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Non-toxic Coloration of Cotton Fabric using Non-toxic Colorant and Non-toxic Crosslinker

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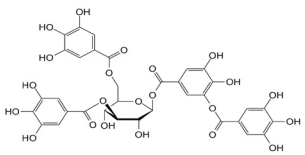
Abstract

A toxicity free cotton fabric coloration and antibacterial technique was developed where natural colorant curcumin and tannin were applied for instigating a substitute of toxic colorant. Considering the negative impacts of man-made dyes in environment, non-toxic coloring and sterile finishing was carried out with curcumin and tannin as non-toxic colorant quintessence from pure natural sources of *Curcuma longa* (Turmeric) and *Camellia sinensis* (Tea Leaf) using environment friendly lemon extracted (Natural Citric acid) solution as natural mordant and their color shading and functional outcomes were evaluated in variety of shades as compared with colour yield/outcomes with ferrous sulphate and copper sulphate as two most extensively practiced conventional chemical mordants. Critical performances of mordanted and unmordanted cotton fabric samples were tested for their color parameters like Colour Strength, K/S value and Colour fastness (color fastness to Washing, UV- Light and Rubbing) properties, besides measurement of other colour interaction parameters (L^*, a^*, b^* and ΔE) as well as anti-bacterial performance as Per AATCC-100-2012 method. The dye absorption determined by color spectrophotometer showed higher color strength when treated with curcumin and tannin colorant in the presence of natural extracted citric acid from lemon as toxic free mordanting agent compared to mordanting with Ferrous sulphate as well as conventional mordanting with Copper Sulphate. Higher rubbing fastness was observed for both dyeing with Turmeric and Tea Leaves when mordanting with lemon and ferrous sulphate. Curcumin demonstrates a pharmacological properties that curcumin found effective against *E. coli* bacteria in laboratory experiment well as FTIR study of Turmeric with lemon mordanted sample was confirmed the natural crosslinking among cotton fibre cellulose, turmeric curcumin and lemon-citric acid. Consequently, this work has led to development of a process of concurrent natural dyeing and finishing with natural extracted agents on cotton with natural mordants to make the fabric colour full and anti-bacterial also. If the present work is trailed commercially to make it feasible, undoubtedly present finding of research may be an exceptional source of eco-garments production which has a higher demand in local and foreign customer.

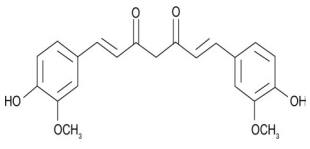
Keywords: Natural dyeing and finishing; Mordanting; *Curcuma longa* (Tumeric); *Camellia sinensis* (Tea Leaf)

Introduction

Environmental scientists and textile engineers concerns have been a great demand to have a pure concentration in green chemistry as the present panorama of environment is being a great threatening for the synthetic based dyeing and finishing and its negative impacts have been a great challenge both for the producers and consumers of textile sector as well as finding an alternative way has become a novel foundation of research for the scientist corner in home and abroad as everyone of human being want to get pleasure from his life. Present research works on toxic-free dyeing and finishing may impact directly to fulfil the demand of green chemistry as on-toxic dyes and mordants have been established to make coloration of cotton fabric because of the environment-friendly dyes and mordants can be reproduced again and again without hampering the environment which may vibrate the green eco-system of nature.



Chemical Structure of Tannin



Chemical Structure of Curcumin

Natural dyes like *Acacia, Catechu, Kerria lacca, Quercus infectoria, Rubia cordifolia and Rumex maritimus* were tested again common pathogens *Escherichia coli, Bacillus subtilis, Klebsiella pneumoniae, Proteus vulgaris and Pseudomonas aeruginosa, Quercus infectoria* dye was effective and showed maximum zone of inhibition thereby indicating best antimicrobial activities against all the microbes tested [1]. Experimental trailing was executed by a variety of the most commonly used mordants, namely potassium aluminium sulfate, copper(II) sulfate, iron(II) sulfate, and tin(II) chloride, were used for mordanting of cotton fabrics in order to compare the differently mordanted and unmordanted dyed fabrics' color efficiencies (K/S) and CIE L $^*a^*$ b^* color values. It was found that mordant type had an effect on color efficiency and the color coordinates of fabrics dyed with both thyme and pomegranate fruit peel. Moreover, the antimicrobial properties of the fab-rics only dyed directly with thyme and pomegranate peel without any mordanting process were determined to demonstrate the usability of these natural dye sources without use of any mordanting agents. The obtained antimicrobial activities were compared with undyed samples. Undyed samples showed no antimicrobial activity,

