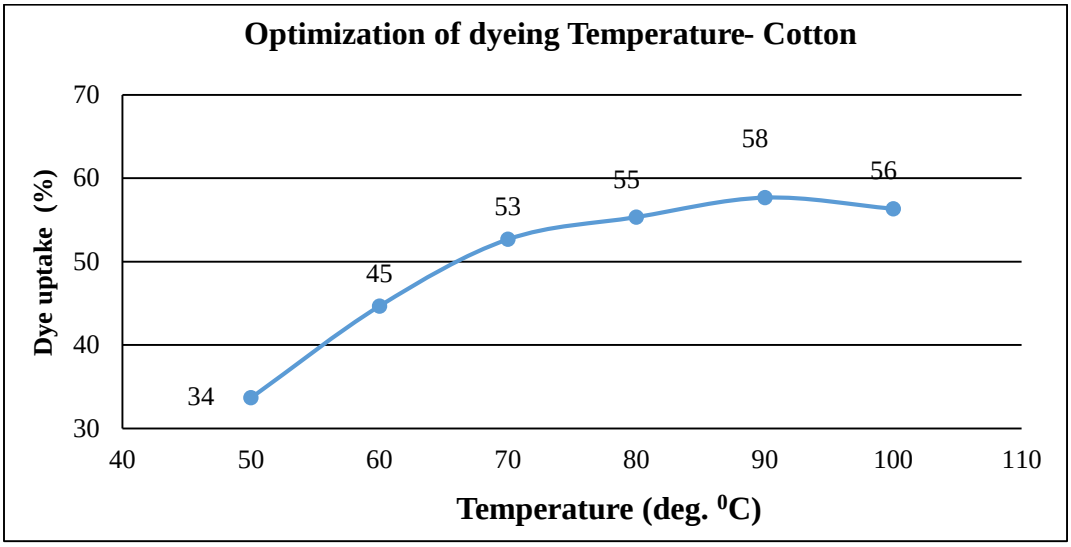
Dyeing conditions for cotton fabric were obtained were pH 9, temperature 90°C, duration 60 minutes and dye 15% concentration. The pH values of the dye bath have a considerable effect on the dye-ability of cotton fabric. As the pH increased the dye-ability increases for cotton, longer dyeing time and temperature means higher colour strength is achieved but there is no significant increase after further increase in the dyeing time and temperature. With regard to the mordants the results show a better performance in dye uptake in respect of bio mordant, post mordanting as led to a significant improvement in dye uptake in all the cases (Graph 4.6-4.9).

