Developing Odor Free Textile with Natural Colorants Using New Nontoxic Mordant.

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Abstract — Odor free textiles enhance freshness of apparel and prevent staleness when stored or in contact with body sweat without use of toxic chemicals. In this research work, to create odor free fabrics, natural colorants such as flower of *tagetes erecta*, leaves of *syzygium cumini , swietenia mahagoni* and *psidium guajava* were selected for dyeing of cotton-jute blended single jersey structure fabric using potash alum mordant along with two new ecofriendly mordant like aliphatic polyamine and cellulase enzyme. To provide these dyed samples as good textile substrate, all dyed fabrics were tested in the following: color fastness to wash, perspiration, rubbing, dry cleaning and light fastness, mechanical test like bursting strength, physical test like bending length and odor test. Fabrics dyed with natural colorant using aliphatic polyamine and cellulase enzyme mordant demonstrate better results compared to others. All color fastness values are moderate to good. No significant strength loss is observed, but in all cases fabric softness is improved without using any softener. All fabrics dyed with natural colorants are odor free when using both potash alum and aliphatic polyamine mordant but not with cellulase enzyme.

Key words — Odor, *swietenia mahagoni*, *syzygium cuminni*, aliphatic polyamine, cellulase enzyme.

1 INTRODUCTION

ODOR free textile material means on which no odor is developed after sweating while fabric remain in contact with the human skin although sweated skin contain unpleasant odor.

Human senses can easily grasp pleasant to unpleasant odor often unconscious with feelings. Textile materials can easily attract, absorb and store various gaseous or volatile substances through desorption process as it contain a very large surface area. This is accelerated by temperature, time and gaseous exchange by airing [1].

Microbes which is not be seen with naked eye are present everywhere and textile substrate and human beings can carry different microorganism [2]. Textile apparel in contact with human body make an ideal environment for microbial growth [3]. Because of this after sweating odor is generated in textile material which is in contact with human body. Apocrin and sebaceous gland of human body secrete protein rich fluid on body skin which is good food for skin microbes resulting in unpleasant odorous bacterial metabolism. Textile apparel has been involved in contributing to this unpleasant odor intensity by the transfer of secretions, skin debris and bacteria from the body to the textile goods [4].

Now a days researchers seek methods not only dyed to textile materials but also provide different functional properties. Odor free finished fabric is such outcome and as significant one. Wearing these apparel can lead more hygienic life style. To provide cleaner and healthier lifestyle a range of textile products are treated with synthetic chemicals such as triclosan, metal and their salts, organometallics, phenols and quaternary ammonium compounds and natural product like aloe vera, tea tree oil, eucalyptus and tulsi leaf and many others natural extraction [5]. Although synthetic chemicals are very effective against odor producing microbes, they are causes of concerns due to health hazards, action on non-target microorganisms, and environmental pollution [6]. Whereas natural extraction have many advantages such as being non-toxic, non-irritant, biodegradable, cost effective and easily available [7]. Since ecological consideration and green-minded consumers are increasing all over the world, so the use of natural products are increasing [8].

Odor free is an important functional property of textile substrate because odorous substrates have a negative impact on textiles and their comfort so it is essential to develop permanent anti-odor finished products [1]. Many natural compounds can exhibit anti-microbial and deodorant properties for clothing which provide greater comfort and remain fresh and odor free in use [9]. To make odorous textile material, it is essential to contact with human skin which contain odor producing bacteria such as staphylococci, coryneform bacteria, micrococci, yeasts, propionibacteria. After physical exercise or emotional stress there are secretion from eccrine and sebaceous gland on human skin which are acidic and pungent, biting odorous compounds are produced due to bacterial metabolism. It has been identified that isovalerian acid and 3-methyl-2-hexenoic acid as significant odor molecules [10].