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whereas significant antimicrobial activity was obtained after the dyeing procedure using thyme and pomegranate peel on unmordanted fabrics. Washing, rubbing, perspiration, and light fastness properties of dyed fabrics were also evaluated. Thyme and pomegranate fruit peel as natural dye sources revealed sufficient results even for un-mordanted samples [2]. A special method named Sonication in conjunction with metal mordanting has shown marked improvement in terms of dye adherence and fastness properties and can thus be recommended for industrial application [3]. Natural dyeing on textile substance using Annatto seed extract and the antimicrobial property of the ex-tracted dye samples is studied by using Gram positive and Gram negative culture and provided constructive results [4]. Natural dyeing from the Acacia family, in the presence of a controlled amount of chitosan resulted in darker shades and imparted chitosan imparted excellent antibacterial properties to the cotton fabric [5]. Natural coloration of textiles from Acacia plant family with different mordanting showed acceptable fastness in shades [6].

Natural dyes selected from *Indigofera tinctoria, Mallotus phillipinesis, Curcuma tinctoria and Lawsonia inermis* and assessed by measuring the colour strength (K/S) values of dyed samples using UV spectrophotometer where various fastness properties (wash, light and rub) of the dyed samples have been evaluated [7]. A natural colourant of Marigold flower *(Tagetes erecta)* was applied on 100% cotton utilized as mordants, cross linkers, finishing agents and experimentation had better colour fastness and enviable K/S value of cotton [8]. Natural dyeing with Chitosan pretreatment was effective to eliminate the difference in dye uptake between soybean and cotton fibers and antimicrobial activity was imparted [9].

Curcuma dye was investigated in the chitosan microspheres released from the chitosan through an adsorption process [10]. Natural dyeing with tamarind leaves as natural mordant was also investigated experimentally [11]. Natural dyeing with Kamala natural and found color depth with good fastness properties [12]. Natural dyeing of cationized cotton fabrics with the extractable solution of chicken feather using colouring matter extracted from acacia bark showed good color fastness [13]. Natural dyeing with extracted pea-nut pod powder with mordanting agents (Alum, Copper Sulphate and Ferrous Sulphate) and fast-ness properties were observed [14]. Natural dyes can be extracted from plant -leaves, roots and barks, insect secretions, and minerals which may create very minor side effect to environment [15]. Fresh Aloe Vera (*Aloe barbadensis*) could be used on cotton with the help of different mordants to produce different colours varying from yellow, pink, khaki to brown [16]. Natural biocides can be applied on textiles as natural antimicrobial agents [17]. Natural dyeing with roots of Ratanjot (*Onosma echioides*) was applied on textile substance with different mordanting and found good fast-ness properties [18].

The timber industry releases considerable amount of wastes which contain natural dyes. Such wastes could serve as sources for the extraction of natural dyes for textile dyeing operations [19]. Natural dyeing with Callistemon citrinus plant applied on cotton textiles and found good light fastness, rub fastness and wash fastness in fabrics mordanted with ferrous sulphate [20]. In natural coloration of nontoxicity, Tannins are used in the dyestuff industry as cationic dyes (tannin dyes) [21]. All dyed fabrics acquired admirable rubbing as well as washing fastness, and the relevant dyeing mechanism has been analysed [22]. Natural dyes extracted from different types of microorganisms as well as various parts of the plants that contain coloring materials such as tannin, flavonoids and quinonoids [23]. Ethanalic dye extracts of Acacia eburnean (L.f.) Willd was dyed to the bleached cotton fabrics were dyed with different chemical mordants using pre-mordanting, post-mordanting and simultaneous-mordanting method. In this method post mordanting of ferrous sulphate compound gave good K/S -value than others. The dyed fabrics showed good washing, light, rubbing fastness and perspiration fastness proper-ties. Antimicrobial activities of the dye extract were also studied [24].

Materials Used

Following material mentioned in below table is used during experiment (Table 1).

Experimental Works

Colorant preparation of curcumin and tannin

Green Turmeric was cut into very small pieces by knife and colorant extracted with automatic grinding, boiling and filtration method and pre-pared 2.5% stock solution of turmeric for natural coloration on cotton fabric. Similarly Green Tea leaves was extracted with grinding, boiling and filtration to prepare 2.5% stock solution of Tannin colorant although several stock solution was trailed to find an optimum percentage of colorant solution.

