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Ecofriendly Dyeing of Cotton Fabric after Biopolymer Treatment by using Outer Skin of Onion

Mona Verma[1*], Saroj S. Jeet Singh[2], Neelam M. Rose[3] and Rajvir Singh[4]

Ph.D. scholar [1], Professor[2,3],

Deptt. of Textile And Apparel Designing,I. C. College of Home Science[1,2,3], Deptt. of biochemistry

Chaudhary Charan Singh Haryana Agricultural University, Hisar

*Corresponding Author E-mail: mona.verma35057@gmail.com

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ABSTRACT

Nature has gifted us more than 500 dye-yielding plant species. Coloring agents of these plants are derived from roots, leaves, barks, trunks or fruits. All colors of rainbow are obtained from plants. Natural dyes have better bio degradability and generally have higher compatibility with the environment. They are non toxic, non-allergic to skin, non-carcinogenic, easily available and renewable. Color fastness is the resistance of a material to change any of its color characteristics or extent of transfer of its colorants to adjacent white materials in touch. Generally light fastness, wash fastness and rub fastness are considered for textile fibers.

The outer skin of onion contains different phytochemicals which are responsible for giving colour, antimicrobial and antioxidant properties to the fabric. In this study, natural colourants were extracted using different solvents. The dye potential of the colourants obtained from the onion skin dye was evaluated by colouring cotton fabric. Bio mordanting of cotton fabric was done by using biopolymer before dyeing to improve the dyeabilty 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 perspiration fastness. From an ecological point of view, dyeing of cotton fabric with natural dye with bio mordanting may better alternative to conventional mordanting with chemicals.*

Key words: *Chitosan, Onion skin dye, Cotton, Dyeing, Natural dye*

INTRODUCTION

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. Ecological considerations are becoming important factors in the selection of consumer goods all over the world. Concerns of the environmental pollution have also created a growing interest in natural dyes utilization in instead of synthetic dyes.

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Dyes extracted from plants are interesting because of their environmental compatibility. Production and application of synthetic dyes release vast amount of waste and unfixed colorants poses serious health hazard and are disturbing the eco-balance of the nature. During the mid 1980s, more interest have been shown in the use of natural dyes as it is believed that these primitive dyes are less toxic, less allergic and more eco-friendly than the synthetic ones (Mehrabian et al., 2000). But the problems associated in dying with natural dyes are lower extraction of natural colorants the low exhaustion colours and the poor fastness properties of dyed fabrics. To overcome these problems various metallic salts are being used as mordant, which are traditionally used to improve fastness and produce different shades with the same dye (Hwang et al., 1998, Lee and Kim, 2004). But this type of compounds (metallic salts) are more complex and it will take a long time for them to complete their natural cycles and return to nature, for this reason they cause a lot of environmental pollution. So in the present research chitosan is used as mordant instead of metallic salts. 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 was also found that chitosan increase dye sorption on cotton. Chitosan is well known for its nontoxic, biocompatible and biodegradable properties. Onion (Allium cepa L.) has also been successfully grown in all parts of the world. The outer dry layers of onion bulbs, which are not edible and removed before processing, have been used all over the world as sources of natural dye that gives yellowish-brown colorants. The use of natural colorants offers promise in developing antimicrobial textiles for aesthetic, hygienic, and medical applications owing to the presence of potent highly active agents such as tannins, flavonoids, quinines carotenoids, and alkaloids in their extracts. This research paper presents dyeing of cotton fabric with onion skin after bio-mordanting of cotton and will be useful to the textile and polymer chemists engaged in development of health care bioactive textiles. In particular, it discusses recent developments in coloring textiles with different class of compounds isolated from natural colorants having antimicrobial properties, highlights current challenges, and finally concludes by providing a perspective on future research directions in this area. The availability of natural dyes needs to be increased in a sustainable manner by utilizing the by-products and wastes from agriculture and agroprocessing industries and judicious collection of forest produce. This may be supplemented by growing important dye-bearing plants on wastelands and marginal lands thus providing an alternative cash crop to cultivators. Establishment of proper characterization and certification protocols for natural dyes would definitely improve consumer confidence in natural dyed textiles and would benefit both producers and users.

MATERIAL AND METHODS

***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^2.

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 543and 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^{0}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 ^{0}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 in 1% (v/v) aqueous acetic acid solution. The treatment was applied on scoured cotton fabric by using standard concentrations of chitosan, citric acid and di-sodium hypophosphite by pad dry cure method. Further chitosan treated fabrics were dried and cured.

