Application of Purified Curcumin as Natural Dye on Cotton and Polyester

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***Abstract*-- Though there are some limitations with the use of natural dyes, the use of the dyes is increasing day by day due to the eco-friendly approach of the people. The extraction process of natural dyes from their sources and methods of application of the dyes on different fabrics are very important factor. This work concerns with the extraction and purification of natural dyestuff from a plant Curcuma Longla L. and dyeing of cotton and polyester fabric in exhaust dyeing method. The main coloring component of turmeric is curcumin, which produces yellow color on the textile materials. The purified curcumin produces various shades on cotton and polyester fabric with different dyeing parameters and use of mordants. The color fastness properties of the dyed fabrics are also good.**

***Index Term*-- Column chromatography, Curcumin dye, Solvent extraction method, Thin layer chromatography.**

1 INTRODUCTION

People have dyed the textile materials since thousands of years and most of the times for this purposes, the dyes have come from natural sources [1]. People used these dyes in cosmetics, food [2,3], leather and also in medicine [4]. The use of natural dyes has increased for dyeing the textile materials after the development of weaving technique [5].

After the invention of synthetic dyes by William Henry Perkin in 1856 [6], the use of synthetic dyes has become faster acceptability in the field of food [7], cosmetic [8] and more importantly in the field of textile industries [9]. The manufacturers preferred the dyes because of easy dyeing process, variation of shades, color fastness properties as compared to natural dyes. But, the dyes cause some serious health hazards like allergic, carcinogenicity, and skin diseases [10], though they are widely and commercially popular. They pose also threat towards its eco-friendliness, as most of the dyes contain azo groups of aromatic amines that may be harmful to human health and environment [11]. Due to environmental awareness and harmful effects of either toxicity or non-biodegradable nature of synthetic dyes, natural colorants are thinking globally due to the fact that the majority of the sources are safer, more environmental friendly and thus the application of natural dyes should be considered as a better alternative to synthetic dyes. Natural dyes are more acceptable to environmentally conscious people around the world (A. K. Roy, 2006). These types of dyes also have antibacterial, deodorizing, UV-protection properties (Feng, Zhang, Chen, 2007; Daniele, Laura, 2011; Shafat, Aijaz, 2012 & Gupta, Khare, 2004).

The present study focuses on the development of the optimum extraction conditions of coloring component from the natural material turmeric i.e. Curcuma Longla L. and then the major component of the dye i.e. curcumin is purified. The chemical structure of curcumin is shown in figure 1. Finally the purified dye is applied on cotton and polyester (PES) fabric in exhaust dyeing method. The color strength value, dye exhaustion% and color fastness properties of the dyed fabrics are evaluated and compared with each other.

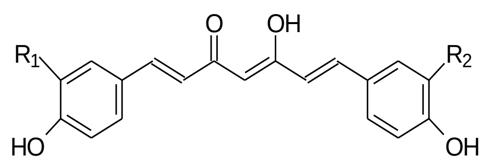

Fig. 1. Chemical structure of soluble curcumin[13]

2 MATERIALS AND EXPERIMENTAL METHODS

2.1 Dyestuff used

Turmeric i.e. Curcuma Longla L. is used to obtain the dyestuff of curcumin, which is yellow in color. Turmeric is collected from the local market of Bangladesh.

2.2 Fabric used

Cotton and polyester fabrics are selected for the present study, which are collected from WFK- GmbH, Germany. Cotton fabric is scoured-bleached and heat setting is done for PES fabric before dyeing.

2.3 Chemicals used

Ethanol (C_2H_5OH), chloroform ($CHCl_3$), acetone (C_3H_50H) and n-hexane (C_6H_{14}) are used as solvent to extract the dye from Curcuma Longla L. The chemicals are collected from Sigma- Aldrich GmbH and Carl Roth GmbH, Germany.