Stock solution preparation of different mordants

Fresh Green lemon was cut and squeezed by manual pressure machine and after filtration got a clear ready solution for dye bath application as natural mordanting agent. 5% $FeSO_4$ and $CuSO_4$ solution was prepared with heating and boiling process with 700°C for 30 minutes where distilled water was used.

Dyeing with curcumin extracted from green turmeric

5 gm cotton fabric was dyed with cur-cumin solution extracted from Turmeric at 70 to 75°C temperature for 30 minutes by keeping a material and liquor ratio: 1:5 under open bath stirring condition while several ratio was also trailed to observe the penetration of color.

Dyeing with turmeric solution and different mordants

5 gm cotton fabric was dyed with cur-cumin solution extracted from Turmeric and (added 10% of total solution in dye bath) lemon, $FeSO_4$ and $CuSO_4$ solution at 70 to 75°C temperature for 30 minutes by keeping a material and liquor ratio: 1:5 under open bath stirring condition while several ratio was also experimented to observe the penetration of color.

Dyeing with tannin extracted from tea leaves

5 gm cotton fabric was dyed with Tannin solution extracted from Green Tea Leaves at 70 to 75°C temperature for 30 minutes by keeping a material and liquor ratio: 1:5 under open bath stirring condition while

Materials	Sources
Cotton Fabric: GSM 150 (Scoured & Bleached)	From Local Markert, India and also from Alim Knit Bd Ltd., Kashimpur, Gagipur, Bangladesh
FeSO4, CuSO4 (For Comparison) Distilled water	E Marc –India and also from Mother trade International Mirpur, Dhaka, Bangladesh
Green Lemon	Vegetable Garden, Ashulia, Savar, Dhaka, Bangladesh.
Green Tumeric	Vegetable Garden, At IIT Kanpur and also from Ashulia, Savar, Dhaka, Bangladesh.
Green Tea Leaf	Tree Garden at IIT-Kanpur and also from Ashulia, Savar, Dhaka, Bangladesh.

Table 1: Material used for trialing works during experimentation.

several ratio was also monitored to maintain the optimum percentage of color penetration.

Dyeing with Tannin solution and different mordants: 5 gm cotton fabric was dyed with Tannin solution extracted from Tea Leaves and (added 10% of total solution in dye bath) lemon, $FeSO_4$ and $CuSO_4$ solution at (70 to 75)°C temperature for 30 minutes by keeping a material and liquor ratio: 1:5 under open bath stirring condition while several ratio was also assessment to observe the color penetration.

Testing Methods

- Color strength (K/S value)

Testing method: The K/S values were calculated by Kubelka Munk equation.

K/S= (1-R)2 /2R

Where, R is the light reflectance of the dyed samples at max. K is the absorption coefficient and S is the scattering coefficient.

Machine type: Macbeth Spectrophotometer

- Color fastness to light

Testing method: AATCC TM16

Machine type: Q-SUN XE-2 Xenon Test chamber against.

- Colour Fastness to washing

Testing method: ISO-I

Machine type: launder-o meter

- Color fastness to Rubbing

Testing method: ISO 105-X12:2001(E)

Testing type: Dry or wet rubbing

- Antimicrobial Testing

Testing method: AATCC-100-2012 method

Results and Discussion

Spectrophotometer analysis of natural colorant curcumin treated fabric showed higher color deposition and distribution on the surface of cotton fabric as curcumin ($C_{21}H_{20}O_6$) colorant structure is responsible to react with cellulosic structured cotton fabric, but k/s value was found double in case of lemon mordanting with curcumin com-paring to $FeSO_4$ and $CuSO_4$ mordanting. Color parameters like Da^*, Db^*, Dc^* are also deeply analysed for curcumin colorant where deeper col-or absorption was found for dyeing with curcumin and lemon mordanting and DE value was also similar comparing with $FeSO_4$ and $CuSO_4$ mordanting.

Spectrophotometer analysis of natural colorant Tannin treated fabric showed higher color deposition and distribution on the surface of cotton fabric as Tannin has OH group is conscientious to react with OH group of cellulosic structured cot-ton fabric, but k/s value was also found better having a very minor difference comparing to $FeSO_4$ and $CuSO_4$ mordanting. Color parameters like Da^*, Db^*, Dc^* were also deeply analysed for Tannin colorant where deeper color absorption was found for dyeing with Tannin and lemon mordanting and DE value was also lower and almost similar comparing with $FeSO_4$ and $CuSO_4$ mordanting.

FTIR study of Turmeric with lemon mordanted sample was confirmed the natural crosslinking among cotton fibre-cellulose, turmeric-curcumin and lemon-citric acid.

