

Effect of Enzymatic Treatment Sequence on The Dyeability of Cellulosic Fabrics with Natural Dyes

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Abstract:

This work focuses on the effect of enzymatic treatment sequence on the natural dye absorption by cellulosic fabrics. The extent of enzymatic treatment and the dye absorption by cellulose fabrics without seriously affecting its mechanical properties were studied..

Attempts have been made to modify the conventional enzymatic treatment sequence of cellulose fabrics, i.e. enzymatic treatment and post dyeing in separate two baths, or dyeing and post enzymatic treatment, using three natural dyes, Curcuma Turmeric, Henna, and Tea. Enzyme treatment enhanced the natural dye absorption and gave the highest results in terms of dye fastness.

The extents of bio-attacked as well as dye ability using Cellulosic fabrics with different weaving structure under investigated follows the decreasing order: **Lenin > fibran > cotton**

The differences among these substrates in weaving structure for a given enzyme treatment condition, decrease in the depth of shade, K/S, follows the descending order: **Henna dye > Tea dye > curcuma Turmeric dye**

Further reveals that the effect of the used mordents on the depth of shade, K/S, follows the descending order: **Chrome > alum > tin**

Keywords:

Cellulosic fabrics
Enzymes
treatment sequence
natural dyes

Paper received 19th July 2016, Accepted 6th September 2016 , Published 15st of October 2016

1. Introduction

Enzymes have been increasingly used in the textile industry over the decade. They have been used for desizing, scouring, polishing, washing, degumming, and bleaching as well as for dye house wastewater decoloring. As enzymes show a large variety of side chains of outer amino acids and a large 3D protein structures, It can expected that enzymes interacting with virtually any chemical agent can always be found.⁽¹⁾

In general the use of enzymes cause a reduction in water and energy consumptions, in addition they can often substitute toxic chemical agents and be recovered from the wastewater and reused, satisfying both economics requirements and environments.

Cellulase through hydrolysis reaction, removes the readily accessible surface fibrils. This yields a softer fabric hand⁽²⁾. The short duration of the treatment and low concentration of cellulose don't affect the strength properties. Enzymatic treatment in Cellulosic fabrics show a clear improvement of due uptake^(3,4)

In recent years, there has been a great growth in the use of enzymes in the wet processing of textiles, to enhance softness, upgrade surface and reduce pilling propensity, especially with increasing consumer and industrial concern for environmental issues⁽⁵⁻⁹⁾.

Of the many enzymes suitable for textile

applications, cellulose is one of the most important ones. Cellulase is used in biopolishing of cellulose fabrics to enhance their smoothness, softness and wetability⁽¹⁰⁾

The extent of enzymatic treatment is governed by many factors such as accessibility of cellulosic substrate to cellulases enzymes, confirmation of the enzyme protein as well as enzyme activity, , i.e. enzyme dosage, pH, treatment conditions, temperature, time, coexisting chemicals in the treatment bath, fabric's processing history, as well as mechanical action⁽¹¹⁻¹⁵⁾ ..

Cellulase enzyme treatment can be carried out before or after the dyeing process⁽¹⁶⁻¹⁸⁾ .

Up to the nineteenth century natural dyes were the main colorants used in textile dyeing . Most natural dyes are supplied by plants. In the absence of sufficient scientific knowledge and clearing defined procedures the coloring with natural dyes was a difficult job, and obtaining the same shade two times was difficult. The development of synthetic dyes led to higher quality and more reproducible colors. As a result, the twentieth century has seen a progressive dease cin the use of natural dyes, in the past decade however the use of natural dyes in the textile industry has been growing in the popularity because many consumers have developed a new task for natural dyes. As for color reproducibility, most consumers are now ready to accept that thoroughly natural

products can have some variations.

In many cases this naturally occurring variation is guaranteeing the uniqueness of each product. In response to this new trend, research groups around the world have increasingly reported studies on natural dyes. Examples include dyeing of cotton and jute with tea using copper, alum sulphate, or ferrous sulphate as mordant⁽¹⁹⁾, and the dyeing of jute using the selected natural dyes⁽²⁰⁾.