2.4 Mordants used

The following mordants are used: potassium aluminium sulphate ($AlK(SO_4)_2.12H_2O$), copper sulphate ($CuSO_4$) and tartaric acid ($C_4H_6O_6$). They were pure grade chemicals used for laboratory purpose and Collected from Carl Roth GmbH, Germany.

2.5 Extraction of curcuminoid from turmeric

Fresh rhizomes of turmeric are washed and cleaned with distilled water. They are then sliced and dried in the sunlight for one week and again dried at 80^0c for one hour in a hot air oven. Dried rhizomes are made in powder form for getting proper extraction result. 10 gm of turmeric powder are taken into a thimble and placed in a soxhlet apparatus. 200 ml of solvent is added in soxhlet for 10 gm of sample and the extraction is carried out according to the boiling point (B.P.) of solvent for 8 hours. After extraction the dark brown extract is cooled, filtered, concentrated by evaporation. Finally, a crude dried extract is obtained, which is black orange in color. The dye is extracted from turmeric using different solvent according to their boiling point by the same method and yield is calculated. The extracts are dissolved in the same amount of water to compare the concentration of the color by using UV/VIS spectroscopy for getting the best extraction result.

2.6 Separation of curcumin from curcuminoid by TLC

Solvent extracts are tested in TLC for presence of three different color components of turmeric. The process used a thin plastic plate (Merck-60 F254, KGaA, Deutschland). Using a capillary tube, a row of spots of the appropriate extract along a line about 1 cm from the bottom of TLC strip is applied. The spot have to dry completely and this took 30 seconds or more. The plate is placed vertically in a jar, which is pre-saturated with the mobile phase. Chloroform with methanol and chloroform with hexane with different ratio are used as solvent to separate the color components. The polarity of the solvent is changed as the mixing ratio of the chemicals is different. When the solvent runs upward, it passes the sample and starts to carry the compounds upward with it. The different components in the mixture move up the plate at different rates due to differences in their partitioning behavior between the mobile liquid phase and the stationary phase. The more polar a molecule, the more strongly it is adsorbed. As a result, the compounds, the relatively more polar ones, remain near the bottom of the plate while others, less polar ones are carried by the solvent nearer the top. When the solvent front reaches the other edge of the stationary phase, the plate is removed, dried and spots are visualized in UV light. Finally, the R_f values of the components of different mobile phase mixture are calculated from the TLC plate as shown in figure 2.

The R_f values are determined by the equation no (1). The R_f value is used to separate the components due to their uniqueness to each compound.

$$R_f = \frac{\text{Distance from baseline traveled by solute}}{\text{Distance from baseline traveled by solvent}} \qquad (1)$$