Higher rubbing fastness was observed for both dyeing with Turmeric and Tea Leaves when mordanting with lemon and ferrous sulphate. Therefore, the antibacterial properties of Turmeric extracted dyed cotton fabric using lemon mordanted sample resulted better reduction/ killing of bacteria. Consequently, this work has led to development of a process of simultaneous natural dyeing and finishing with natural extracted agents on cotton with natural mordants to make the fabric non-toxic coloration and antiseptic also.

Color parameters for dyeing with turmeric on cotton fabric

Higher K/S value showed for the sample when treated with lemon mordanting and other color parameters to spectrophotometer marked in graph that the mordanted and unmordanted both samples (turmeric and turmeric with lemon) color parameters Da^* Db^* Dc^* and DE always better color exhibited than mordanted samples (turmeric with $CuSO_4$ and $FeSO_4$) (Figures 1-5 and Table 2).

Fastness properties for dyeing with Turmeric on cotton fabric: Dyeing with Turmeric, the color fastness to wash marked in graph that the treated sample with mordanting with lemon and ferrous sulphate showed improved wash and light fast-ness comparing to synthetic and conventional Copper sulphate mordanting as well as very excellent rubbing fastness were showed both mordanting with lemon and ferrous sulphate comparing to copper sulphate (Table 3 and Figures 6-9).

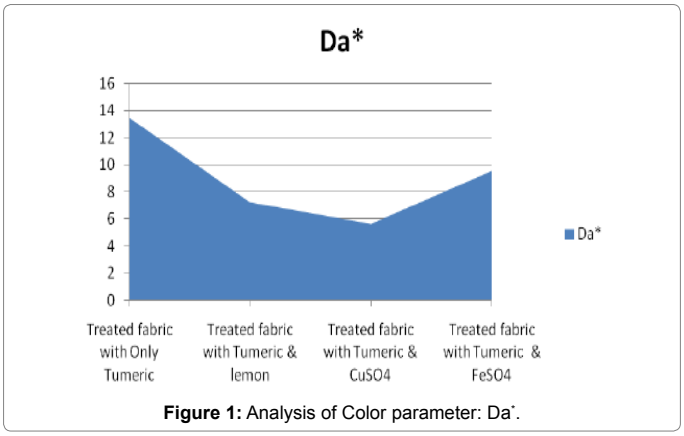

Figure 1: Analysis of Color parameter: Da^*.

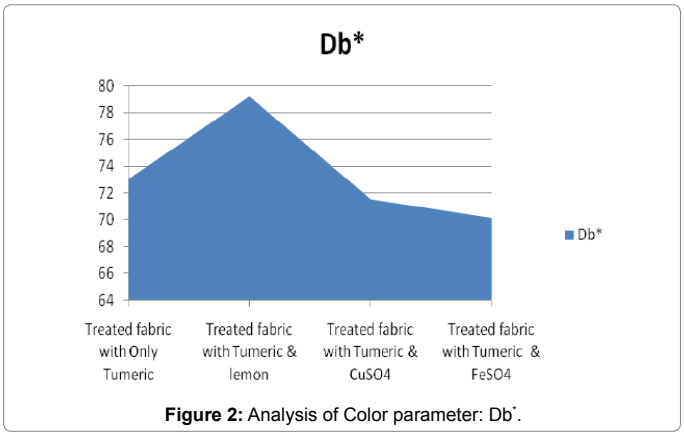

Figure 2: Analysis of Color parameter: Db^*.

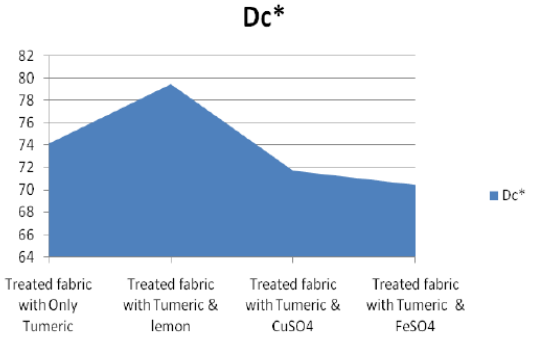

Figure 3: Analysis of Color parameter: Dc*.