Accordingly, in the present work an attempt has

been made to determine what is the impact of selected enzymatic treatment sequence on the natural dye absorption by cellulose fabrics.

2. Experimental

2.1 Material:

The experimental cellulose fabrics used throughout this work are fully described in Table 1

Table 1: Fabric description of Cellulosic fabrics used throughout this work.

Material	Weave Structure	Weight/area (g/m ²)	Yarn count			Thread per cm		tensile strength (kg)		Stiffness (mg/cm ²)		Wrinkle recovery angle		Wet ability sec
			warp	Weft p1	Weft p2	ends	picks	warp	Weft	warp	Weft	warp	Weft	
Lenin	Satin4	193.5	27/1	Flax44 lea	Flax40 lea	20	21	50	86.3	76.6	79.9	84	106	3.4
Cotton	Satin4	182.5	27/1	Flax44 lea	cotton 16/1 E	20	21	50.3	55.5	69.8	80.3	98	106	7.8
Fubran	Satin4	189.5	27/1	Flax44 lea	Fubran 16/1 E	20	21	57.4	44.7	77.7	66.8	93	116	5.4

commercial grade multifunctional acid cellulases enzyme formulations namely:

Cellusoft[®] L (Novo Nordisk),

Three natural dyes namely Henna (Lawsonia inermis), Tea (Lipton Tazza CTC leaf tea powder marketed by Hindustan Lever Ltd, Bombay, and (Curcuma) and three types of mordant namely Potassium Aluminum sulphate (KAL(SO₄)-12 H₂O), Potassium di chromate (K₂CrO₇), and Tin Chloride (SnCl₂-2H₂O), were used through this work. Acetic acid, Na₂CO₃, Na₂SO₄, were of reagent grade.

2.2 Methods:

2.2.1 Sequential enzymatic treatment and post dyeing

2.2.1.1 Enzymatic treatments of cellulose fabrics:

Enzymatic treatment was carried out, using a lab scale rotary machine to the following conditions: Enzyme dosage (2% enzyme of fabric weight), liquor ratio (1:15), pH (4.5 with acetic acid), at (45) °C for (40) min.

Enzymatic treatment was then terminated by raising then the pH to 10 by Na₂CO₃. enzymatic treatment, follow by rinse thoroughly with hot and cold water, and the fabric samples then dried.

2.2.1.2 Dyeing of bio- treated cellulose fabrics:

Portion of cellulose fabric samples and un treated control were dyed, using, three natural dyes, (curcuma Turmeric), (Henna), and (Tea)

2.2.2 Sequential pre- dyeing and post enzymatic treatment

2.2.2.1 Dyeing of cellulose fabrics:

Portion of cellulose fabric samples and un treated control were dyed, using, three natural dyes, (curcuma Turmeric), (Henna), and (Tea)

2.2.2.2 Enzymatic treatments of pre-dyed cellulose fabrics:

Enzymatic treatment was carried out, using a lab scale rotary machine. to the following condition:

Enzyme dosage (2% enzyme of fabric weight), liquor ratio (1:15), pH (4.5 with (acetic acid), at (45) °C for (40) min.

Enzymatic treatment was then terminated by raising then the pH to 10 by Na₂CO₃. enzymatic treatment, follow by rinse thoroughly with hot and cold water, and the fabric samples then dried.

2.3 Testing:

Weight loss: The following equation was used to calculate the weight loss (WL):

$$WL (\%) = \frac{W_1 - W_2}{W_1} \times 100$$

Where W₁ and W₂ are the weight of the fabric before and after cellulases treatment respectively.

Tensile strength of warp (TS-warp) was determined by the strip method according to ASTM procedure D2256-66T.

Stiffness was assessed according to ASTM procedure D1388-64 (1975).

Wet ability was evaluated according to AATCC standard test No.79 (1968).

Color strength (K/S) of dyed fabrics was measured at the wave length of maximum absorbance using an automatic filter spectrophotometer. Color values of dyed samples were calculated by the Kubelka Munk equation :

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

Where K is the absorbance coefficient, S is the scattering coefficient, and R is the reflectance of the dyed samples at wave length of maximum absorption.