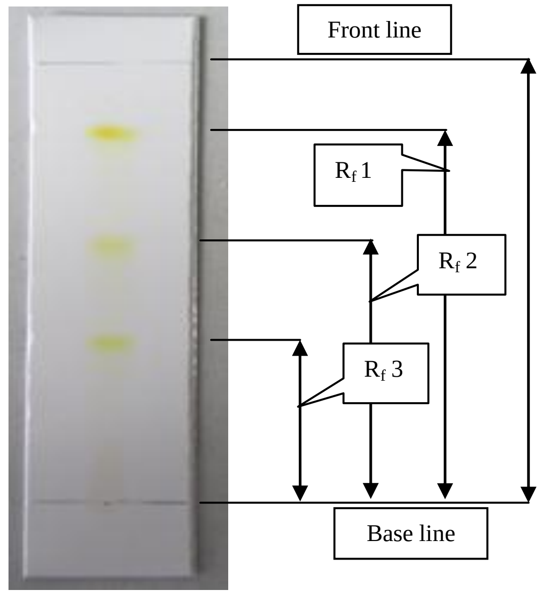

Fig. 2. Calculation of R_f from developed TLC plate

2.7 Purification of curcumin by column chromatography

The column chromatography consists of a glass tube with a diameter of 30mm and a height of 70cm with a tap at the bottom. The column is clamped vertically. The stopcock of the column is closed and it is fitted with fritted disk. The silica gel is weighed out in a flask and enough solvent (eluent) is added with stirring to form a slurry. Then, slurry is slowly transferred into the column using a glass funnel until the silica gel level is about 30-40cm. When the solvent reached the silica gel surface, slowly added the mixture solution of curcuminoid into the column using a dropper. After the sample is loaded, a small layer of sand is added to the top of the column. This helped to keep the top of the column level when added solvent eluent. Once the mixture is added and the protective layer of sand is in placed, continuously added the solvent eluent while collecting small fractions at the bottom of the column. The individual components are retained by the stationary phase differently and separated from each other while they are running at different speeds through the column with the eluent. In this way the major component of dye is obtained from turmeric.

2.8 Identification of curcumin by IR spectroscopy

5mg of purified dry sample is placed directly into the infrared beam of IR spectroscopy. As the IR radiation is passed through the sample, the transmitted energy is measured and a spectrum is generated. The resulting spectrum represents the molecular absorption and transmission creates a molecular fingerprint of the sample. As there is a unique combination of atoms in every material, so it is not possible to produce the same infrared spectrum of two compounds. Therefore, the instrument gives a positive identification of the purified material.

2.9 Optimization of dyeing conditions for cotton and polyester fabrics

Cotton and PES fabrics are dyed with the curcumin dye at a liquor ratio of 1:40. For optimizing the dyeing conditions, at first, experiments are carried out to optimize the dyeing pH. Dyeing processes are carried out at 75^{o}C for cotton and 130^{o}C for PES fabric with 2% shade (o.w.f) of purified dye at pH 3, 4.5 and 7. To get the effect of dyeing temperature on the color strength, another set of experiment is carried out in optimized dyeing pH at 60, 75, 100 and 130°C for cotton and 100,120,130 and 140^{o}C for PES fabric. The time are taken 30, 45, 60 and 90 minutes. Another set of experiment is also carried out at different dye concentration such as; 2%, 4%, 6%, and 8% in optimum dyeing conditions. Based on the K/S values of the dyed samples, optimum dyeing pH, temperature and time are selected and taken for further study. To study the effect of different mordants and their varying concentrations on color strength, mordanting is carried out with three metallic salts such as; aluminium potassium sulphate, copper sulphate and tartaric acid. Mordanting is carried out in pre-mordanting technique. Mordanting is done at 100^{o}C.

After dyeing, the dyed samples are rinsed with cold water, washed in a bath with a liquor ratio of 1:50 using 1 gm/liter of the soaping agent at 60^{0}C for 10 minutes and then they are rinsed and finally dried in a dryer.

2.10 Evaluation of color strength

Estimation of color strength of the dyed fabrics are carried out by determining the K/S values using a computer color matching system (CS-5, Applied color system, USA). The reflectance value (R) in the visible wavelength region is measured by means of the ACS spectrophotometer. The value of reflectance (R) of the dyed fabric is measured at the wavelength of 420 nm and also the K/S value of the sample is found directly from the instrument. Every dyed sample is measured in the same way and the K/S values are obtained directly from the instrument, which followed the Kubelka-Munk theory as in equation (2).

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

Where, K and S are the absorption and scattering co-efficient of the sample. R is the absolute reflectance.

2.11 Measurement of dye exhaustion by UV/VIS spectroscopy

Degree of exhaustion is the amount of dyestuff, which is diffused in the fiber from the dye bath at the time of dyeing. UV/Vis spectrophotometer is used to measure the exhaustion of the dyestuff. By measuring the concentration of dye bath before and after the dyeing process, the percentage of exhaustion is estimated with the equation (3) considering the color of metal salts.

$$E\% = \frac{C_1 - C_2}{C_1} x10 \qquad (3)$$

Where, C_1 and C_2 are the concentrations of the dye bath before and after dyeing process respectively.