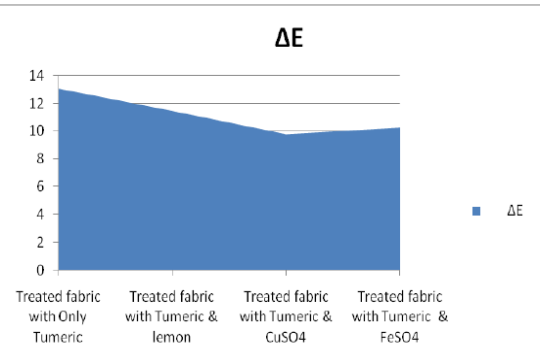

Figure 4: Analysis of Color parameter: DE.

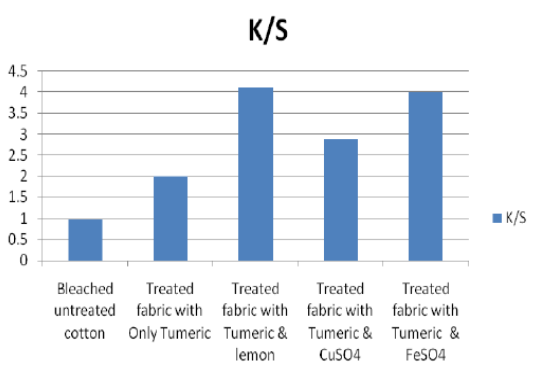

Figure 5: Analysis of color strength, K/S value.

Treatments	Color Parameters				
	Da*	Db*	Dc*	DE	K/S value
Bleached untreated cotton	-	-	-	-	0.98
Treated fabric with Only Turmeric	13.50	73.02	74.12	13.04	2.00
Treated fabric with Turmeric & lemon	7.22	79.20	79.47	11.43	4.10
Treated fabric with Turmeric & $CuSO_4$	5.63	71.53	71.70	9.75	2.90
Treated fabric with Turmeric & $FeSO_4$	9.55	70.11	70.43	10.22	4.00

Table 2: Color parameters for dyeing with turmeric on cotton fabric.

Treatments	Analysis of Color fastness to wash	Light fastness	Color fastness to Rubbing	
			Dry	Wet
Treated fabric with Only Turmeric	2	3	3	2
Treated fabric with Turmeric & lemon	2-3	2	4	2
Treated fabric with Turmeric & $CuSO_4$	3-4	4	4	3
Treated fabric with Tumeric & $FeSO_4$	2-3	3	4	3

Table 3: Color fastness properties for dyeing with tumeric on cotton fabric.

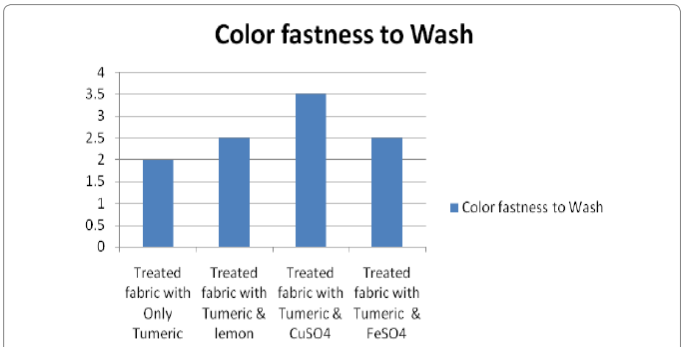

Figure 6: Analysis of color fastness to wash.

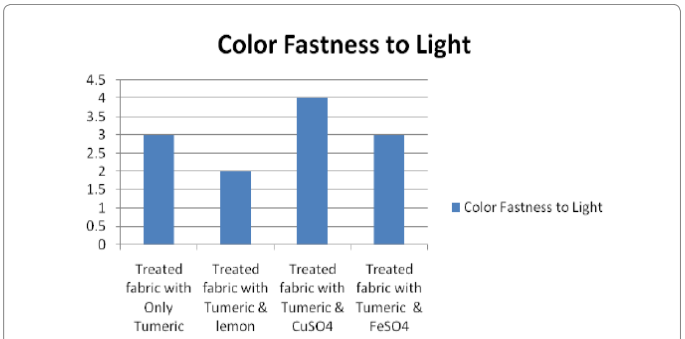

Figure 7: Color fastness to light.

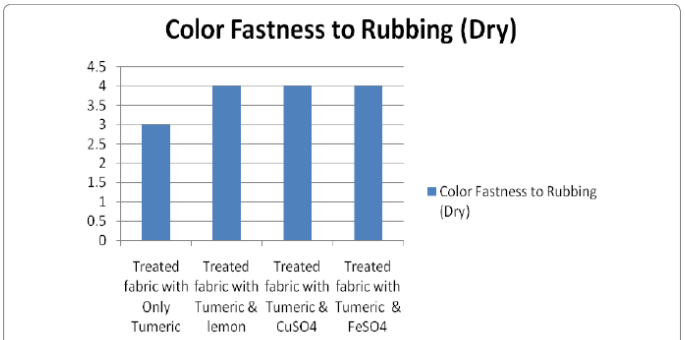

Figure 8: Analysis of Color Fastness to Rubbing (Dry).

