Optimizing Color Fading Effect of Cotton Denim Fabric by Enzyme Treatment

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ABSTRACT: In this article, the effect of enzyme treatment using neutral cellulase on the color fading property of cotton denim fabric was studied. Four enzyme processing parameters namely treatment temperature, treatment time, pH value, and agitation were considered. To investigate the optimum condition for the enzyme treatment, an orthogonal analysis was used and, based on the K/S summation value (K/S Sum), the optimum condition for enzyme treatment in this study was treatment temperature = 50°C; treatment time = 30 min; pH value = 8; and agitation = 50 steel balls (simulated mild agitation) for the best color fading achievement with desired worn and aged effect. Meanwhile, the level of importance based on the orthogonal analysis was in the order: treatment temperature > treatment time > agitation > pH and the effect of each processing factors was also discussed. In addition, other properties like CIE Lab values, weight loss, and color fastness to laundering and crocking were also evaluated. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 120: 3596–3603, 2011

Key words: enzymes; fibers; surface modification; surfaces

INTRODUCTION

Denim garment plays an important role in fashion and textiles industry. Denim jeans provide durability and slightly worn look for fashionable appearance which is the reason why denim jeans are welcomed by most people over the world. However, it also gives wearers heavy and rigid sensations due to the sturdy construction of twill weave and the finishing processes involved. Nowadays, consumers are more concerned with garment comfortable attributes such as lightweight, smoothness, and softness.[1] There are a number of methods for denim garment for achieving smooth and soft hand feel[1,2] and among these methods, enzyme treatment produces novel fashionable stone-washing appearance.[2–4] It can also improve fabric quality such as softer hand feel and smoother surface by removing protruding fibers on fabric surface.

Recently, cellulase, as an enzyme, can be used for treating denim fabric biologically for getting desired worn and aged effect without going through conventional denim washing with pumice stone.[5,6] The action of cellulases and mechanical agitation, simultaneously or sequentially, will abrade fiber surface, releasing cotton fiber and causing defrillation at the surface. In the denim fabrics, due to the enzyme abrasion, dye or dye aggregates with cotton will be released from yarns resulting contrasts in the blue color. The fibrillation produced during the ageing process is a result of the synergistic action of cellulases and mechanical action, and therefore, the aged look is reproduced by less abrasive action.[7–9] Acid and neutral cellulases are available in the market in which the acid cellulase is commonly used.[10] However, acid cellulase may cause degradation to the cotton fiber because the cellulase is being applied under acidic condition (operation pH at about 5.5).[11] To get minimized fiber degradation, in this paper, denim fabric was treated with neutral cellulase enzyme in recommended concentration and liquor ratio. There were four factors varied in the cellulase application to achieve different treatment conditions and desired worn and aged effects without much altering the original indigo blue color, they are: treatment temperature, treatment time, pH and agitation and each of these factors has three levels. To determine an optimum condition of neutral cellulase enzyme treatment of the denims, experimental design method was used for obtaining the optimum condition for the cellulase enzyme treatment.[12,13] The color yield was used as the property for determining the optimum condition.

EXPERIMENTAL

Denim fabric specification

Hundred percent desized 3/1 right hand twill cotton denim fabric with fabric weight 382 g/m^2 was used.

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TABLE I
Factors and Levels Used in Orthogonal Analysis

Factor level	Treatment temperature (°C)	Treatment time (min)	pH	Agitation (No. steel balls)
I	50	30	6	0
II	55	60	7	50
III	60	90	8	100

The warp and weft densities were 25 ends/cm and 20 picks/cm, respectively. The yarn count of the warp and weft yarns were 6.57Ne and 10.28Ne, respectively.

Enzyme treatment on denim fabric

Enzyme treatment was carried out by using a Launder-Ometer (Atlas Material Testing Technology LLC, Chicago) which is a thermostatically controlled water bath system with stainless steel containers could be mounted. The containers were mounted on a horizontal shaft and rotated at speed of 40 to 45 rpm. Mechanical agitation was simulated by using stainless steel balls with size of 6 mm diameter. Four pieces of denim fabric specimens were cut into square of 22 cm × 22 cm (about 20 g in weight) and the specimens were conditioned under standard atmospheric pressure at 65% ± 2% relative humidity and 20 ± 2°C for at least 24 h before enzyme treatment.