The concentrations of the dye solution before and after dyeing are measured using UV/VIS spectrometer at the wavelength of 420nm. Before measuring the absorbency, the wavelength of maximum absorbency is determined for the dye by using the calibration standard solutions. For the calibration standard solutions, a dye stock solution of 20 mg in 20 ml water is prepared. By pipetting 1 ml to 5 ml of this stock solution are taken and diluting to 100ml, 5 different calibration standard solutions are prepared. The concentrations of the calibration standard solutions ranged from 0.01 g/l to 0.05 g/l. The dye solution is taken into a cuvette with a width of 1cm and subsequently the absorption spectrum is recorded, which are presented in figure 3.

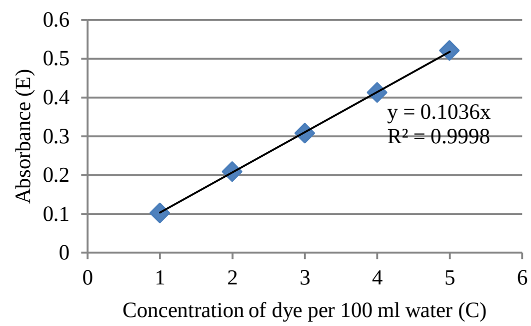

Fig. 3. Calibration curve of curcumin dye

The value of extinction coefficient is obtained from the linear regression line (y = mx), where the value of 'm' corresponds the extinction coefficient of the dyes.

The extinction coefficient of the dye is determined as per Beer-Lambert Law, based on the equation no (4):

$$\log\frac{I_0}{I} = E = \varepsilon c d \qquad (4)$$

Where,

I_o - is intensity of the initial light

I - is intensity of the light after passing through the dye liquor

E - is Extinction at a specific wavelength of λ

ε - is extinction coefficient

c - is concentration of the dye liquor

d - is cuvette width

2.12 Evaluation of color fastness properties

Wash fastness of the samples dyed under the optimized conditions is tested according to ISO 105-CO3 method. ISO 105-X 12 test methods is followed to measure the rubbing fastness. Color fastness of textile material to day light is of considerable importance to the consumer. The specimen should be tested according to ISO test method. The light fastness of the dyed test samples (1x 3 cm) are exposed to UV light of a Xeno tester for 40 hours. The temperature of the machine was 30^{0} C, R.H. 65% and irradiation dose 980 KJ/m^{2}. The fastness is assessed by comparing the fading of the specimen with that of blue wool patterns.

3 RESULTS AND DISCUSSION

3.1 Result of solvent extraction

Soxhlet extracts are weighted after drying and weight percentage of curcuminoid dye are calculated, which are shown in table I. The curcuminoid dye is obtained in the form of dark black orange color by using methanol and ethanol, acetone and water. Ethanol or methanol extract showed better result than the other. In this experiment, the ethanol extracted curcuminoid dye is used to separate the curcumin dye.

Table I
Amount of extracted curcuminoid dye in percentage

Solvent	Extraction % of curcuminoid
Ethanol	5.00
Methanol	5.05
Acetone	4.10
Water	2.60

The absorbance of the extracted curcuminoid dye using different solvents is also analyzed by UV-visible spectroscopy at 420nm. The extracted dyes are dissolved in a definite amount of water to compare the concentration of different solvent extract. The absorbance of dye solution is directly related to the concentration according to Lambert-Beer's Law. It is found that ethanol extracted dye has more concentrations than the other. So ethanol extracted dye is used in column chromatography to obtain pure curcumin dye.

3.2 Result of column chromatography

The better R_f values are found 0.28, 0.19, 0.12 for curcumin (C), dimethoxy-curcumin (DMC), bisdimethoxy-curcumin (BDMC) respectively, when chloroform and hexane are used as the mobile phase in the ratio of 4:1.