Color parameters for dyeing of tea leaves on cotton fabric

Higher K/S value showed for the sample when treated with lemon mordanting and other color parameters to spectrophotometer marked in graph that the mordanted and unmordanted both samples (turmeric and turmeric with lemon) color parameter Da^* Db^* Dc^* and DE^* always better color exhibited than mordanted samples (turmeric with $CuSO_4$ and $FeSO_4$) (Table 4 and Figures 10-14).

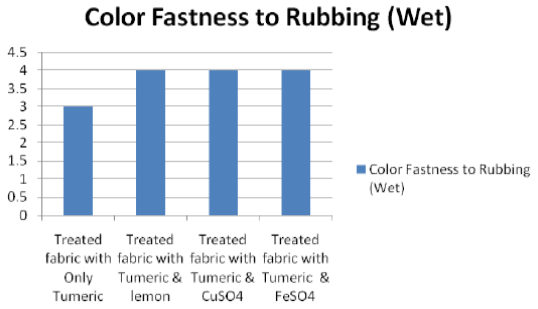

Figure 9: Analysis of Color Fastness to Rubbing (wet).

Treatments	Color Parameters				
	Da*	Db*	Dc*	DE	K/S valve
Bleached untreated cotton	-	-	-	-	0.89
Treated fabric with Only Tea	11.55	23.09	25.29	8.97	1.25
Treated fabric with Tea Leaves & lemon	8.53	22.91	24.11	7.41	3.65
Treated fabric with Tea Leaves & CuSO4	7.57	15.02	16.32	7.18	2.80
Treated fabric with Tea Leaves & FeSO4	9.65	19.32	20.33	7.11	4.00

Table 4: Color parameters for dyeing of tea leaves on cotton fabric.

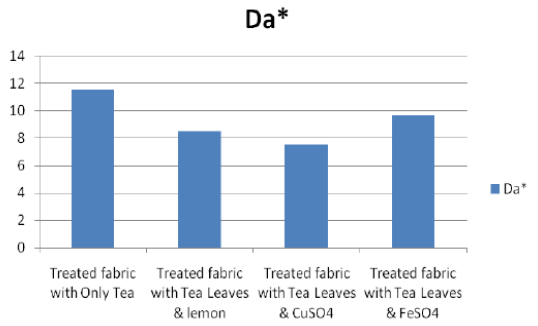

Figure 10: Analysis of Color parameter: Da*.

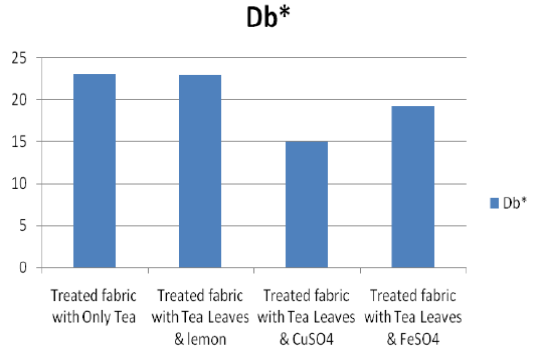

Figure 11: Analysis of Color parameter: Db*.

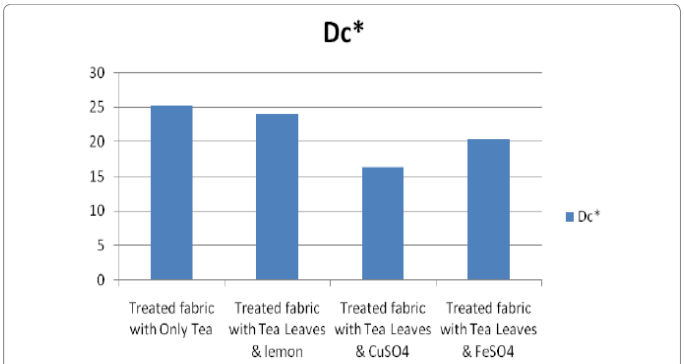

Figure 12: Analysis of Color parameter: Dc*.