A neutral cellulase enzyme (Lava Cell NNM, supplier: DyStar) with activity 130 EGU/g [1 EGU/g, (endo-glucanase activity) unit is defined as the amount of enzyme required to reduce the viscosity of a solution of carboxymethylcellulose to one-half] in powder form was used for treating the denim fabric. A stock solution of 0.1% enzyme was prepared. The denim fabric was treated with 0.1% on weight of the fiber (owf) of enzyme with a liquor ratio of 50 : 1 and 20 mL of the stock enzyme solution was used for the experiments. In preparing the required pH condition for enzyme treatment, buffer solution [mixture of 1*M* dipotassium hydrogen phosphate (K_2HPO_4) and 1*M* potassium dihydrogen phosphate (KH_2PO_4)] was added into the liquor with thorough stirring for keeping the pH values at 6, 7, and 8. The pH values were checked by electronic pH meter.

To determine an optimum condition of neutral cellulase washing of the denims, a $L_9(3)^4$ orthogonal analysis was used. The factors and levels used in the orthogonal analysis were shown in Table I and nine test runs were required as shown in Table II.[12,13] The addition of 0, 50, and 100 steel balls (6 mm) in enzyme treatment aimed for simulating the treatment condition with no agitation, mild agitation, and vigorous agitation correspondingly.

After enzyme treatment, the denim fabric specimens were rinsed with tap water to lower the temperature quickly and wash away abraded fibers attached on specimens. The specimens were then treated with water at 80°C to deactivate the enzyme action. After that, the specimens were squeezed to remove excessive water and dried in an oven at temperature 70°C. After drying for two hours in oven, the specimens were placed for conditioning under standard atmospheric pressure at 65 ± 2% relative humidity and 20 ± 2°C for at least 24 h before further evaluations.

Measurement of color

The color characteristics of the denim fabric samples were measured by a Macbeth Color Eye 7000A Spectrophotometer. The condition for measurement was set under specular excluded with large aperture. The fabric was folded twice for ensuring opacity and measured twice. The color yield expressed as *K/S* value, which could be obtained from the reflectance curve, ranging from the visible spectrum wavelength of 400 nm to 700 nm with 10 nm interval within the visible spectrum was calculated. The *K/S* value was calculated according to Eq. (1) and finally a summation of individual *K/S* value (i.e., *K/S* Sum) over the visible spectrum wavelength was calculated. The higher the *K/S* Sum value, the better color yield will be.[12–14]

$$K/S = (1 - R)^2/2R \tag{1}$$

where K = absorption coefficient, depending on the concentration of colorant; S = scattering coefficient, caused by the dyed substrate; R = reflectance of the colored sample.

In addition, the CIE $L^*a^*b^*$ values were measured and ΔE (total color difference) was obtained.

Measurement of weight loss

The weight of denim fabric samples before and after enzyme treatment was measured by a weight meter (Shimadzu BX300 m) after conditioning at 20 ± 2°C and relative humidity of 65 ± 2% for 24 h.

TABLE II
Enzyme Treatment Conditions for Denim Fabric Under a L9(3)4 Orthogonal Analysis

Test run	Treatment temperature (°C)	Treatment time (min)	pH	Agitation (No. steel balls)
1	50	30	6	0
2	50	60	7	50
3	50	90	8	100
4	55	30	7	100
5	55	60	8	0
6	55	90	6	50
7	60	30	8	50
8	60	60	6	100
9	60	90	7	0

TABLE III
Orthogonal Table for the Optimization of Cellulase Treatment

Test Run	Factors: Temperature (°C)	Agitation	Time (min)	pH	K/S sum
1	50	0	30	6	648.75
2	50	50	60	7	637.67
3	50	100	90	8	632.05
4	55	100	30	7	594.52
5	55	0	60	8	522.81
6	55	50	90	6	552.53
7	60	50	30	8	676.10
8	60	100	60	6	620.87
9	60	0	90	7	572.81
ΣI	**1918.47**	1744.37	**1919.37**	1822.15	
ΣII	1669.86	**1866.30**	1781.35	1805.00	
ΣIII	1869.78	1847.44	1757.39	**1830.96**	
Difference	248.61	121.93	161.98	25.96	

The figures in **bold** are those with the greatest value in the levels of different factors used.