Although microorganism can easily attack cotton under hot, moist favorable condition but cotton-jute blended knit fabrics are more resistant to microbes due to jute containing about twenty percent lignin [11]. It is possible to make odor free substrate by dyeing with natural extract using eco – friendly mordant if they contain active components like tannins, phenols and saponins in the extracted dye liquor of natural plant [3], [5], [12].

In this research work, petals of *tagetes erecta* known as

marigold, family *Asteraceae*, leaves of *syzygium cumini*, family *Myrtaceae,* leaves of *swietenia mahagoni*, family *Meliaceae* and leaves of *psidium guajava*, family *Myrtaceae* were used in colorant extraction. Substrate was dyed using potash alum mordant along with two new ecofriendly mordant like aliphatic polyamine and cellulase enzyme. It was found that from their phytochemical screening they contain tanins, flavonoids, saponin and triterpenoids which show antimicrobial property [13], [14] & [15].

2 EXPERIMENTAL

2.1 Material

Table 1
Fabric specification of the knitted samples.

Features	Description
Yarn type	Blended yarn
Yarn composition	65% cotton, & 35 % jute
TPI (Twist per inch)	24
Twist type	Z
Yarn count	49 Tex
Fabric type	Knitted
Fabric construction	Single jersey plain (1x1)
CPI (coarse per inch)	44
WPI (Wales per inch)	30
GSM	240

2.2 Dye-stuffs and chemicals

Wetting agent & detergent (Imerol DLJ liq.), sequestering agent (Sirrix 2UD), lubricating agent (Imacol C2G), stabilizer (Stabilizer SOF), washing off agent (Ladipur SP), aliphatic polyamine (Indosol E-50), washing durability chemical (Optifix EC Liq.), reactive dye-stuff (Drimaren Blue HF) and antimicrobial finish (Sanitized T 99-19) were collected from Archroma International Ltd.(Dhaka, Bangladesh office).Acetic acid, Hydrogen peroxide 50%, sodium hydroxide, Glauber's salt, sodium carbonate, tannic acid, sodium chloride, methanol (Merck), potassium aluminium sulfate, ECE detergent, sodium perborate, L-histidine monohydrochloride, di sodium hydrogen orthophosphate, tetrachloroethylene were purchased from local market.

2.3 Natural Colorant

The color components from the selected leaves and petals which were extracted in solvent (methanol) extraction process was identified by botanical expert, Md. Islam Kazi, Director (corporate affairs, BHB group).At first the leaves of *syzygium cumini*, *swietenia mahagoni*, and *psidium guajava* were washed thoroughly with distilled water so that any dirt is removed. The petals of *tagetes erecta* were separated from the flowers carefully. The petals and leaves were exposed to sunlight until they became so dry that they could be grinded into small units. The dried petals and leaves were then broken into small units with the help of a blender. These broken units were blended into almost powder form. Large particles were removed by using a fine strainer. After weighing 100 gm of powder was dissolved in one liter of methanol (1:10) in a conical flask of thousand milliliter volume. The open end of the flask was tightly closed with a rubber cork to prevent the evaporation of methanol. The solution was kept for five days at not more than 30°C for better extraction. After five days the extracted dye liquor was filtered off by using filter paper. The very small particles dispersed in the solution were removed by filtering. Then the solution was concentrated to 50 ml by heating and continuous stirring (at a temperature not more than 50°C) and then it was ready for dyeing.

2.4 Sample preparation

Samples were pretreated and dyed with reactive dye-stuff and finished with antimicrobial finishing agent which recipes are illustrated in table 2 and 3. Again pretreated fabrics were dyed with colorants of patels of *tagetes erecta*, leaves of *syzysium cumini*, *swietenia mahagoni* and *psidium guajava* using potash alum, aliphatic polyamine and cellulase enzyme mordant and related recipes are illustrated in tables [4],[5] & [6].

Table 2
Recipe of one bath scouring and bleaching.