Different compositions of mobile phase are also tested in TLC for the separation of curcumin dye. The R_f values of the components are calculated, which are shown in table II. If the other ratio is used for separating curcumin dye component by column chromatography, which has high or low R_f values, then the separation will take very short time or very long time. This can affect on the result of purification of curcumin dye. That's why; the other results are not used in column chromatography. Hence the mixture of chloroform and n-hexane in the ratio of 4:1 is suitable eluent for the separation of curcumin in column chromatography.

Table II
R_f values using different mobile phase

TLC mobile Phase	Ratio	R_f values		
		C	DMC	BDMC
$CHCl_3$:CH_3OH	19:1	0.49	0.33	0.21
$CHCl_3$:Hexane	4:1	0.28	0.19	0.12
$CHCl_3$:Hexane	3:1	0.33	0.13	0.04

The ethanol extracted curcuminoid dye is subjected to column chromatography by using the mobile phase of chloroform and n-hexane and the fractions are collected and tested with TLC. The test showed that the curcumin dye is separated. From the collected fractions of the component, the percentage of curcumin is calculated and it is found 3.15% of total turmeric.

3.3 Result of IR spectroscopy

The infrared spectrum of curcumin is shown in figure 4, which is determined by IR spectroscopy. The purified curcumin spectrum is almost same as the spectrum of Ferrari and Lazzari [14].