Evaluation of color fastness

The color fastness to laundering and crocking of the denim fabrics were assessed by AATCC Test Method 8 and AATCC Test Method 61, respectively.

RESULTS AND DISCUSSIONS

Optimum condition for enzyme treatment

The optimum condition for enzyme treatment on denim fabric could be obtained by means of the orthogonal analysis which provides a convenient way of determining the optimum condition and level of importance of different factors in a treatment process.[12,13,15,16] The evaluation used for analyzing the orthogonal analysis is the K/S value. Although K/S values were measured from wavelength 400 nm to 700 nm with 10 nm interval within the visible spectrum, the summation of K/S value for the visible spectrum (i.e., K/S Sum) was used for analysis. The quantity of K/S Sum value is linearly related to the concentration of the colorant in the substrate. In the enzyme treatment, K/S Sum value could reflect the color retained after enzyme treatment and the results are summarized in Table III. The higher K/S Sum value, the higher amount of dye retained in the denim fabrics after enzyme treatment.[17] The dye is preserved in fabric instead of being washed away as waste during enzyme treatment. It will be in turn a saving in dye and cleaner sewage.

As the level of parameter which contributes to give higher K/S Sum value will be the favorable level of that parameter in condition for the enzyme treatment. Therefore, the optimum condition for the denim fabric specimens was treatment temperature = 50°C; treatment time = 30 min; pH value = 8, and agitation = 50 steel balls (simulated mild agitation). The level of importance based on the orthogonal analysis was in the order: treatment temperature > treatment time > agitation > pH.

Effect of treatment temperature

Temperature is the most important factor on enzyme treatment according to orthogonal analysis. Generally speaking, enzyme reaction increases with temperature but it is only activated within a temperature range in which the enzyme structure maintains stable and unchanged. Beyond this optimum range, the enzyme activity will decrease sharply as the protein structure of enzyme is tangled through thermal agitation. The effect of temperature on K/S Sum values is shown in Table III. The temperatures chosen for enzyme treatment were within the optimum temperature range of the neutral cellulase used. In fact, there is no great difference of K/S Sum values between these temperatures as the enzyme activity will only have great change over 10°C variation. However, there is a trend of decrease of K/S Sum values when temperature increases from 50 to 60°C. It is because the enzyme reaction is activated by higher temperature within optimum temperature range, thus more surface fibers on denim fabrics are hydrolyzed by cellulase[6] and the weaken fibers are further removed by abrasion of fabrics and mechanical agitation.[18,19] The indigo dye particles are also removed with the cotton fibers. As the quantity of K/S Sum value is linearly related more or less to the concentration of the colorant in the substrate (i.e., shade), thus an assumption could be made that there is loss of dye from denim fabric during enzyme treatment. The lower temperature used as 50°C has the highest K/S Sum value which shows more dye was retained in the cellulase treated denim fabric.

Effect of agitation

The level of mechanical action is altered by varying number of steel balls applied in enzyme treatment. By increasing the number of steel balls applied, the abrasion of the fabric surface will increase which creates more accessible sites for enzyme attack. The effect of agitation on K/S Sum values is shown in Table III. Since the K/S Sum value is linearly related to the concentration of colorant in the substrate. The lower K/S Sum value of specimen means a lighter shade is resulted. From Table III, enzyme treatment with no agitation (0 steel balls) has the lowest K/S Sum, followed by vigorous agitation (100 steel balls) and then mild agitation (50 steel balls). Therefore, specimen treated under no agitation has lighter shade than that treated with mild and vigorous agitation. It is because the cellulose fibers are weaken after treating with enzyme alone and are not well removed by mechanical agitation.[11,19] The surface of fabric becomes more hairy and forming a layer of fuzz on surface.[3] For specimen treated under vigorous agitation, a biopolishing effect is obtained. The weaken fibers are well removed by strong mechanical agitation by 100 steel balls. Thus, a cleaner and smoother surface is resulted which produce a lighter shade of fabric as dye particles are also removed with cellulose fibers. For specimen treated with mild agitation (50 steel balls), the situation is between zero agitation and vigorous agitation. Mild agitation is not strong enough to remove as many weaken surface fibers as vigorous agitation can, thus rather uneven surface is produced under mild agitation. The uneven surface causes higher light diffusion on surface and thus darker shade of specimen is resulted as they achieve lower reflectance on surface.