Chemicals and process parameters	Quantity
Wetting agent and detergent	1.0 g/l
Sequestering agent	0.8 g/l
Stabilizing agent	0.80 g/l
Anti-creasing agent	1.00 g/l
NaOH solid	1.50 g/l
H_2O_2 (35%)	4 ml/l
Material : Liquor	1:15
pH	11.5
Temperature	98°C
Time	60 m

Fabrics were neutralized using acetic acid after completing the pretreatment and this dried fabrics were ready for dyeing.

Table 3
Recipe of reactive dyeing and antimicrobial finishing

Chemicals and process parameters	Quantity
Reactive dye-stuff	1 % o.w.f
Glauber's salt	35 g/l
Soda ash	18 g/l
Material : Liquor	1:10
Temperature	60°C
Time	60 minutes

After draining the dyeing bath, fabrics were washed with cold water and then neutralized and were washed using washing off agent, Ladipur SP (1 g/l) at 95°C temperature for 15 minutes. Now reactive dyed fabrics were finished using antimicrobial finishing agent (Sanitized T 9919) 0.5 g/l maintaining pH 5.5 using acetic acid at 60°C for 30 minutes.

Table 4
Recipe of natural dyeing using potash alum mordant.

Chemicals and process parameters	Quantity
Potash alum	28.35 g/l
Soda ash	7 g/l
Material: Liquor	1:40
Temperature (for mordant)	98°C
Time (for mordant)	1 hours
Natural colorant	3 % o. w. f.
Temperature	60°C
Time	60 minutes

After treating with potash alum mordant fabrics were kept for 12 hours in the treated bath at 25°C and normal wash was done and fabrics were dyed with natural colorants. Then dyed fabrics were boiled for 10 minutes and dried, fabrics were then ready for tests.

Table 5
Recipe of natural dyeing using aliphatic polyamine as a mordant.

Chemicals & process parameters	Quantity
Aliphatic polyamine	2 % o. w. f.
Soda ash	1.5 g/l
Material : Liquor	1:20
Temperature	70°c
Time	60 minutes
Each natural colorant	3 % o. w. f.

At 70°C bleached fabric was treated first 30 minutes only with aliphatic polyamine and then it was treated further 30 minutes after adding soda ash in the same bath. After draining the bath, the fabric was neutralized with acetic acid. And then the fabric was washed with cold water and dried at normal temperature. This treated fabric was dyed with natural colorant at 60°C for 60 minutes according to the recipe. After completing dyeing the fabric was washed with both cold and hot water and the fabric was boiled for 10 minutes. Again that fabric was washed with cold water and dried.

Table 6
Recipe for natural dyeing using cellulase enzyme as a mordant.

Chemicals and process parameters	Quantity
Tannic acid	10 g/l
Enzyme	10 g/l
Material: Liquor	1:15
Temperature	60°C
Time	60
pH	7
Each natural colorant	3 % o. w.

At first tannic acid and enzyme were taken in a bath and the pH was adjusted by adding acetic acid. The temperature was raised to 60°C and kept for 30 minutes for treating the fabric. After that colorant was add to the bath and dyed the fabric for 60 minutes. After draining the bath the fabric was given cold wash, hot wash and boiled for 10 minutes. Again after one cold wash the fabric was dried.

2.5 Measurement of K/S, L, a, b, C, H values of dyed fabrics

Any shade is represented by the K/S (color strength), L (lightness of the color), a (red-green character of a color with positive values for red color and negative values for green color), b (yellow-blue character with positive values for yellow and negative for blues), C (Chroma), H (hues) values. All values were measured by spectrophotometer, model Data color 650 TM using Data color tools software [19].

2.6 Color fastness to wash (method ISO 105-C06C2S)

To make a composite test specimen a 10 cm x 4 cm dyed sample was sewed to a same size DW multi-fiber fabric. The test bath was prepared containing required quantity of ECE detergent, sodium per borate and this test was carried out at 60°C for 30 minutes in a machine. After that composite specimen was rinsed and dried in air not exceeding 60°C and

faded and stained adjacent fabric were assessed by colorimetric methods (A05 and A04) by spectrophotometer.