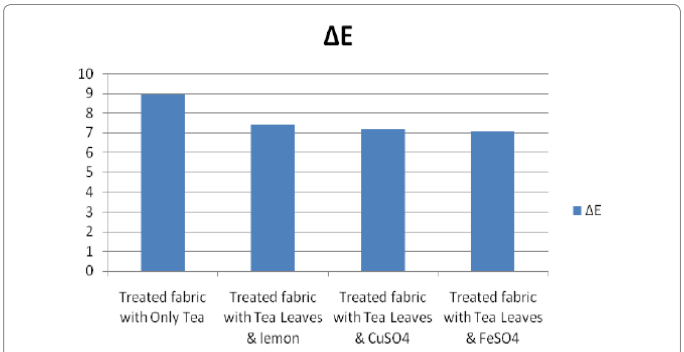

Figure 13: Analysis of Color parameter: DE.

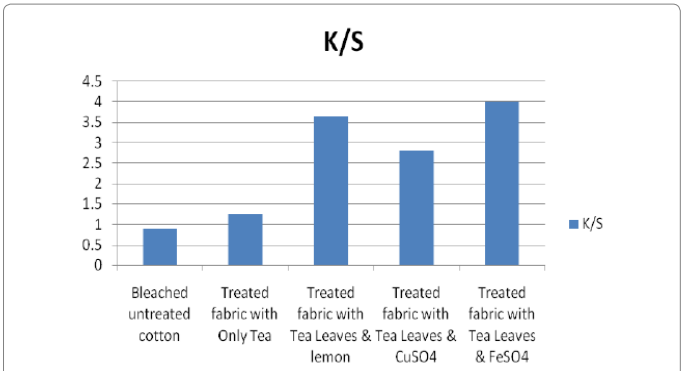

Figure 14: Analysis of color Strength K/S value.

Fastness properties for dyeing with tea leaves on cotton fabric

For the treatment with Tea Leaves, the color fastness to wash marked in graph that the treated sample with natural mordanting, lemon and synthetic mordanting, ferrous sulphate showed improved wash fastness comparing to conventional copper sulphate mordanting as well as very excellent light and rubbing fastness showed both mordanting with lemon and ferrous sulphate comparing to copper sulphate (Table 5 and Figures 15-18).

FTIR study of natural colored cotton fabric

Fourier Transform Infra-Red (FTIR) graphical analysis for natural colored cotton fabric showed an interaction among Cellulose-Curcumin-Citric Acid and it was clearly focused a curve that is given below. In this graph, the acquaintance of cotton fabric with Turmeric-curcumin-Lemon is good (Figure 19).

Treatments	Analysis of Color fastness to wash	Light fastness	Color fastness to Rubbing	
			Dry	Wet
Treated fabric with Only Tea Leaves	2	4	4	3
Treated fabric with Tea leaves & lemon	2-3	4	4	3
Treated fabric with Tea Leaves & $CuSO_4$	4	4	4	3
Treated fabric with Tea Leaves & $FeSO_4$	3	4	4	3

Table 5: Color fastness properties for dyeing with Tea Leaves on cotton Fabric.

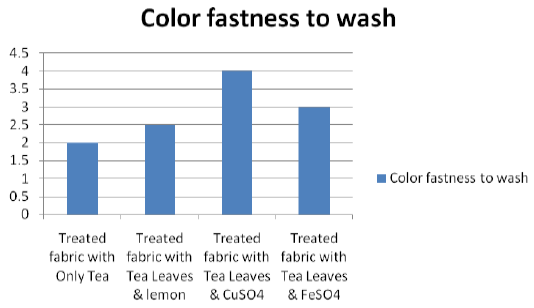

Figure 15: Analysis of Color Fastness to Wash.

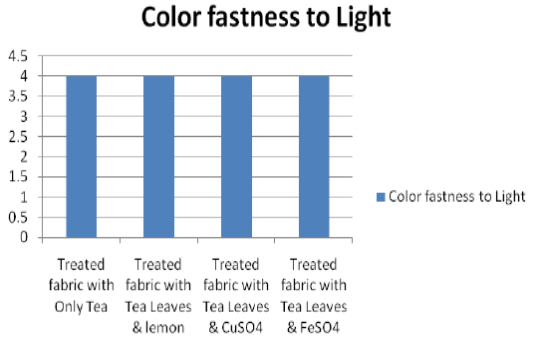

Figure 16: Analysis of Color Fastness to light.