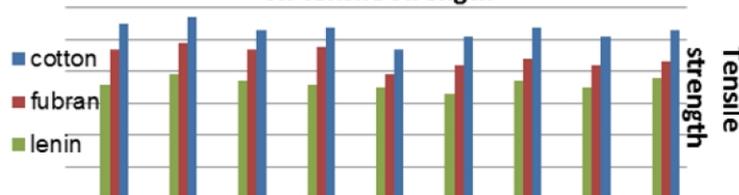
Fastness properties for colored fabrics to washing (WF), were assessed according to AATCC test methods (61-1972).

Results and discussion

With a view towards enhancing the extent of enzymatic treatment and the dye ability of cellulose fabrics without seriously affecting its mechanical properties, attempts have been made to modify the conventional enzymatic treatment sequence of cellulose fabrics, i.e. enzymatic treatment and post dyeing in separate two baths, or dyeing and post enzymatic treatment , using, three natural dyes, (curcuma Turmeric), (Henna), and (Tea)

Furthermore, the impact of treatment sequence has been evaluated. Results obtained along with appropriate discussion follow.

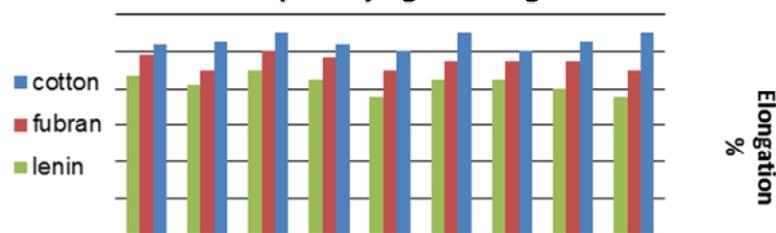
Fig(1)The effect of enzymatic treatment and postdying on tensile strength



Dye preparation: (Curcuma), (Tea), and (Henna)
 1g\15ml water, Temp: 100C Time: 60m
Mordant Bath: (KAL (So4)-12H2o) (K2Cr2O7)
 (Sncl2-2H2o) 20g\100g material Temp: 80C

Time: 60m
Dye Bath: (Curcuma), (Tea), (Henna) 30ml water\
 1g material Temp: 80C Time: 45m

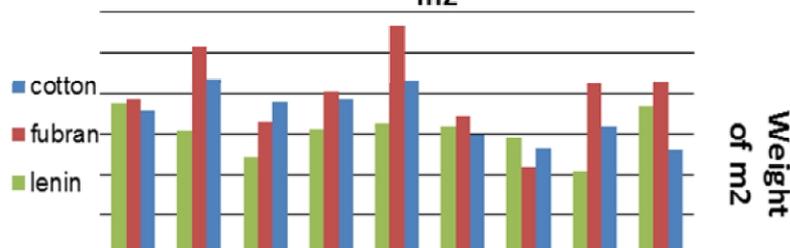
Fig(2)The effect of enzymatic treatment and post dying on Elongation



Dye preparation: (Curcuma), (Tea), and (Henna)
 1g\15ml water, Temp: 100C Time: 60m
Mordant Bath: (KAL (So4)-12H2o) (K2Cr2O7)
 (Sncl2-2H2o) 20g\100g material Temp: 80C

Time: 60m
Dye Bath: (Curcuma), (Tea), (Henna) 30ml water\
 1g material Temp: 80C Time: 45m