2.7 Color fastness to perspiration (ISO 105-E04)

A 10 cm x 4 cm dyed sample was sewed to a same size of DW multi-fiber fabric. In this way two composite test specimens were made. Both test composite specimens was wetted out thoroughly in the test solutions and excess solution was poured off from the test samples. Then these samples were placed in an oven for 4 hours at 37°C. Shade change and stained multi fiber were assessed by colorimetric method (A05 and A04) by spectrophotometer.

2.8 Color fastness to rubbing (ISO 105X12)

A 14 cmx 5 cm dyed fabric was placed in the crock meter. Then it was rubbed by 5 cm x 5 cm dry crocking fabric (for dry rubbing fastness), 10 strokes in 10 seconds. Wet rubbing fastness was determined using wet crocking cloth following same procedure. Both stained crocking fabrics were assessed by colorimetric method.

2.9 Color fastness to dry cleaning (ISO 105-D01)

A 10 cm x 4 cm dyed sample was placed in a cotton bag in which 12 stainless steel balls were present. The bag size was 10 cm x 10 cm. The bag was then placed in a stainless steel container. Then it was treated by 100 cc perchloroethylene (tetrachloroethylene) for 30 minutes at 30°C temperature in a washing machine. After 30 minutes, the test specimen was removed, squeezed and dried at room temperature. Remaining solvent was filtered off and taken into a test tube and was assessed by using grey scale. Shade change and staining of the solvent were assessed.

2.10 Color fastness to light (ISO 105-B01)

5 cm x 1 cm of dyed fabrics and 5 cm x 1 cm of blue wool reference standards were attached on 14 cm x 5 cm paper boards. Then the sample boards were placed inside the light fastness tester and it was exposed to daylight for 52 hours. The the mirror cutting both the index lines. The bending length could immediately be read off from the scale. Four readings were taken from each specimen, one face up and one face down on the first end and then the same for the second end. The mean bending length for wales and courses were calculated.

2.12 Bursting strength of the fabric (ISO 13938-1, Diaphragm Bursting Test)

The fabric to be tested was clamped over a rubber diaphragm by means of an annular clamping ring and an increasing fluid pressure was applied to the underside on the diaphragm until fabrics were then evaluated by comparing with the blue wool standards. [17].

2.11 Bending length of the fabrics (BS 3356:1990, Cantilever stiffness test)

2.13 Odor test (Subjective analysis, SNV 195651)

The test specimen (40gm) was taken and placed on top of 300 ml saturated of sodium carbonate solution. Then it was kept in a closed container. We used desiccator as a closed container. The container was put into an oven set to a temperature of 37±2°C for 15 hours. Minimum six people were required independently to judge the odor intensity. The rating of odor intensity was done according to the nomenclature [12].

3 Results and Discussion

Table 7
K/S, L, a, b, C, H values of all dyed fabrics.

DF	M	K/S	L	a	b	C	H
TE	PA	3.56	69.40	10.14	36.30	37.69	74.39
	AP	3.06	70.09	4.50	20.72	21.21	77.74
	CE	2.86	62.69	5.48	13.78	14.83	68.33
SC	PA	3.25	68.18	5.04	20.82	21.42	76.40
	AP	3.15	68.21	4.88	20.52	21.10	76.63
	CE	2.75	61.90	5.04	12.19	13.19	67.54
SM	PA	2.69	69.18	7.52	23.25	24.43	72.08
	AP	3.36	64.98	9.66	19.58	21.83	63.75
	CE	2.42	63.40	6.18	12.25	13.72	63.23
PG	PA	1.82	78.03	1.77	21.17	21.25	85.22
	AP	2.39	72.70	4.01	20.24	20.63	78.78
	CE	2.46	62.80	6.36	11.77	13.38	61.63
RD, AMF		5.01	45.16	-2.69	-26.45	26.58	26.2

that the specimen bursts within 20±3 seconds [18]. All measured values of dyed fabrics were recorded.