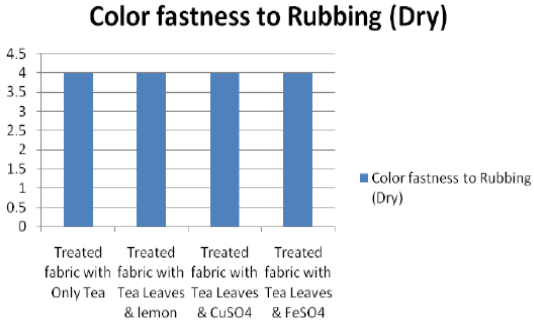

Figure 17: Analysis of Color Fastness to Rubbing (Dry).

Antibacterial Testing of natural colored Cot-ton Fabric: Curcumin demonstrates pharmacological properties that elements of curcumin colorant found effective against *E. coli* bacteria in laboratory experiment. Anti-bacterial testing of cotton fabric dyed and finished with Turmeric and Lemon had been tested in Bangladesh Council of Scientific and Industrial Research (BCSIR). In this testing, *E. coli* bacteria were applied on dyed and finished cotton fabric. After effective times, the *E. coli* bacteria were absent.

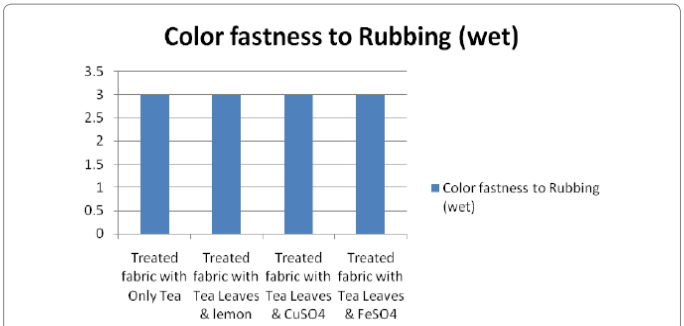

Figure 18: Analysis of Color Fastness to Rubbing (wet).

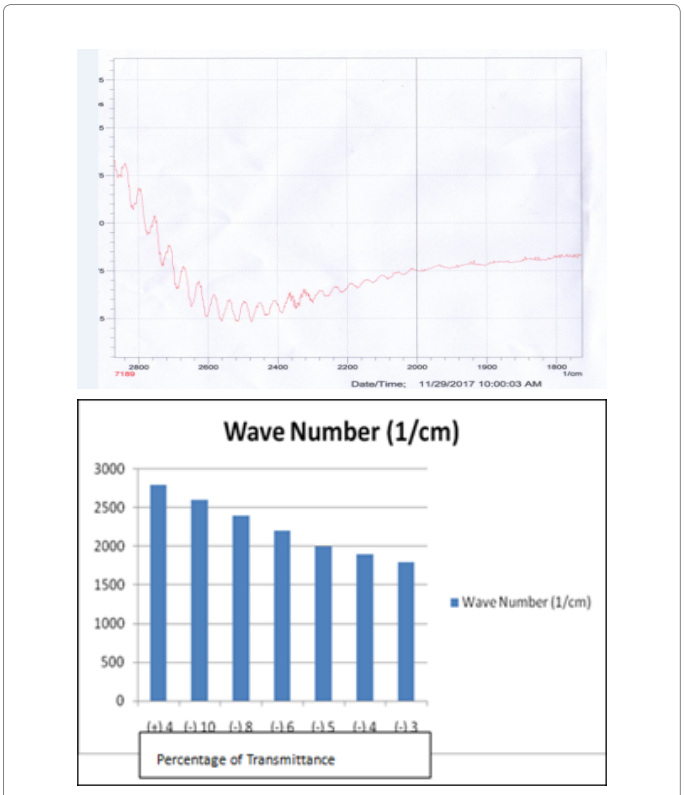

Figure 19: FTIR analysis for natural colored cotton fabric for Curcumin and Lemon.

Conclusion

Improved K/S values were investigated for both samples dyeing with curcumin and tannin colorant while mordanting with lemon comparing ferrous sulphate and copper sulphate mordanting. Curcumin and lemon dyed sample showed anti-bacterial under lab trailing of killing *E. coli* bacteria within a specific time interval which may spark the acceptation of customer requirements. Dyes and mordants concentration may influence the overall performance of dyeing and finishing. Public advertisement may increase to highlight the benefits of natural dyeing and finishing com-paring toxicity of synthetic dyes used in modern manufacturing plant. In this work, there is scope to do future scientific study for green chemistry and have ample opportunities to be commercialized, if it can be confirmed same stringent standards of performance that can be applied in-stead of synthetic dyes.

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