Effect of treatment time

Longer enzyme treatment time will prolong enzymatic degradation of cellulose and the time for further abrasion. The effect of treatment time on *K/S* Sum values is shown in Table III. From Table III, *K/S* Sum value decreases continuously with the increase of treatment time of cellulase treatment. As the quantity of *K/S* Sum value is linearly related more or less to the concentration of the colorant in the substrate, it shows that there is a decrease in color depth of cellulase treated denim fabrics. The decreased color depth with less time is mainly due to desorption of dye particles that are weakly adsorbed on fabric. In addition, the decreased color depth with more time is also due to the fuzziness of fabric caused by prolong time of cellulase treatment. With a longer treatment time, cellulase effectively hydrolyzes fragments of cotton fibrils[6] and thus the fabric would be less fuzzy than the original.[11] Therefore, shorter treatment time with enzyme can preserve desired color depth in denim fabrics. Thus, cellulase treatment with 30 min is optimum.

Effect of pH

Enzyme performs its maximal activity at a particular range of pH values. Deviation of the optimum pH range will alter the internal structure of enzyme due to the electrostatic interactions within the enzyme. Thus, the enzyme will no longer perform its normal function. In the study, the pH values chosen for enzyme treatment are within the optimum pH range of neutral enzyme which is pH 6, 7, and 8. The effect of pH on *K/S* Sum in enzyme treatment is shown in Table III. From Table III, there is no great difference in the *K/S* Sum values from the pH 6 to 8 unless pH 8 has the highest *K/S* Sum value among them. The effect of pH in the study has the least level of importance from the orthogonal analysis because the pH values are within the optimum pH range for neutral cellulase treatment.

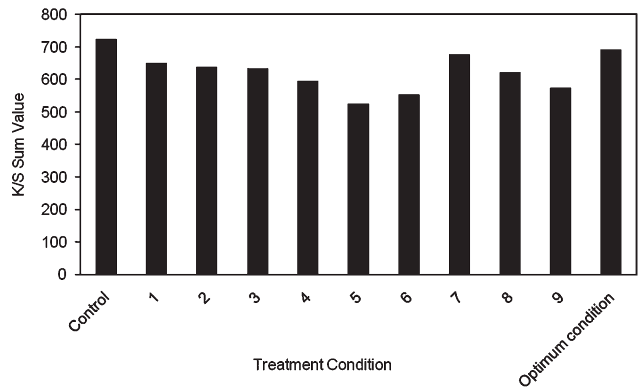

Figure 1 Color yield of denim fabrics treated under the different treatment conditions.

Effect of optimum condition for cellulase treatment

Orthogonal analysis is a useful and simple technique for analyzing the process variables or factors involved in a production process. Previous researches[12,13,15,16] showed that it could provide a simple and convenient way for finding out the optimum condition and the level of importance of different factors in a production process.

After considering the results obtained from the orthogonal analysis as shown in Table III, it was concluded that all four factors used namely treatment temperature, treatment time, pH value and agitation could affect the *K/S* Sum value by contributing different effect on the final color yield. However, the level of importance based on the orthogonal analysis was in the order of treatment temperature > treatment time > agitation > pH. Based on the results of the orthogonal analysis, the optimum condition obtained for the neutral cellulase treatment was concluded as treatment temperature = 50°C; treatment time = 30 min; pH value = 8, and agitation = 50 steel balls (simulated mild agitation). To verify the accuracy of the optimum condition, further experiment was conducted using the optimum condition for treating the denim fabrics and the result were compared as shown in Figure 1.