TE=*Tagetes erecta*, SC= *Syzygium cumini*, SM= *Swietania mahagoni*, PG= *Psidium guajava*, RD=Reactive dyed, AMF= Antimicrobial finish, PA= Potash alum, AP= Aliphatic polyamine, CE= Cellulase enzyme.

Color fastness to wash, method ISO 105 – C06 C2S
Shade change after color fastness to wash test.

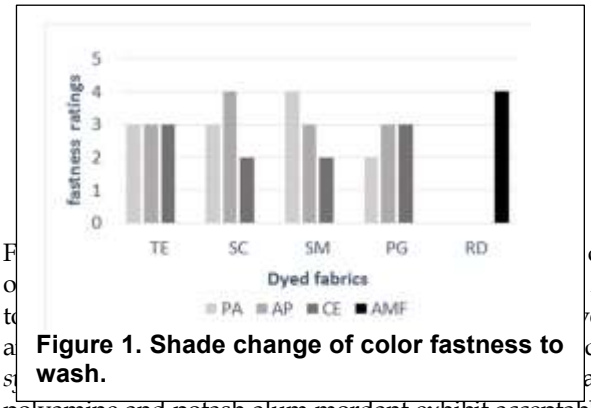

Figure 1. Shade change of color fastness to wash.

F changes
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polyamine and potash alum mordant exhibit acceptable result which is 4 but others values are 2 to 3 which are poor to moderate.

Staining on multi fiber of color fastness to wash

Table 8
Staining on multi fiber of dyed fabrics of color fastness to wash test.

DF	M	Ratings of stained multi fiber					
		DC	BC	PA	PET	PAC	WO
TE	PA	4.5	4.5	4.5	4.5	4.5	5
	AP	4	4	3.5	4	4	5
	CE	3.5	3.5	4	4	4	5
SC	PA	3.5	3.5	3.5	3.5	3.5	4.5
	AP	4.5	4.5	4.5	4.5	5	5
	CE	3	3	3	3	3	4
SM	PA	5	5	5	5	5	5
	AP	5	5	5	5	5	5
	CE	3.5	3.5	4	4	4	4.5
PG	PA	5	5	5	5	5	5
	AP	4	4.5	4.5	4.5	4.5	5
	CE	4.5	4	4	4.5	4.5	4.5
RD, AMF		4	4.5	4	4.5	5	5

Where, DC= Di acetate, BC= Bleached cotton, PA= Polyamide, PET= Polyester, PAC= Poly acrylonitrile, WO= Wool.
From table 8, good to excellent values 4 to 5 are obtained for dyed fabrics with colorants of *tagetes erecta* using potash alum, *syzygium cumini* using aliphatic polyamine, *swietenia mahagoni* using potash alum and aliphatic polyamine, *psidium guajava* using potash alum, aliphatic polyamine and cellulase enzyme mordant and reactive dyed antimicrobial finished fabrics. Others all values are moderate.

Color fastness to perspiration
Shade change of dyed fabrics of color fastness to acid perspiration.

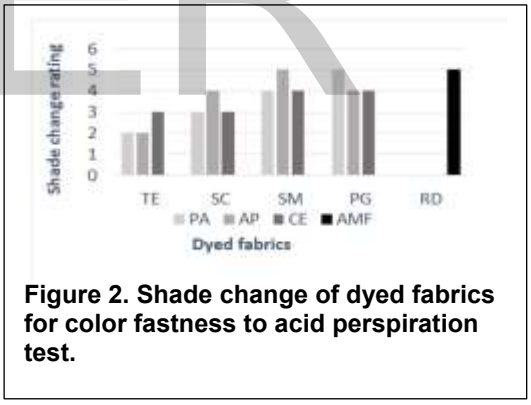


Figure 2. Shade change of dyed fabrics for color fastness to acid perspiration test.