Optimization of pH on dyeing



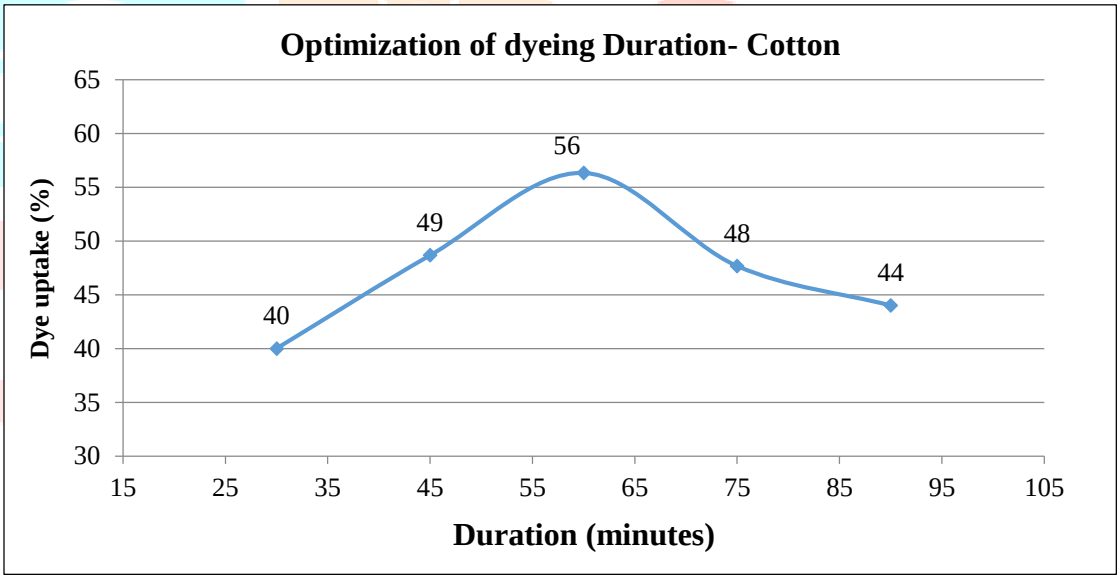
Graph 4.6 – Effect of pH on dyeing

Optimization of Extraction Temperature



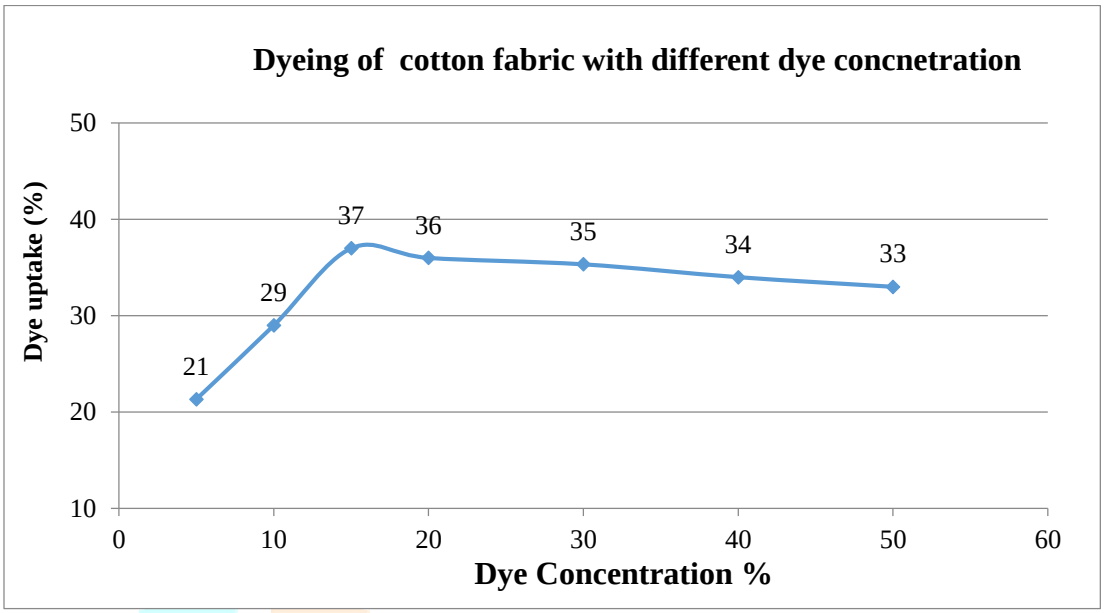
Graph 4.7 – Effect of Temperature on dyeing

