

# **Ecofriendly Dyeing of Cotton Fabric after Biopolymer Treatment by Using Leaves of *Psidium guajava***

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**ABSTRACT:** India has a very rich tradition of diversity and plant kingdom is certainly a treasure of house of diverse natural products. One such product from nature is dye. In this study, natural colourants were extracted using different solvents. The dye potential of the colourants obtained from the guava leaves was evaluated by colouring cotton fabric. Bio mordanting of cotton fabric was done by using different biopolymers and mordant before dyeing to improve the dyeability of cotton fabric with natural dye. L\*, a\*, b\*, C\* and H\* values and k/s value was studied. Study about the different fastness tests were undertaken by using standard test methods. The chitosan mordanted cotton fabric showed higher k/s value. Good light fastness, wash fastness, rubbing fastness and satisfactory perspiration fastness was found. From an ecological point of view, dyeing of cotton fabric with natural dye with bio mordanting may be a better alternative to conventional mordanting with chemicals.

**KEYWORDS:** Cotton, Bio-Mordanting, Dyeing, Biopolymer.

## **I. INTRODUCTION**

Textiles colored with natural dyes are preferred by environmentally conscious consumers and today there is a niche market for such textiles. But the total share of natural dyes in the textile sector is approximately only 1 % due to certain technical and sustainability issues involved in the production and application. Natural dyes are considered eco-friendly as these are renewable and biodegradable; are skin friendly and may also provide health benefits to the wearer. Natural dyes can be used for dyeing almost all types of natural fibers. Recent research shows that they can also be used to dye some synthetic fibers. Currently, scientific advancement is used for the development of innovative textile products to produce functional textiles inclusive of fragrances, dyes, antimicrobial agents, and wrinkle free. The consumer is increasing in awareness and concern for the safety of products [1]. Thus, there is a need to develop textiles that can serve various purposes and the finish can be applied at the same time so that the use of resources can be minimized. Now a day's such type of natural dyes are used which provide antibacterial property to the fabrics besides dyeing and serves the dual purpose. Natural dyes are soft in colour shades compared to synthetic dyes. Besides, with the increase of the worldwide concern for the environment, the possibility of using natural dyes is being studied because natural dyes may overcome the defects of synthetic dyes such as harmfulness to human body, pollution and waste water. Accordingly there have been active researches for the natural dyeing Samanta and Gorkem et al. [2-3]. Natural dyes demonstrate superior biodegradability and are highly environmentally compatible and hence, they attract the awareness of people around the globe, it is stated by Teli *et al.* [4.] Various wet processing operations of textiles from initial preparatory process to final finished clothes are now focused for green technology. Several conventional non-ecofriendly chemicals are being replaced by natural based products that are safe to environment and health during manufacturing and usage. Applications of enzymes in preparatory, natural dyes for colouration, biopolymers and their derivatives in fiber production and finishing processes, etc. are some of them. One such biopolymer of great interest in recent years is chitosan. Several bio-mordants have been studied by researches, such as chitosan. This is a deacetylated derivative of chitin, a natural polymer found in the shell of crabs and shrimps. Structurally, chitosan contains two main functional groups, namely hydroxyl and amino groups, as well as ether linkages. It has also been used to treat cotton in dyeing

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processes. It was found that chitosan increase dye sorption on cotton. Sundrarajan et al. [5] found that the dyeing of an cotton with natural dye using biocompatible and biodegradable modification agents such as chitosan and cyclodextrin will be the cost effective environmental friendly approach in the field of dyeing industry. Mahbulul et al. [6] investigated that the introduction of cationic sites within the cellulose is the most expected technique to increase the dye adsorption. Cationic sites can be introduced either by aminization or cationization. Treatment of cotton with chitosan is an aminization technique to introduce cationic site within the fiber polymer structure.

*Psidium guajava* Linn. (family Myrtaceae) is commonly called guava [7]. Guava has been shown to have several biological activities such as antidiabetic, anticough, antioxidant, antibacterial and antispasmodic properties. It is stated by Nantitanon et al. [8]. Most of the pharmacological and chemical work has been carried out on the leaf, since the leaf of the guava is rich in flavonoids and phenols including terpenoids, tannins, essential oils, chlorophyll and saponins by Venkatachalam et al. [9]. Guava leaves have long been recognized for their antimicrobial activity. Its antimicrobial activity was beneficial when applied as an antimicrobial agent in textiles. In the present study the guava leaves are used as dye to coloration of cotton textiles.