The results shown in Figure 1 demonstrates clearly that the denim fabric treated under the optimum condition achieved and retained the best color yield when compared with those conditions (1–9) shown in Table III with desired worn and aged effect. The optimum condition could achieve *K/S* Sum value of 690.13 which is about 5% reduction in the color yield when compared with the *K/S* Sum

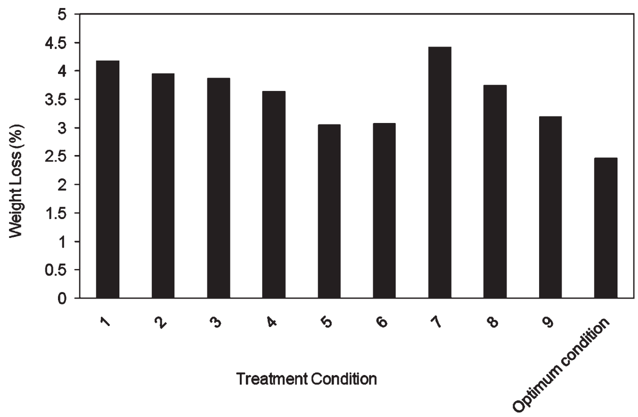

Figure 2 Weight loss yield of denim fabrics treated under the different treatment conditions.

value of the control fabric (724.64). Inside the cellulosic fiber, it consists of the crystalline and amorphorous regions. In the crystalline region, the molecular chains are well organize and closely packed, the size of cellulase is not small enough to enter this region to initiate hydrolysis action.[20] The cellulase is then entering the amorphous region easily where the space is sufficient for the cellulase to enter and consequently hydrolysis is taking place here.[3] After hydrolysis, some of the cellulosic fibers together with the dye in the amorphous region would be removed. The amount of dye in the fiber would then be reduced resulting in the reduction of K/S Sum value.

Weight loss

The action of enzyme and mechanical agitation will abrade fiber surface and cotton fiber would be released resulting in weight reduction. Figure 2 illustrates the weight loss of denim fabrics under different treatment conditions as shown in Table II. After enzyme treatment, weight loss was observed but the degree of weight loss was various with different treatment conditions. However, minimum weight loss was noted for the denim fabric treated under optimum condition. When Figures 1 and 2 were compared, it was found that the effect of cellulase treatment on the color yield and weight loss were in the same trend. In enzyme treatment of cellulosic materials, weight loss due to hydrolysis could not be avoided but a high degree of hydrolysis is not expected because it is also correlated with the strength of the cellulosic materials.[15] Although some hydrolysis is required to obtain the desired effect of color removal, this study indicates that by selecting an optimum condition, the cellulase action can be achieved with desired color fading effect with low degree of hydrolysis with desired worn and aged effect.

Color fastness to laundering

Table IV shows the results of color fastness to laundering of different fabric samples. It was noted that all fabric samples treated with enzyme treatment could achieve same rating in color change and staining. In accordance with the ASTM D 6554: Standard Performance Specification for 100% Cotton Denim Fabrics, the minimum rating of color change and staining of denim fabric subjected to the laundering color fastness test should be 2 and 2, respectively. Obviously, both control and enzyme treated denim fabrics reached the industrial requirements. When the ratings of the enzyme treated samples were compared with the control sample, slightly improvements were noted in the staining of the acrylic, polyester and nylon fibers present in the multifiber fabric. During the enzyme treatment, the surface fiber together with the unfixed dye in the fiber surface could be removed as a result; no unfixed dye would be removed from the denim fabric during the laundering color fastness test.

TABLE IV
Colour Fastness to Laundering of Different Fabric Samples

Treatment condition	Colour change	Staining					
		Wool	Acrylic	Polyester	Nylon	Cotton	Acetate Rayon
Control	4–5	4–5	4	4	2–3	4–5	4–5
1	4–5	4–5	4–5	4–5	3	4–5	4–5
2	4–5	4–5	4–5	4–5	3	4–5	4–5
3	4–5	4–5	4–5	4–5	3	4–5	4–5
4	4–5	4–5	4–5	4–5	3	4–5	4–5
5	4–5	4–5	4–5	4–5	3	4–5	4–5
6	4–5	4–5	4–5	4–5	3	4–5	4–5
7	4–5	4–5	4–5	4–5	3	4–5	4–5
8	4–5	4–5	4–5	4–5	3	4–5	4–5
9	4–5	4–5	4–5	4–5	3	4–5	4–5
Optimum condition	4–5	4–5	4–5	4–5	3	4–5	4–5