Optimization of Extraction Duration

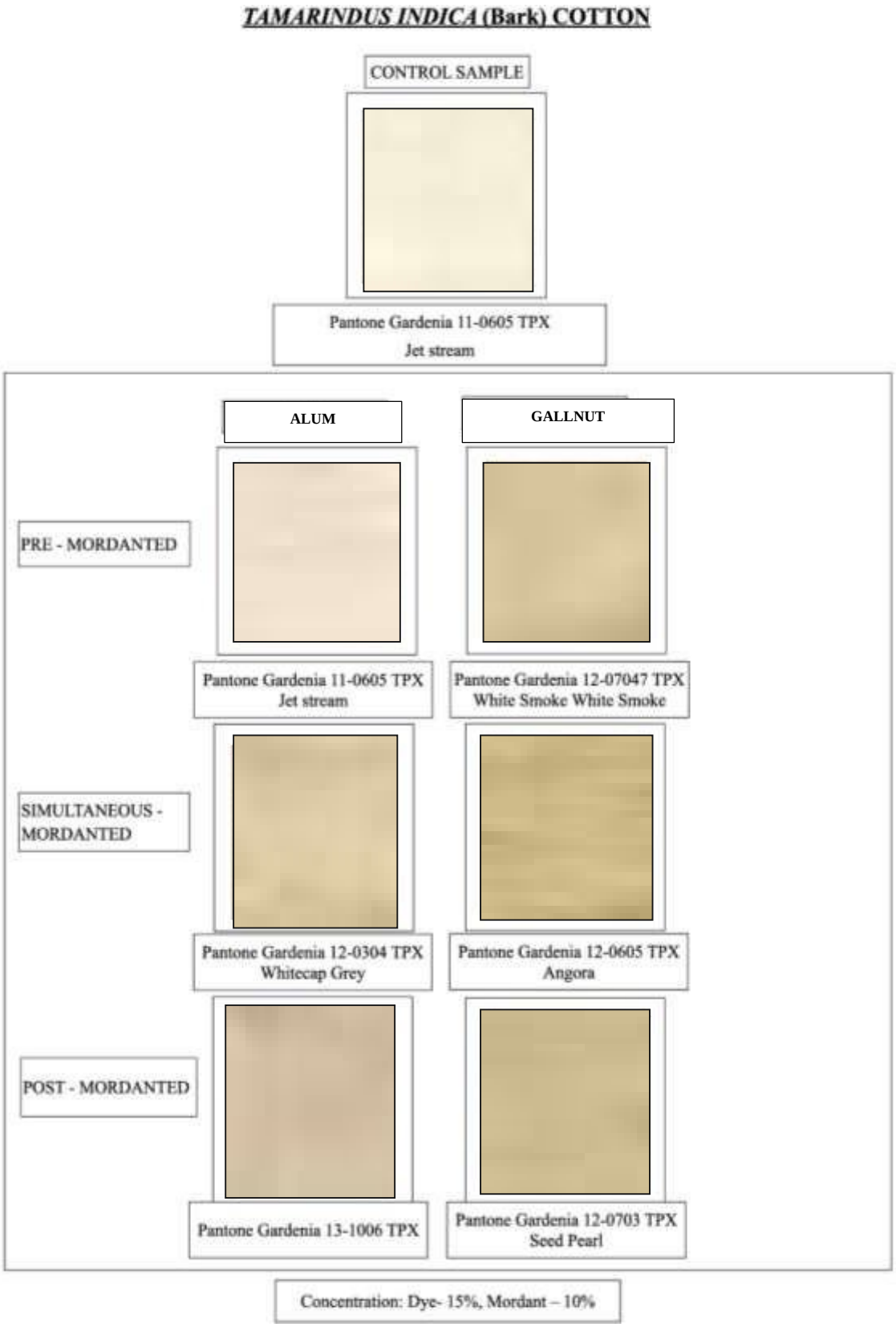


Graph 4.8– Effect of Duration on dyeing

Optimization of Extraction Dye Concentration



Graph 4.9– Effect of dyeing on cotton with different dye concentration



4.5 K/S and colour co-ordinates of cotton samples dyed with *Tamarindus Indica L* dye

From the below table results show that the K/S and colour co-ordinates of cotton samples dyed with *Tamarindus Indica L* dye bark extract. The K/S values of the mordanted samples were found to be lesser than the unmordanted sample. The K/S values improved considerably with control to Gallnut dyed samples when compared to alum.

Table 1

K/S and colour co-ordinates of cotton samples dyed with *Tamarindus Indica L* dye

	Mordants	Mordanting method	K/S	L	a*	b*	C	h
1.	Control		1.10	84.93	2.48	9.09	9.42	74.75
2.	Alum	Pre	1.21	82.19	3.38	11.17	11.67	73.16
3.		Meta	1.01	87.49	1.26	7.31	7.42	80.22
4.		Post	1.60	80.12	2.91	12.46	12.90	71.85
5.	Gallnut	Pre	1.87	77.27	2.97	13.56	13.08	76.88
6.		Meta	1.49	79.30	2.31	14.99	15.18	81.19
7.		Post	2.91	67.75	3.51	19.14	19.46	79.60

The maximum reflectance value in all the cases were found to occur at 360nm. In case of cotton dyed *Tamarindus Indica L it* is noticed that the 'L' values higher in respect of Gallnut, a bio mordant in comparison to Alum, a metallic mordant. The same comments hold well for a, b, c & h values with few exceptions, Post- mordanting has led to higher values of a, b, c & h values. In case of cotton fabrics Gallnut mordant-a bio mordant has led to a significant reduction in 'L' values, which is an indication of higher dye uptake. The unmordanted cotton sample found to have a K/S value of 1.10 and other mordanted samples were observed in the range of 1.01 to 2.91.