(d) Preparation of the dye powder and dye extract: The outer skin of red onion was collected. Then, the outer skin was cleaned to remove debris and the collected outer skins were shade dried for 25 days and after being completely dried, then 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 onion skin dye powder to 100 ml distilled water. The mixture was heated at 100ºC 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 onion skin dye 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.

Ethanolic extraction: Onion skin dye 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.

(e) Phytochemical analysis of the dye extract:

Phenol: Equal volume of each and $FeCl_3$ solution (which is prepared by dissolving 135.2g of $FeCl_3.6H_2O$ in distilled water containing 20 ml of conc. HCl dilute to 1 litre) are added together. Adeep 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% $FeCl_3$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. H_2SO_4 indicated the presence of flavonoids. ***Terpenoids:*** Extract (5 mL) was treated with 5 mL $CHCl_3$ with few drops of conc. H_2SO_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 saponins. ***Reducing sugar:*** 1 ml of extract was added 5to 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 60-100ºC 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. The colour strength expressed as K/S values was assessed by applying the Kubelka Munk equation:

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

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

(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 abrics: 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 60^0 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.

RESULTS

Preliminary Phytochemical Screening: The preliminary phytochemical tests were performed for testing different chemical groups present in extracts. Phytochemicals are non-nutritive plant chemicals that have protective or disease preventive properties. It is well-known that plants produce these chemicals to protect themselves but recent research demonstrates that they can also protect humans against diseases. There are more than thousand known phytochemicals. These phytochemicals exhibit a wide range of pharmacological activities that include antibacterial, antifungal and antioxidant activities. In the present study the phytochemical analysis of onion skin extract obtained through extraction into different medium i.e. aqueous, ethanol, and methanol. The results of phytochemical analysis of onion skin extracts are presented in the table 1.

Table 1: Phytochemical Screening of Various Extracts of *Onion skin*

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

This table shows that the presence of cardiacgycocids, tannins, flavonoids, anthraquinone was common in the aqueous, ethanol and methanol extract. While the presence of Terenoids was same in the ethanol and methanol extract. The presence of flavonoids and anthraquinone was responsible for the yellow- brown and red colour of natural colourant(onion skin dye). The aqueous extract of onion skin showed the satisfactory presence of phytochemicals. Hence the aqueous extract of onion skin was used for the dyeing of cotton fabric after chitosan treatment.

Colour strength of onion skin dyed fabric:

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 colours are given in CIE coordinates L*, a*, b*, C* and H*. Coordinates: L*corresponding to the brightness (100 =white, 0 =black), a* to the red-green coordinate (+= red, - =green) and b* to the yellow-blue coordinate (+= yellow, - = blue), C* to vividness– dullness (100= vivid, 0 = dull).

Table 2: k/s values of fabric pretreated with biopolymer

S.No.	Natural dye	L*	a*	b*	C*	H*	k/s value
1.	**Onion skin dye**	47.94	12.80	20.93	24.53	58.52	15.16

The k/s value was 15.16 for the onion skin dyed sample with at 5%. L* value was 47.94. While the a* and b* value was positive for dyed sample which shows that the sample have redder and yellower. C* depicts the chroma value which was 24.53. The hue angle (H*) was below the 90 degree, having the positive a* and b* value for the dyed sample. It depicts the colour of the sample was brown and yellowish khaki.

Table: 3 Fastness properties of dyed fabric after chitosan pretreatment

Dye	Wash Fastness		Light Fastness Days							Perspiration Fastness				Rubbing Fastness			
										Alkaline		Acidic		Dry		Wet	
	CC	CS	1st	2nd	3rd	4th	5th	6th	7th	CC	CS	CC	CS	CC	CS	CC	CS
Onion skin dye	4/5	4/5	4/5	4/5	4/5	4/5	4	4	4	4	4	4	4	4/5	4/5	4	4

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, perpiration fastness and light fastness. Dye molecules are strongly hydrogen bonded with free amino groups of chitosan and strongly retained on the fabric. Saravanan et al. (2013) conclude 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.

CONCLUSION

The purpose of this work was to study the effect of chitosan on the dyeing properties of onion skin on cotton fabric. The results of this study are concluding that, chitosan can improve the colour intensity on cotton fabric. This may be because the chitosan provided more dye sites on the fabric surface. Chitosan treated fabric not only provide better depth of shade, also provided better fastness property.

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