Dyed with colorant extracted from *syzygium cumini* using aliphatic polyamine, *swietenia mahagoni* using all three mordant, *psidium guajava* using all three mordant and reactive dyed antimicrobial finish fabrics exhibit good to excellent values 4 to 5. Others values are poor to moderate.

Shade change of color fastness to alkali perspiration.

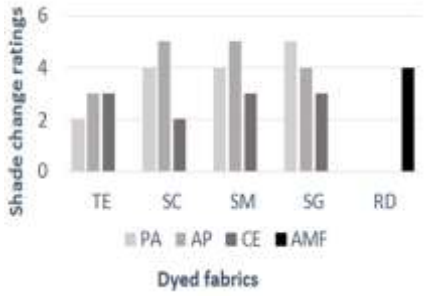


Figure 3. Shade change of dyed fabrics for alkali perspiration test

Good to excellent values of 4 to 5 is observed for dyed fabrics dyed with colorant extracted from leaves of *syzygium cumini*, *swietenia mahagoni*, *psidium guajava* using potash alum and aliphatic polyamine and reactive dyed antimicrobial finish. Others values are poor to moderate.

Staining on multi fiber of acid perspiration test.

Table 9
Staining on multi fiber of dyed fabrics for color fastness to acid perspiration.

DF	M	Staining on multi fiber					
		DC	BC	PA	PET	PAC	WO
TE	PA	4.5	4.5	4	4.5	4.5	4.5
	AP	5	5	5	5	5	4.5
	CE	4.5	4.5	4	4.5	4.5	4
SC	PA	4.5	4.5	4.5	4.5	4.5	5
	AP	5	5	5	5	5	5
	CE	4	4	4	4	4	4.5
SM	PA	4.5	4.5	4	4	4.5	4.5
	AP	5	5	4.5	4.5	5	5
	CE	4	4.5	4	4.5	4.5	4.5
PG	PA	4.5	4.5	4.5	4.5	4.5	5
	AP	5	5	5	5	5	5
	CE	4	4.5	4	4.5	4.5	4
RD, AMF		5	5	4.5	5	5	5

Table 10

Staining on multi fiber of dyed fabrics for color fastness to alkali perspiration test.

DF	M	Stained on multi fiber					
		DC	BC	PA	PET	PAC	WO
TE	PA	4.5	4.5	4.5	4.5	4.5	4.5
	AP	4.5	5	5	5	5	5
	CE	3.5	3.5	3	3	3	4
SC	PA	4.5	4.5	4	4.5	4.5	5
	AP	4.5	4.5	4.5	4.5	5	5
	CE	4.5	4	4	4	4	5
SM	PA	4	4.5	4	4.5	4.5	5
	AP	4.5	5	4.5	5	5	5
	CE	3.5	3.5	3	3	3	4
PG	PA	4.5	4.5	4.5	4.5	4.5	5
	AP	5	5	5	5	5	5
	CE	4	4	4	4	4	4.5
RD, AMF		4.5	5	4.5	5	5	4.5

From table 9 and 10 it is deduced that staining on multi fiber of all dyed fabrics show good to excellent results after acid and alkali perspiration except cellulase enzyme mordant.

Color fastness to rubbing test.
Dry rubbing fastness test.

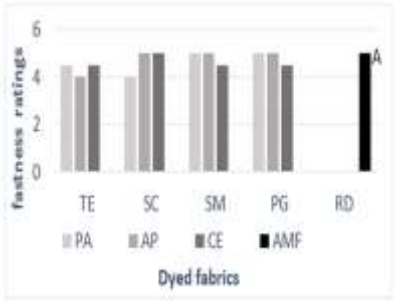
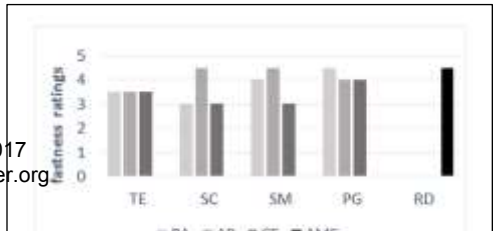


Figure 4. Color fastness to dry rubbing of dyed fabrics.