TABLE V
Colour Fastness to Crocking of Different Fabric Samples

Treatment condition	Warp		Weft	
	Dry	Wet	Dry	Wet
Control	3	1–2	3	1–2
1	3–4	2	3–4	2
2	3–4	2	3–4	2
3	3–4	2	3–4	2
4	4	2	3–4	2
5	3–4	2	3–4	2
6	3–4	2	3–4	2
7	3–4	2	3–4	2
8	3–4	2	3–4	2
9	3–4	2	3–4	2
Optimum condition	4	2	4	2

Color fastness to crocking

The results of color fastness to crocking of denim fabrics treated under various cellulase treatment conditions are shown in Table V. According to the ASTM D 6554: Standard Performance Specification for 100% Cotton Denim Fabrics, the minimum rating of dry and wet staining of denim fabric subjected to the crocking fastness test should be 3 and 1–2, respectively. Although the control denim fabric reached the industrial requirement, the various enzyme treatments further improved the crocking fastness results by half grade. For the denim fabric treated under the optimum condition, the best crocking fastness results were obtained. During the enzyme treatment, the fabric surface would be abraded with the cellulase and the mechanic agitation. The surface fiber and the unfixed dye in the fiber surface would be removed subsequently and also a smooth surface could be obtained. Therefore, during the crocking test, no further surface fiber and unfixed dye could be rubbed out from the fabric surface resulting in improved crocking fastness results.

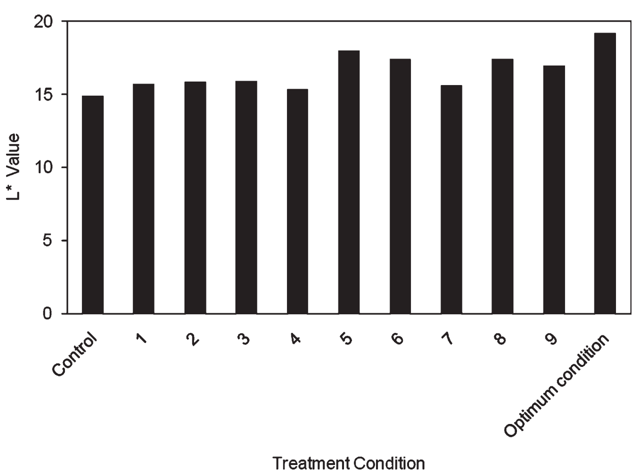

Figure 3 L^* value of denim fabrics treated under the different treatment conditions.

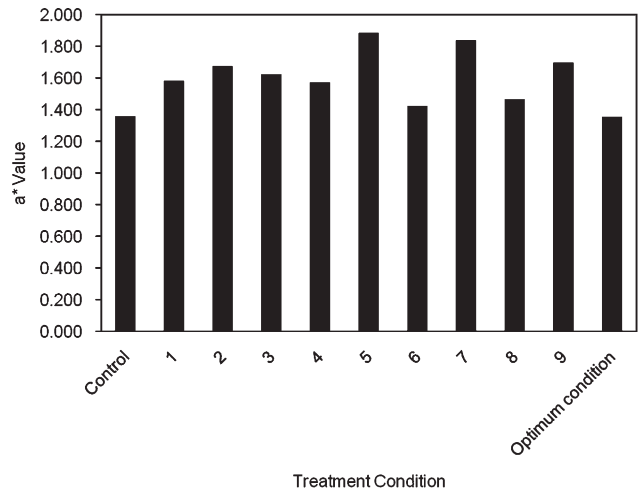

Figure 4 a^* value of denim fabrics treated under the different treatment conditions.

CIE L^* value

Figure 3 shows the L^* values of denim fabrics treated under the different treatment conditions. L^* value indicates the lightness of the sample. The higher L^* value is, the lighter the sample is. It is a fact that after the cellulase treatment, the L^* values increased accordingly. The lightness depends on greatly on the surface texture of the sample. There are two factors contributing the lightness of cellulosic materials influenced by cellulase action. The first factor is that the cellulase action gives indigo-dyed cotton fabric an aged appearance (i.e., biostoning) while the second factor is to give cotton fabric a revived appearance by removing the surface fiber (i.e., depilling or biopolishing). Both factors contribute to change the lightness of the cellulase treated denim fabric. During the action of cellulase and mechanical agitation, the depilling or biopolishing effect occurs at the fabric surface and consequently the surface fibers were removed.[11] At the same time,