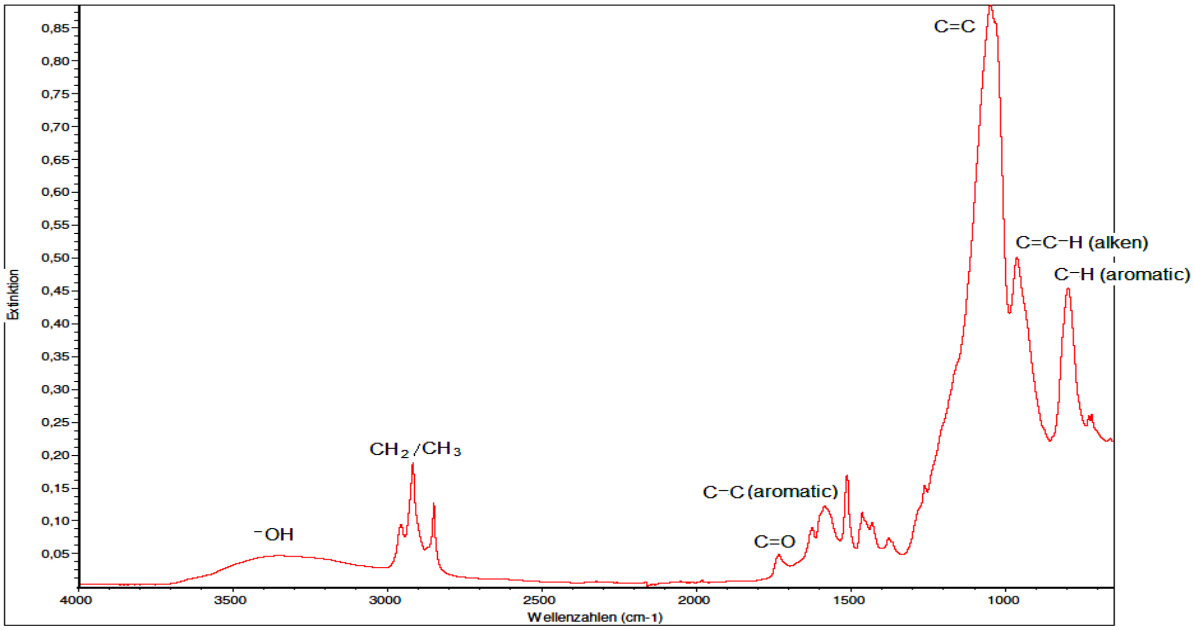

Fig. 4. IR spectrum measured by IR spectroscopy

3.4 Effect of dye concentration on color strength of cotton and polyester fabric

The color strengths (K/S) of the dyed cotton and polyester fabrics are dependent on the concentration of the extracted curcumin dye as shown in figure 5. The hue of the dyed cotton and polyester fabrics are yellowish, when the percentage of dye (o.w.f) is less than 2. If the percentage of dye is increased, then the fabrics showed yellowish brown color. From the results, it is seen that the color strength, expressed as K/S, increased with increasing concentration of curcumin dye up to a certain limit. It is also observed from the result that there is a significant difference in the depth of shade between cotton and polyester fabrics; though the dyeing condition was optimum. It is observed that the color strength of the polyester fabric is lower than the cotton fabric at the same concentration. The color strength of polyester is lower than cotton because it is highly crystalline fiber and it is hydrophobic in nature.

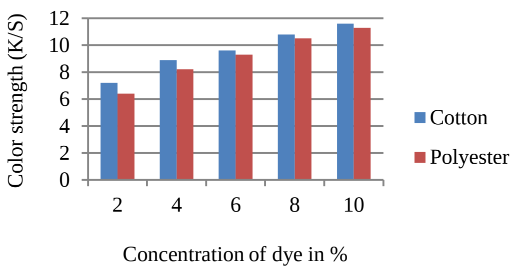

Fig. 5. Effect of concentration of curcumin dye on color strength of cotton and polyester fabrics

3.5 Effect of pH on color strength of cotton and polyester fabric

Figure 6 showed that the color strength (K/S) of cotton fabric is increased with increases in the pH value of the dyeing bath from pH 3 to 7. For polyester fabric the better result is observed in terms of K/S value at pH 4.5. The maximum dye uptake is obtained for cotton at pH 7. In the alkaline solution, curcumin reacts with alkali, that's why it is not possible to dye the fabrics in the alkaline medium.

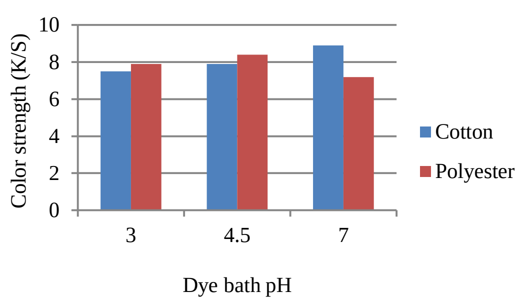

Fig. 6. Effect of pH on color strength of cotton and polyester fabrics dyed with curcumin (4% shade)

3.6 Effect of temperature on color strength of cotton and polyester fabric

The effect of dyeing temperature on color strength of cotton and polyester fabric is demonstrated in figure 7. As evident**,** the maximum color strength is obtained at 75^0C for cotton fabric and for polyester fabric it is found at the temperature of 130^{o}C. The shade is also found very uniform in both cases. If the temperature is increased more than 75^0c for cotton fabric, then the depth of shade is decreased. The result is also not good, if the fabric is dyed less than 75^0c. The polyester fabric required the temperature of 130^{o}C for maximum exhaustion because of the crystalline structure of the fiber.

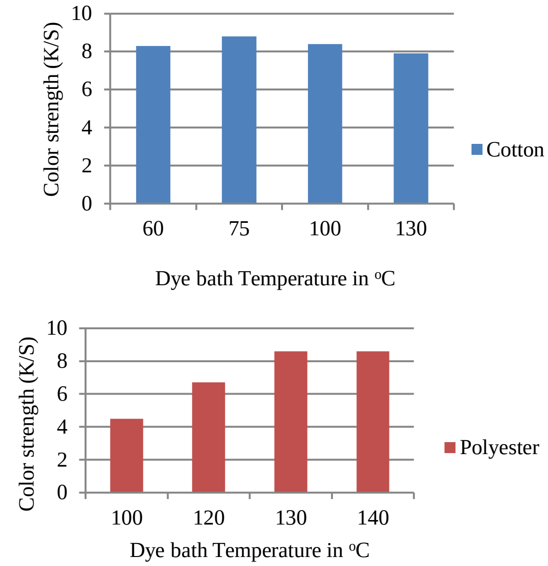

Fig. 7. Effect of temperature on color strength of fabrics dyed with curcumin dye (4% shade)