According to above figure, it is concluded that the ratings for dry rubbing are good to excellent that is 4 to 5.

Wet rubbing test



Fabrics dyed with *psidium guajava* colorant shows good result, whereas *tagetes erecta* colorant gives poor result. It can be said that color fastness for wet rubbing is lower than dry rubbing.

Color fastness to dry cleaning

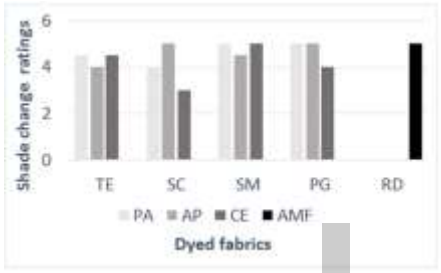


Figure 6. Shade change of dyed fabrics for color fastness to dry cleaning.

All shade change values are good except dyed fabric with *syzygium cumini* colorant using cellulase enzyme mordant.

Staining of bath liquor of color fastness to dry cleaning

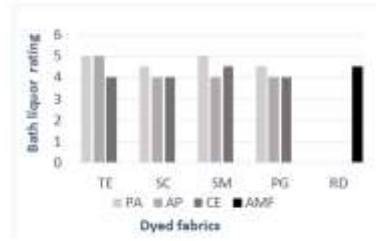
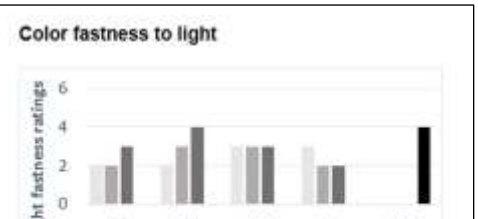


Figure 7. Staining on bath liquor of dyed fabrics for color fastness to dry cleaning.

From above illustration it is clearly understood that liquor staining of dry cleaning test is quietly satisfactory due to all values being well within acceptable ranges which are 4 to 5.

Color fastness to light



Poor light fastness is observed for all dyed fabrics except dyed fabrics with *syzygium cumini* colorant using cellulase enzyme and reactive dyed antimicrobial finished fabrics.

Bending length

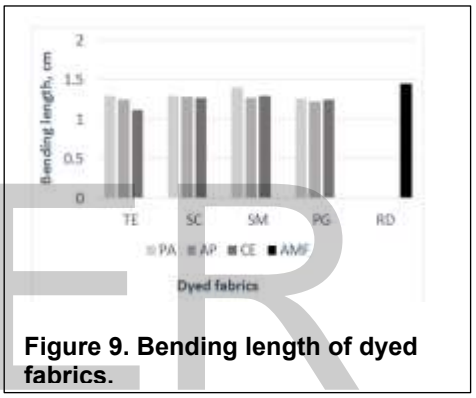


Figure 9. Bending length of dyed fabrics.

From above graphical representation it is deduced that the bending length of fabrics lies nearly between the regions of 1.12 cm to 1.45 cm. Maximum bending length is observed for reactive dyed fabric, which means reactive dyed fabric is stiffer than the natural dyed fabrics. The lowest bending length is found for cellulase enzyme mordanted dyed with *tagetes erecta* colorant, hence this fabric is softer. It means that *tagetes erecta* contains more oily substance than others.

Bursting strength.