## II. RELATED WORK

The study was done in the following sequence to attain the results.

### Material:

**Fabric:** Pure cotton fabric was purchased from local market of hisar city of Haryana. The fabric used was of plain woven cotton construction with 43 ends and 40 picks per centimeter square and the weight per unit area of 140 g/m<sup>2</sup>.

**Chemicals and colorants:** For increasing the absorbency of the cotton fabric desizing and scouring treatment was given to the cotton fabric by using enzyme, viz americos amylase 543 and palkoscour APCL (pectinase and mixture of enzymes) respectively. After that bio mordanting of cotton fabric was done by using chitosan which is purchased from Indian sea Food Company Cochin, Kerala. The guava leaves were collected from the university campus for dyeing of cotton fabric.

### Methods:

- (a) **Desizing of the Cotton Fabric:** The pure cotton fabric was given desizing treatment by using 2ml/L Americos Amylase 543 at 60<sup>0</sup>C temperature for 60 minutes with 1:20 material to liquor ration by maintain 7 pH. After the treatment liquor was drained out; given one hot wash and cold wash and dried.
- (b) **Scouring of the cotton fabric:** After application of desizing, desized cotton fabric was scoured in a bath containing 1.5 % owg palkoscour APCL enzyme wetting agent, at 60<sup>0</sup>C for 60 minute at material to liquor ration 1:15 by maintain 7.0 pH . The fabrics were rinsed, with hot and cold water and dried.
- (c) **Bio- mordanting of the scoured fabric:** Chitosan with 85 % degree of deacetylation was used for application of finish. Citric acid used as a crosslinking agent, di-sodium hypophosphite as catalyst was also used along with chitosan. A chitosan solution was prepared by stirring a dispersion of chitosan (4%) in 1% (v/v) aqueous acetic acid solution. The treatment was applied on scoured cotton fabric by using pad dry cure method. After this the treated fabrics were dried at 100 °C and then cured at 120 °C for 3 min by using standard concentrations of chitosan, citric acid and di-sodium hypophosphite.
- (d) **Preparation of the dye powder and dye extract:**

The fresh leaves of *Psidium guajava* were collected from university campus. Then, the leaves were washed to remove debris and the collected leaves were shade dried for 35 days and after being completely dried, the leaves were crushed into small pieces finally pulverized in to coarse powder. It was stored in a well closed container free from environmental climatic changes till usage.

**Aqueous extraction:** Aqueous extract was prepared by adding 5 g of guava leaves powder to 100 ml distilled water. The mixture was heated at 100°C for 1 hour, allowed to stand for overnight and then filtered. The extracts were sieved through fine mesh nylon cloth. The filtrates were concentrated by water evaporation. The filtrate was used for dyeing.

**Methanolic extraction:** 5 g of leaves powder is soaked in 100 ml methanol and kept in water bath for period of 2 hours for complete dye extraction and then it was filtered.

**Ethanol extraction:** leaves powder was soaked with 100% ethanol and heated in a beaker kept over a water bath for 2 hours to facilitate quick extraction. Then it was filtered and the filterate was collected.

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(e) **Phytochemical analysis of the dye extract:**

**Phenol:** Equal volume of each and  $\text{FeCl}_3$  solution (which is prepared by dissolving 135.2g of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  in distilled water containing 20 ml of conc. HCl dilute to 1 litre) are added together. A deep bluish green precipitate indicates the presence of phenol. **Alkaloids:** Extracts of 1% aqueous HCl over water bath and filtered. The filtered was treated with ( 2g of iodine in 6g of KI in 100 ml of distilled water . formation of brown or reddish brown precipitate indicate presence of alkaloids. **Steroids:** for testing the presence of steroids 1 ml extract was dissolved in 10 ml of chloroform and equal volume of concentrated sulphuric acid was added from the walls of the test tube. Appearance of red colour in the upper layer and yellow with green fluorescence indicates the presence of steroids. **Cardiac glycosides:** to 1 ml of extract glacial acetic acid few drops of ferric chloride and then finally conc. Sulphuric acid were added from the walls of the test tube. Appearance of the reddish brown at the junction of two layers and the bluish green colour in the upper layer indicates the presence of cardiac glycosides. **Tannins:** To 5 mL of extract few drops of 5%  $\text{FeCl}_3$  was added. Presence of deep blue black colour indicated the presence of tannins. **Flavonoids:** To 5 mL of extract few drops of NaOH solution was added. Formation of an intense yellow color, which turns to colorless on addition of few drops of dil.  $\text{H}_2\text{SO}_4$  indicated the presence of flavonoids. **Terpenoids:** Extract (5 mL) was treated with 5 mL  $\text{CHCl}_3$  with few drops of conc.  $\text{H}_2\text{SO}_4$ , shake well and allowed to stand for some time. Formation of yellow colored lower layer indicated the presence of terpenoids. **Antraquinone:** 10 ml benzene was added to each extract and filtered. 0.5 ml of 1% ammonium solution was added and shaken. Pink red or violet colour in the ammoniacal lower phase indicates the presence of antraquinone. **Saponins:** Saponins are tested by boiling 5 ml of extract 10 ml of distilled water in a test tube and are shaken vigorously for about 30 seconds. The test tube is allowed to settle for half an hour. Formation of froth indicates the presence of it. **Reducing sugar:** 1 ml of extract was added 5 to 10 drops of Fehling solution. Mixture was then subjected to boiling for 15 minutes. Appearance of brick red precipitate indicates the presence of reducing sugars.