Fig (3) The effect of enzymatic treatment on weight m2

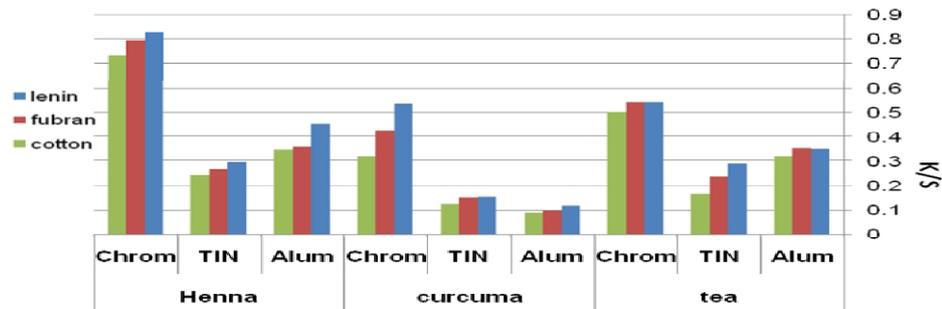


Dye preparation: (Curcuma), (Tea), and (Henna)
1g\15ml water, Temp: 100C Time: 60m
Mordant Bath: (KAL (So4)-12H2o) (K2Cr2O7)
(Sncl2-2H2o) 20g\100g material Temp: 80C

Time: 60m

Dye Bath: (Curcuma), (Tea), (Henna) 30ml water\
1g material Temp: 80C Time: 45m

Fig(4)The effect of enzymatic treatment and post dyeing on K/S



Dye preparation: (Curcuma), (Tea), and (Henna)
1g\15ml water, Temp: 100C Time: 60m
Mordant Bath: (KAL (So4)-12H2o) (K2Cr2O7)
(Sncl2-2H2o) 20g\100g material Temp: 80C
Time: 60m
Dye Bath: (Curcuma), (Tea), (Henna) 30ml water\
1g material Temp: 80C Time: 45m

1- Sequential enzymatic treatment and post dyeing

(fig1- fig 4) shows the effect of enzymatic treatment and post dyeing sequence on the performance properties and dye ability of cellulose fabrics. A perusal of the results indicate that i) Sequential enzymatic treatment and post dyeing brings about a noticeable decrease in loss the weight, tensile strength, stiffness along with a reasonable increase in their resiliency and with marginal decrease in their wettability.

This is a direct consequence of a partial enzymatic hydrolysis of the cellulosic fibers specially on the fabric surface and amorphous regions, yielding soluble products such as short- chain oligomers and glucose⁽²¹⁾, and resulting in a certain loss of tensile strength proportional to the amount of weight reduction along with elimination of hairiness on the fabric surface thereby minimizing stiffness and thickness as well as imparting a smooth surface with improved resiliency and soft handle. the extent of bio finishing, expressed as loss in weight as well as on the dye ability of the enzyme treated samples expressed as K/S. It is clear that ; the K/S value is higher, the greater the loss in weight, regardless of the used dyestuff. It is further noted in (fig1- fig 4) that the extent of bio-attacked as well as dye ability using cellulosic

fabrics with different weaving structure under investigated follows the decreasing order:

Lenin > fib ran > cotton

reflecting the differences among these substrates in⁽²²⁻²⁴⁾ in weaving structure

The enhancement in the extent of dyeing, expressed as K/S value, with loss

In weight observed for all the cellulose fabrics under investigation could be ascribed to:

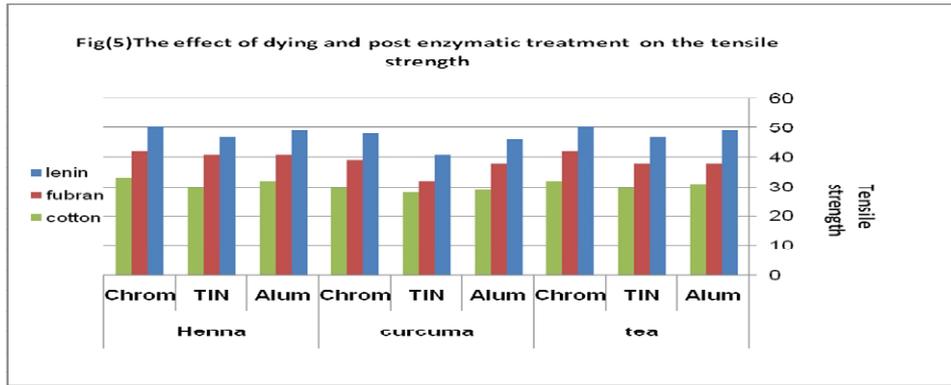
i) the bio-polishing of the fabric surface thereby decreasing the scattering coefficient (S) and, as a consequence higher K/S values can be obtained⁽²⁵⁾, and /or ii) the development of an additional accessible region to dye as a direct consequence of the enzymatic attack thereby compensating the region that was readily digested by the enzymatic hydrolysis as well as enhancing the dye-uptake⁽²⁶⁾. On the other hand, differences in dye ability of enzyme-treated fabric samples upon using, three natural dyes, (curcuma Turmeric), (Henna), and (Tea)