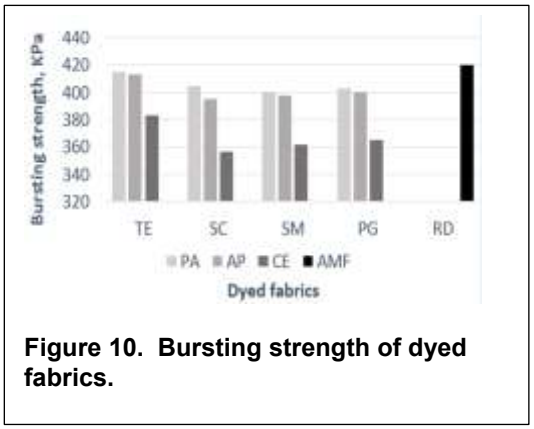


Figure 10. Bursting strength of dyed fabrics.

Although maximum bursting strengths are observed for dyed fabrics dyed with colorants of *tagetes erecta*, *syzygium cumini*, *swietenia mahagoni*, *psidium guajava* using potash alum and aliphatic polyamine mordant and antimicrobial finished reactive dyed fabrics but dyed fabrics dyed with same colorants using cellulase enzyme mordant show lower bursting strength.

Washing durability test.

Table 11
Washing durability test of dyed fabrics dyed with *tagetes erecta* colorant using different mordant and aftertreated with fixing agent.

M	Fixing agent	Absorbance values of washing liquors 1st	2nd	3rd	4th	5th
PA	Untreated	1.6	0.9500	0.945	0.919	0.843
	Treated	0.83	0.874	0.816	0.802	0.800
AP	Untreated	2.24	1.098	1.06	1.00	0.885
	Treated	0.90	0.908	0.907	0.85	0.823
CE	Untreated	1.41	0.93	0.89	0.876	0.822
	Treated	0.82	0.816	0.789	0.815	0.769

All absorbance values were measured from spectrophotometer. Higher absorbance values means more colorants in the wash bath liquor. Almost same absorbance values are seen in the table 11 for 5th wash of untreated and 1st wash of treated samples. So it can be said that fixing agent improves washing durability of natural dyed fabrics.

Odor test of dyed fabrics

For assessing whether the fabric is odor free or not, it is necessary to carry out an odor test. Odor test of the fabric was performed by following SNV 195651.

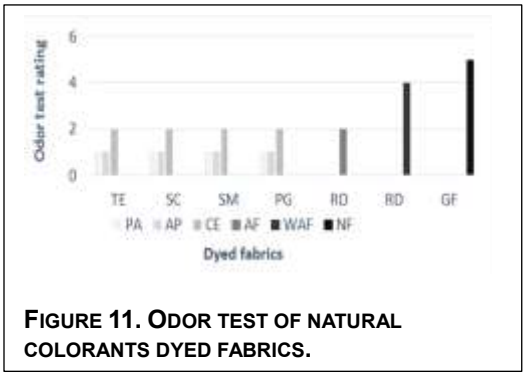


FIGURE 11. ODOR TEST OF NATURAL COLORANTS DYED FABRICS.

According to SNV 195651 (subjective) odor test, natural dyed fabrics with colorant of *tagetes erecta*, *syzygium cumini*, *swietenia mahagoni* and *psidium guajava* using ecofriendly mordants like potash alum and aliphatic polyamine are odor free. However same colorant dyed fabrics using cellulase enzyme as a mordant demonstrates weak odor. On the other hand grey fabric has annoying odor and reactive dyed fabric without using any finishing agent shows intolerable odor. But reactive dyed finished with antimicrobial agent demonstrates weak odor.

4 CONCLUSION

Natural colorants are revealed to be able to produce odor free fabrics. Present innovative research work introduces extracted colorants from flower of *tagetes erecta*, leaves of *syzygium cumini*, *swietenia mahagoni* and leaves of *psidium guajava* which has anti-odor property. So without using any toxic chemicals for finishing it is now possible to develop odor free fabrics. Two new mordant aliphatic polyamine and cellulase enzyme exhibit moderate to good results through tests color fastness, physical and mechanical. So these natural dyed fabrics would be quietly fulfill odor free requirement for apparel purpose. Here only one shade percentage of dyeing has been done it is recommended to do different higher shade percentage so that antimicrobial test can be done.

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