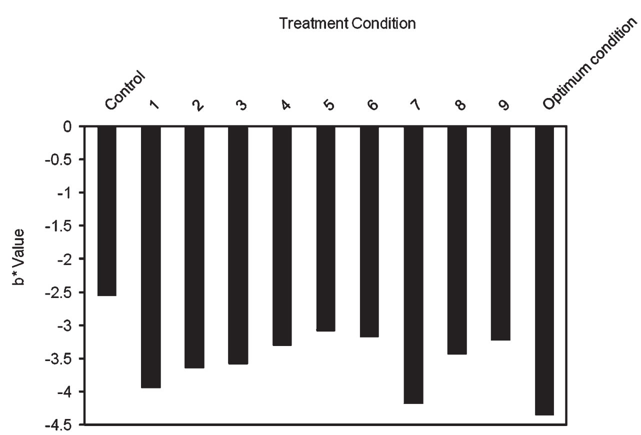

Figure 5 b^* value of denim fabrics treated under the different treatment conditions.

TABLE VI
ΔE of Different Fabric Samples

Sample	1	2	3	4	5	6	7	8	9	Optimum condition
ΔE	1.63	1.48	1.48	0.90	3.21	2.62	1.83	2.72	2.23	4.68

the action of cellulase on the fiber surface would increase the smoothness and evenness microscopically. The smoother surface would enhance the regular reflection of light from the fabric surface and therefore the lightness was increased, although the *K/S* Sum values were decreased after the enzyme treatment.

CIE *a** value

The *a** value represents the redness and greenness of a substrate, positive value indicates a redder shade while negative value shows a greener shades of the samples. Figure 4 shows the *a** value of denim fabrics treated under various conditions. The *a** values of control denim fabric and denim fabric treated under optimum condition are 1.36 and 1.35, respectively, which indicates that no significant change was noted after enzyme treatment under optimum condition. However, when denim fabrics were treated with other conditions, the *a** value increased which means that color of the denim fabrics turned redder.

CIE *b** value

*b** value describes the yellowness and blueness of a sample. The more positive value of *b** means the greater yellowish of the sample. However, the negative the value of *b** is, the bluish the sample is. Figure 5 shows the *b** value of different denim fabric samples. It was noted that after enzyme treatment, the denim fabrics have a bluer shade than the control specimens. It was believed that during the enzyme treatment, hydrolysis of the cellulosic fiber was taken place. The blue indigo dyes originally held in the fiber would no longer be held by the fiber and entered the treatment bath after hydrolysis. Consequently, the blue indigo dyes in the treatment bath would stain and adhere on the fabric surface resulting in a bluer shade.

Δ*E* value

Table VI shows the Δ*E* values of different enzyme treated denim fabrics. Δ*E* value contains the information of color depth, shade and the hue of a sample. Δ*E* value is calculated by using the *L*, *a*, and *b* values with the equation $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ where ΔL^*, Δa^*, and Δb^* values were the difference between *L**, *a**, *b** values of a pair of color standard and sample. The larger the Δ*E* value, the greater will be the color difference between this pair of color standard and sample. It was noted after the enzyme treatment, color difference was appeared between the control and the treated denim fabrics. This color difference was expected because after the enzyme treatment, changes in *L**, *a**, and *b** values were noted and these three values would contribute the total color difference values. When compared, the denim samples treated under the optimum condition results in the largest Δ*E* value.

CONCLUSIONS

With the use of orthogonal analysis, the optimum condition for neutral cellulase enzyme treatment for denim fabric was obtained by considering the *K/S* summation (*K/S* Sum) value. The optimum condition was treatment temperature = 50°C; treatment time = 30 min; pH value = 8, and agitation = 50 steel balls (simulated mild agitation). The level of importance based on the orthogonal analysis was in the order: treatment temperature > treatment time > agitation > pH. After obtaining the optimum condition, the denim fabric was treated under this condition for verifying the process condition. It was found that the denim fabrics treated under the optimum condition retained the best color yield with desired worn and aged effect. Meanwhile, other properties such as weight loss, colorfastness to laundering and crocking reached acceptable results. Also after the enzyme treatment, the CIE *L*a*b** values were changed differently with the greater Δ*E* for the fabric sample treated under optimum condition.

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