differences in : i) molecular size, ii) chemical nature iii) reactivity of the dye, iv) number of reactive groups, v) probable attachment sites, and vi) extent of exhaustion and fixation⁽²⁷⁻²⁸⁾ (iv) for a given enzyme treatment condition, decrease in the depth of shade, K/S, follows the descending order:

Henna dye > Tea dye > curcuma Turmeric dye

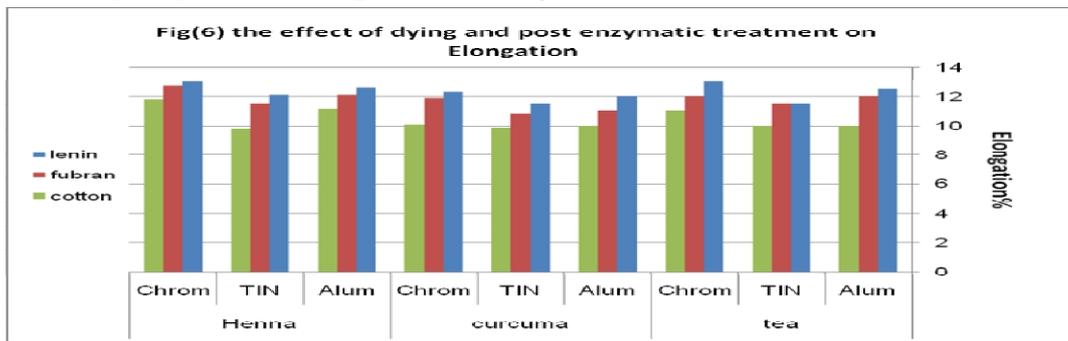
Further, (fig1- fig 4) reveals that the effect of the used mordants on the the depth of shade, K/S, follows the descending order:

Chrome > alum > tin



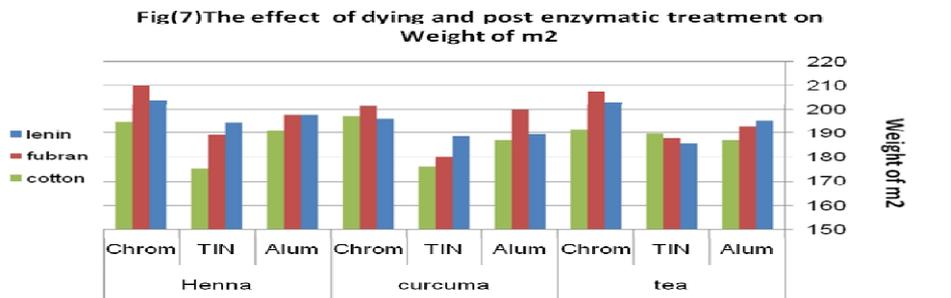
Dye preparation: (Curcuma), (Tea), and (Henna)
 1g\15ml water, Temp: 100C Time: 60m
Mordant Bath: (KAL (So4)-12H2o) (K2Cr2O7)
 (Sncl2-2H2o) 20g\100g material Temp: 80C

Time: 60m
Dye Bath: (Curcuma), (Tea), (Henna) 30ml water\
 1g material Temp: 80C Time: 45m