(f) **Application of dye extract on the cotton fabric:** The aqueous extraction of the corresponding dye solution was double filtered in fine mesh nylon cloth and it was used as a dye extract on the cotton fabric. The scoured cotton fabric was dyed with 5% dye shades for dyeing. Dyeing experiments were performed using M: L (material to liquor) ratio of 1:30 with manual agitation. Dye baths temperatures were raised to 60-100°C for 1 h.

(g) **Evaluation of CIE coordinates:** The dyed samples were subjected to colour measurement by using reflection spectrophotometer. The depths of shade were evaluated by K/S value and CIELAB colour differences values of the dyed cotton fabric.

(h) **Quality assurance tests of dyed fabric:** Most dyes are organic compounds and are, therefore, vulnerable in varying degree to the action of destructive agents. A number of tests are necessary to cover all the important properties of any one dye because good fastness to one inference is not necessarily accompanied by equal fastness to other conditions. For characterization and evaluation, following tests were performed with selected dyed fabrics: Washing fastness, Rubbing fastness and Light fastness.

**Washing Fastness:** Dyed sample was placed between two pieces of non dyed white samples (control). These three pieces were held together by stitching round the edges. The pre heated soap solution (Tide, at 60°C) in the ratio of 1:50 i.e 0.5g/25 mL water, was taken in a vessel added 1.0 g of sandwiched fabric for 30 minutes Then the specimen was removed and rinsed in cold water. The colour fastness is usually rated by the presence of the colour in control sample.

**Rubbing fastness:** The rub fastness of the dyed fabrics was carried out by rubbing the fabrics manually and checking for fading of color.

**Light Fastness:** The fabric was exposed to sun light for 24 h. The colour fastness to light was evaluated by comparison of colour change of the exposed portion to the unexposed original material.

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## III. EXPERIMENTAL RESULTS

**Preliminary Phytochemical Screening:** The preliminary phytochemical tests were performed for testing different chemical groups present in extracts.

**Table 1: Phytochemical Screening of Various Extracts of *Psidium guajava* Linn**

Chemical Test	Aqueous Extract	Ethanol Extract	Methanol Extract
Phenol	√	√	√
Alkaloids	X	√	X
Steroids	√	√	√
Cardiacglycosids	X	X	X
Terpenoids	√	√	√
Tannins	√	√	√
Flavonoids	√	X	√
Anthraquinone	X	X	X
Saponins	X	X	X
Reducing Sugar	X	X	X

Plants may contain significant phytochemicals that can be analyzed by several biochemical and analytical test from solvent and extract of each plant material and also the powdered form of plants as well as powdered extract of samples. In many cases, these substances serve as plant defense mechanisms against predation by microorganisms, insects and herbivores. Some compounds such as terpenoids give plants their odors; others (quinones and tannins) are responsible for plant pigment. The above table shows the presence of different phytochemicals in different solvent. Presence of phenol, steroids, terpenoids and tannins was found in each solvent extract. Tannins rich extract have good resistance against gram positive and gram negative bacteria [10]. It means that there is a presence of tannins in extract; it will also have good antibacterial property.