3.7 Effect of time on color strength of cotton and polyester fabric

The effect of dyeing time on color strength is shown in figure 8. The best results with respect to time for dyeing cotton and polyester fabrics are obtained at 45 minutes. The color strength is decreased, when the fabrics are dyed more than 1 hour. The decrease in color strength may be attributed to desorption of the dye molecules as a consequence of over dyeing, if the process is carried out more than 45 minutes.

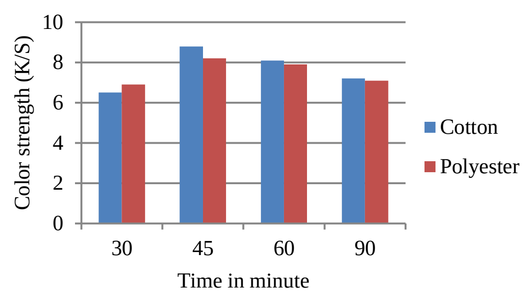

Fig. 8. Effect of time on color strength of fabrics dyed with curcumin dye (4% shade)

3.8 Effect of mordanting on color strength of cotton and polyester fabrics dyed with curcumin

The effects of mordant on relative color strength for the pre-mordanting method are shown in figure 9. It is observed that relative color strength values are higher for cotton fabrics dyed with premordanted fabric at 100°C in comparison with fabrics dyed without mordant. Since the mordant has affinity for curcumin and cotton fabric, it attracts more coloring component from dye bath to cotton fabric. But there is no significant effect of mordant on color strength of polyester fabric as shown in figure 9. The reasons are that the mordant has no affinity for polyester fabric and the fabric required more than 100°C for any chemical treatment.

It is also seen that the effect of different mordants on color strength of cotton fabric are not same. The results of alum and copper sulphate are better in terms of color strength value. But the deviations in shade tone and brilliancy among the above mentioned dyed sample cannot be measured in terms of color strength value. It is seen that different brilliant shade can be produced on cotton with curcumin dye by using the mordants.

Cotton fabric produced yellowish hue, when dyed without mordants. If alum is used, then yellowish-brown hue is produced. The samples mordanted with copper sulphate and tartaric acid produced a medium to dark yellowish-greenish and yellowish brown hue respectively. In case of polyester fabric yellowish hue is produced with or without using mordant.

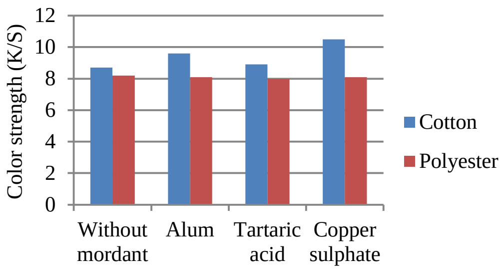

Fig. 9. Effect of pre-mordanting (2 gm/l) on color strength of fabrics (4% shade)

3.9 Result of dye exhaustion

The highest degree of exhaustion of cotton and polyester fabrics dyed in exhaust method, are determined by UV-Visible spectroscopy and the results are shown in table III.

Table III
Maximum exhaustion of curcumin dye by cotton and polyester fabrics

Fabric dyed with curcumin	Dye exhaustion (%)
Cotton without mordant	51
Cotton pre-mordanted with tartaric acid	62
Cotton pre-mordanted with $CuSO_4$	71
Cotton pre-mordanted with alum	72
Polyester without mordant	61
Polyester pre-mordanted with tartaric acid	59
Polyester pre-mordanted with alum	58
Polyester pre-mordanted with $CuSO_4$	60

The degree of exhaustion of polyester fabric dyed with curcumin dye is found lower than that of cotton fabric as shown in table III. This is because of the crystalline structure of the polyester fiber. Moreover, cotton fiber has the polar functional group in its structure. As a result, the dye exhaustion is more in case of cotton fiber than the polyester. The results also showed higher dye exhaustion is occurred on mordanted cotton than unmordanted samples but not in case of polyester.