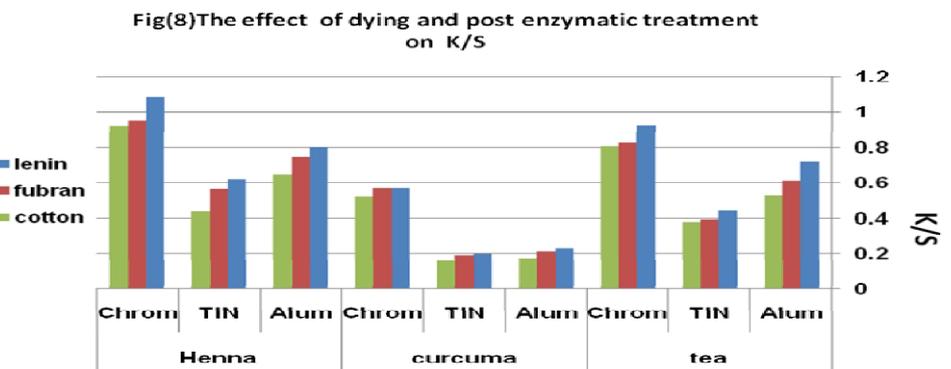
Dye preparation: (Curcuma), (Tea), and (Henna)
 1g\15ml water, Temp: 100C Time: 60m
Mordant Bath: (KAL (So4)-12H2o) (K2Cr2O7)
 (Sncl2-2H2o) 20g\100g material Temp: 80C

Time: 60m
Dye Bath: (Curcuma), (Tea), (Henna) 30ml water\
 1g material Temp: 80C Time: 45m



Dye preparation: (Curcuma), (Tea), and (Henna)
 1g\15ml water, Temp: 100C Time: 60m
Mordant Bath: (KAL (So4)-12H2o) (K2Cr2O7)
 (Sncl2-2H2o) 20g\100g material Temp: 80C

Time: 60m
Dye Bath: (Curcuma), (Tea), (Henna) 30ml water\
 1g material Temp: 80C Time: 45m



Dye preparation: (Curcuma), (Tea), and (Henna) 1g\15ml water, Temp: 100C Time: 60m

Mordant Bath: (KAL (So4)-12H2o) (K2Cr2O7) (Sncl2-2H2o) 20g\100g material Temp: 80C Time: 60m

Dye Bath: (Curcuma), (Tea), (Henna) 30ml water\ 1g material Temp: 80C Time: 45m

2 - Sequential pre- dyeing and post enzymatic treatment:

(fig 5- fig 8) shows the effect of pre dyeing different cotton fabrics with various dyestuffs on the extent of subsequent enzyme treatment the results obtained reveals that:

i) in the reference treatment, i.e. in absence of Cellusoft[®] L, a weight loss is obtained regardless of the treated substrate, attributable to the mechanical action,⁽³⁰⁾.

ii) treatment with 2% enzyme brings about a loss in weight and tensile strength, along with a decrease in stiffness of the bio-treated samples attributable to the enzymatic hydrolysis of cellulose specially in the amorphous regio⁽³¹⁻³²⁾.

iii) treatment with Cellusoft[®] L (2% owf) reduce the depth of shade, K/S improves washing fastness of all dyeing under investigation, probable due to the adsorption and removal of surface dyes that are weakly adsorbed on the substrate, e.g. direct dyeing, and / or partial hydrolysis of the surface of the fibers or projecting fuzzes that include the dye as in case of reactive dyeing⁽³³⁾,

iv) the extent of enzymatic attack, expressed as the changes in the aforementioned properties, is governed by the nature of pre-existing dye, i.e. molecular dye size, dye/ fiber association, reactive groups and aggregation of dye molecules, as well as the construction of cotton fabric.

v) for a given enzyme treatment condition, decrease in the depth of shade, K/S, follows the descending order:

Henna dye > Tea dye > curcuma Turmeric dye reflecting the ionic nature of the dye as well as the nature of dye fiber interaction regardless of the depth of shade, and vi) the enhancement in wrinkle recovery angle of enzyme-treated dyed cotton fabrics may be due to elimination of hairiness and minimizing of thickness thereby imparting a smooth fabric surface with improved resiliency

It is further noted in (fig 5- fig 8) that the extent of bio-attacked as well as dye ability using cotton fabrics with different weaving structure under investigated follows the decreasing order:

Lenin > fib ran > cotton

reflecting the differences among these substrates in fabric construction.

Further, (fig 5- fig 8) reveals that the effect of the used mordents on the the depth of shade, K/S, follows the descending order:

Chrome >alum>tin

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