**Table 2: k/s values of fabric pretreated with different biopolymer and mordant**

S.No.	Natural dye	Methods	L*	a*	b*	C*	H*	k/s value	Rank
1.	Guava leaves	Mordant treated (Alum)	73.18	1.98	14.25	14.39	82.03	8.10	II
		Chitosan treated	73.16	2.25	14.42	14.59	81.07	<b>12.86</b>	I
		Sericin treated	74.67	0.59	18.12	18.13	88.08	7.00	IV
		β-cyclodextrin treated	73.19	2.80	14.94	15.20	79.34	7.77	III

The depth of colour of dyed fabric was analyzed by k/s value. This value numerically represents the nature of the colouring material layer and an easy way to determine a colour as a concentration. The colour concentration decreases as the value for reflectance increase, and vice versa.

The depth of shade i.e. k/s value found for all the dyed fabric samples are shown in the table 2. The above table shows that the dye absorption, i.e. the k/s value is highest for the chitosan pre-treated fabric. It is followed by the mordant treated fabric. There was very small difference in the k/s value of sericin and beta-cyclodextrin treated fabric. The increased k/s value in the chitosan pre-treated fabric indicates that the presence of higher amount of dye was absorbed in the chitosan treated fabric. This indicates that the chitosan is capable of enhancing natural dye (guava leaves) fixation on cotton fabric. The results indicated that chitosan treatment on fabric provided more dye sites than the other pre-treated fabric. These can be explained that the natural dyes contain unsaturated moiety bearing ionizable groups such hydroxylic and carboxylic groups. In water with right ph value, they become water soluble due to their presence in anionic forms. Cotton by its nature is negatively charged in water, thus exhibiting poor absorption for natural dye due to repulsive effect. The application of chitosan could help to improve the absorption of natural dye due to its cationic characteristics. It is well known that the chitosan is capable of forming ionic interaction with the cotton cellulose,

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rendering cotton cellulose positive charged. As a result, chitosan treated cotton is anticipated to favorably absorb natural dye through the ionic interaction between dye-anions and fibre -cations mechanism. This result is in line with findings of Suitcharit et al. [11] who reported that the presence of the chitosan on cotton fabric was found to enhance the dyeability of fabric.

**Table: 3 Fastness properties of guava leaves extract dyed fabric after chitosan pretreatment**

Dye	Wash Fastness		Light Fastness Days							Perspiration Fastness				Rubbing Fastness			
										Alkaline		Acidic		Dry		Wet	
	CC	CS	1 <sup>st</sup>	2 <sup>nd</sup>	3 <sup>rd</sup>	4 <sup>th</sup>	5 <sup>th</sup>	6 <sup>th</sup>	7 <sup>th</sup>	CC	CS	CC	CS	CC	CS	CC	CS
<b>Natural dye (<i>Psidium guajava</i> leaves)</b>	4	4	4	4	4	4	4	4	4	3/4	3/4	3/4	3/4	4	4	4	4

Washing fastness is usually poor for the natural dye. The results of wash fastness, light fastness, perspiration fastness and rubbing fastness of the chitosan pre-treated dyed fabric are shown in the table 3. The fabrics were compared with the grey scale to obtain the colour change compared with the fabric before testing. Grey scale has scale from 1 to 5 that scale 1 indicates the most colour difference and scale 5 means no colour difference. The chitosan treated dyed fabric showed good wash fastness, rubbing fastness and light fastness. Perspiration fastness was satisfactory. Dye molecules are strongly hydrogen bonded with free amino groups of chitosan and strongly retained on the fabric. Saravanan et al. [12] concluded that the cotton fabrics treated with chitosan not only provided better depth of shade but also provided better fastness and light fastness than those of untreated fabrics.

### IV. CONCLUSION

Dyeability of cotton fabrics can be improved in better way using chemicals, but the pollution load increases due to non-biodegradability and toxic nature of chemicals. Hence in this study biopolymers (chitosan, Sericin) and beta-cyclodextrin is used to improve the dyeability of cotton fabric because of their non toxic, biodegradable and biocompatible nature therefore environment is protected. The purpose of this work was to study the effect of different pre-treatment on the dyeability on the cotton fabric with natural dye (guava leaves). The results of this study concluded that the chitosan showed higher k/s value compared to the other pretreatments. It also reduces effluent load by improving the dyeability of cotton fabric. Therefore dyeing of cotton fabric with natural dye using an ecofriendly, biocompatible and biodegradable agent chitosan will be the cost effective environmental friendly approach in the field of dyeing industry.

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