3.10 Result of fastness properties

The wash fastness rating of cotton and polyester fabrics dyed with curcumin dye with or without mordants at the concentration of 2% and 6% (owf) is presented in table IV. The result showed that the color fastness rating of the cotton fabric without mordanting method is not good. The fastness properties have improved significantly, when different types of mordants are used before dyeing. In case of polyester fabric the fastness rating is very good and the results showed that there is no effect of mordants on fastness properties of polyester dyed fabric.

It is observed that the light fastness rating of cucumin dye is lower than wash and rubbing fastness ratings in case of both the fabrics. It is because of the chromophore group structure of the curcumin dye.

Table IV
Fastness properties of cotton and polyester fabrics dyed with curcumin dye

Dyeing Technique	Shade% (owf)	Fastness Rating									
		Wash fastness				Rubbing fastness				Light fastness (40 hours)	
		Color change		Staining		Dry rubbing		Wet rubbing			
		cotton	polyester	cotton	polyester	cotton	polyester	cotton	polyester	cotton	Polyester
Without mordant	2 6	2 1-2	4-5 4-5	2-3 2	4-5 4-5	3 2-3	4-5 4-5	2-3 2	4-5 4-5	2-3 2-3	3 2-3
With tartaric acid	2 6	2-3 2-3	4-5 4-5	3 2-3	4-5 4-5	3-4 3	4-5 4-5	3-4 3	4-5 4-5	2-3 2-3	3 2-3
With alum	2 6	2-3 2-3	4-5 4-5	3 2-3	4-5 4-5	3 2-3	4-5 4-5	3-4 3	4-5 4-5	2-3 2-3	3 2-3
With copper sulphate	2 6	3 3	4-5 4-5	3-4 3-4	4-5 4-5	3 3	4-5 4-5	3 2-3	4-5 4-5	3-4 2-3	3 2-3

4 ECONOMICAL ASPECTS IN THE DYEING OF SUPERCRITICAL CO_2 METHOD

The cost of natural dye i.e. curcumin dye is more than synthetic dye. But the auxiliaries cost and dyeing process cost of fabric by natural curcumin dye is less than synthetic dye. Moreover the natural dye is not harmful of environment, so effluent treatment plant is not necessary for such type of technology. So the technique can be applied commercially for dyeing synthetic and cellulosic fabric like PES and cotton.

5 CONCLUSION

The main goal of the work is to extract and purify the curcumin dye from the plant Curcuma Longla L., which is used to dye the cotton and polyester fabric to compare the properties of the dyed fabric. To fulfill the aim of the task, solvent extraction method is applied to extract the curcuminoid dye using soxhlet and then the curcumin dye is separated by column chromatography method. The purified dye is used to color the cotton and polyester fabric in exhaust dyeing method. Then the properties of the dyed cotton fabric are compared with the properties of dyed polyester fabric.

The result showed that the color strength values of dyed cotton fabric are higher than the dyed polyester fabric, though they are dyed in same method. The dye exhaustion percentage of the cotton fabric is also higher than polyester fabric. It is observed that mordant has a great effect to increase the color strength of the cotton fabric but not in case of polyester fabric. However, the effect of different mordants is not same on cotton fabric. Copper sulphate shows the better result than the other two.

So extraction & purification of natural dyes and their application can be a great significance in the future for the commercial and domestic dyeing industries. The dye processing cost and the cost for effluent treatment plant (ETP) are certainly lower than that of other synthetic dyes.

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