4.6 Fastness properties

Table 2
Persipiration Fastness properties cotton samples dyed with *Tamarindus Indica* (bark) extract

Sl. No.	Mordants	Mordanting methods	Acid			Alkaline		
			CC	CS	SW/SILK	CC	CS	SW/SILK
1	Cotton Control		4	5	4/5	4	4-5	4
2	Alum	Pre	3/4	5	4/5	4	5	4/5
3		Meta	4	5	4/5	4	5	4/5
4		Post	4	5	4/5	4	5	4/5
5	Gallnut	Pre	3	5	4/5	4-5	5	4/5
6		Meta	4	5	4/5	4	5	4/5
7		Post	3	5	4/5	4	5	4/5

Table 3
Fastness properties cotton samples dyed with *Tamarindus Indica* (bark) extract.

Sl no.	Mordants	Mordanting method	Light fastness	Wash fastness			Rubbing fastness	
				CC	SC	SW/SILK	Dry	Wet
1.	Cotton Control		2	3/4	5	4/5	3/4	4
2.	Alum	Pre	2/3	3/4	5	4/5	4/5	4
3.		Meta	2/3	3/4	5	4/5	4/5	4

4.		Post	2/3	4	5	4/5	4/5	4
5.	Gallnut	Pre	2/3	3	5	4/5	4	4
6.		Meta	2/3	4/5	5	4/5	4/5	4
7.		Post	3	3/4	5	4/5	4/5	4

As far as perspiration, rubbing and wash fastness of the dyed samples using the natural dyes are concerned; with the exception of the light fastness regardless of the type of mordant & mordanting techniques used the fastness properties were found to be good when compared to light fastness.

Table 4 – Effect of Antimicrobial activity of dyed cotton samples- *Tamarindusindica L* bark extract

COTTON Antimicrobial activity (zone of inhibition in mm)					
				MEAN (D) (5 washes)	MEAN (SD) 10 washes)
	B1	UNMORDANTED		0.96 (0.02)	0.62 (0.05)
		M1	T2	0.73 (0.05)	0.48 (0.04)
			T3	0.53 (0.05)	0.35 (0.03)
			T4	0.83 (0.05)	0.54 (0.01)
		M2	T2	0.63 (0.06)	0.41 (0.04)
			T3	0.60 (0.05)	0.40 (0.04)
			T4	1.10 (0.01)	0.72 (0.07)
	B2	UNMORDANTED		0.92 (0.10)	0.60 (0.06)
		M1	T2	0.54 (0.05)	0.35 (0.03)
			T3	0.50 (0.05)	0.33 (0.03)
			T4	0.60 (0.06)	0.39 (0.04)
		M2	T2	0.67 (0.06)	0.43 (0.04)
			T3	0.57 (0.06)	0.37 (0.04)
D4			T4	0.90 (0.10)	0.59 (0.03)

Note: Values are mean± and SD of three trial measurements. D1- *Psoralea corylifolia L*, B1-*Escherichia coli*, B2-*Staphylococcus aureus*. M1-alum, M2-myrobalan, T2-pre-mordanting, T3-meta-mordanting, T4- post- mordanting, 0 = No zone of inhibition.

Dyed samples for 5 washes showed a slightly better zone of inhibition and lesser for 10 washes, for dyed unmordanted cotton samples and alum, myrobalan mordanted and dyed samples. Among mordants myrobalan, dyed samples showed significant results when compared to alum mordanted and unmordanted samples. In the case of bacteria, *E. coli* (0.35-0.72) had better antimicrobial activity compared to *S. aureus* (0.33-0.60) bacteria. M1-alum, M2-myrobalan, T2-pre-mordanting, T3-meta-mordanting, T4- post- mordanting, 0 = No zone of inhibition.

5 CONCLUSION

Tamarind tree is extensively cultivated and chiefly in the coastal regions of India. It is also useful for medicinal purposes. Bark shredded off is a plant waste can be exploited as a good source of natural dye for cotton. The bark can be used for dyeing silk to obtain range of brown to dark brown with regards colour fastness. Mordant treatment not only improved the colour strength and fastness properties of this natural dye but also resulted in many shades. Dyed sample were shown good to excellent fastness to washing and rubbing where as good to moderate to perspiration in both acidic and alkali media and poor to fair for light fastness, this can be improved by using suitable mordants. Apart from dyeing yielding property, these four selected plant materials possess antimicrobial properties which serve a dual purpose.

As the use of natural dyes does not cause pollution it is of the immense importance to explore the additional sources of natural dyes from the bark which is the byproduct of the abundantly occurring tamarind tree. Since the trees grown in abundance, it cheaper source of raw material so potentially it's a good source of natural colourant for cotton. The use of this natural dye can therefore be a potential substitute for the synthetic dye

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