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Basic and Reactive-Dyeable Polyester Fabrics Using Lipase Enzymes

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Abstract

Plain weave polyester fabrics were treated with lipase enzymes; namely lipase Type II and lipolase 100L-EX enzyme at different reaction conditions to enhance its dyeability with basic dye. Fourier Transform Infrared spectroscopic investigation proved the creation of carboxylic as well as hydroxyl groups as a result of controlled rupture of ester links along polyethylene terephthalate macromolecule. This led to improved dyeability with the cationic dye "Basic Red 18" as well as reactive dye "Reactive Red 120". Physical as well as mechanical properties of the treated fabrics; namely wettability, moisture regain, and tensile properties, were assessed compared to those of the untreated one. No significant deteriorative action of the lipolase enzyme, under the used reaction conditions, was detected by scanning electron microscopy.

Keywords: Polyester; Fabrics; Lipolase; Enzyme; Basic; Reactive; Dyes; Dyeing

Introduction

The potential of using green technology in textile industry becomes mandatory within the last few decades, presumably due to the high cost of energy required for waste water treatment as well as the hazardous effect of the aggressive chemicals discharged with industrial effluents which harm marine organisms as well as man-kind through food chain. Bio-preparation and bio-finishing of textiles were investigated by many authors. Most of these investigations concerned with bio-modification of natural fibers; namely cotton, flax, and wool [1-7]. Few reports focused on synthetic fibers such as polyester [8-11].

Being the king of fibers, which have various applications in clothing and non-clothing applications, the annual production of polyester fabrics within the last three decades inclined by more than three folds [12]. Polyester fibres have taken the major position in textiles all over the world although they have many drawbacks; namely (a) low moisture regain (0.4%), (b) the fibres has a tendency to accumulate static electricity, (c) the cloth made up of polyester fibres picks up more soil during wear and it also difficult to clean during washing, (d) the polyester garments from pills and thus, the appearance of a garment is spoiled, (e) the polyester fibre is flammable [13]. Thus, it has been suggested that surface modifications can have an effect on hand, thermal properties, permeability, and hydrophilicity. Numerous research papers and patents are available and considerable amount of research works is in progress on chemical modification of partial hydrolysis of polyester fibres to overcome their disadvantages [14-18] according to the following mechanism:

$$R - COO - R' + H_2O \; R - COOH + R' - OH$$

Kantouch *et al.* utilized Lipase type II and lipolase 100 L-Type EX in removing the hydrophobic lipid barrier on the surface of wool to enhance its hydrophilicity and dyeability [2]. Lipases are known to catalyze the hydrolysis of esters in aqueous solutions and the synthesis of esters in non-aqueous solutions [19]. They also produce hydroxyl and carboxylic groups through the hydrolysis of ester linkages in poly (ethylene terephthalate) [20].

The main objective of this study was to explore the possibility of enhancement the dyeability of polyester fabrics by basic and reactive dyes using lipases enzyme. To the end, mechanism for microwave affecting the absorption properties of dyestuff on to the polyester fiber surface from dye path was discussed.

Experimental

Material

Two lipase enzymes (E.C. 3.1.1.3) were used in this study. Lipolase 100L-EX (from *Thermomyces lanoginosus* solution) and lipase type II (from porcine pancreases), with declared activity 100,000 U/g and 100-500 U/mg respectively, were purchased from Sigma-Aldrich, USA.

Dyes

Basic Red 18 and Reactive Red 120 were used in this study. The structural formulae of these dyes are shown in Figure 1a and 1b.

Treatment

Polyester fabrics were treated with an aqueous solution containing different concentrations (0.5%-4.0% o.w.f.) of Lipolase 100L-EX or lipase type II for different period of times (15-90 min). The reaction was conducted at 40°C at pH 8, for lipolase 100L-EX, and pH 7.4, for lipase type II. The material-to-liquor ratio was 1:40.

Dyeing

Enzyme-treated as well as untreated polyester fabrics were dyed in an aqueous solution containing 1% (o.w.f.) Basic Red 18, and Reactive Red 120 using liquor ratio 1:50 at the boil for 30 min. The pH of the dyebath was adjusted at 5.0 in case of basic Red 18, and 5.0-5.5 in case of Reactive Red 120.

Measurement and analysis

Color measurements: The relative color strength (K/S) of dyed fabrics was measured by the light reflectance technique using the Kubelka-Munk equation [21]. The reflectance of dyed fabric was

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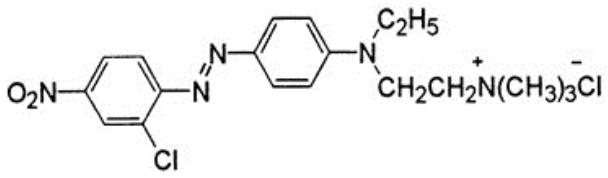

Figure 1A: Structural formulae of the used dyes: (A) Reactive Red 120.

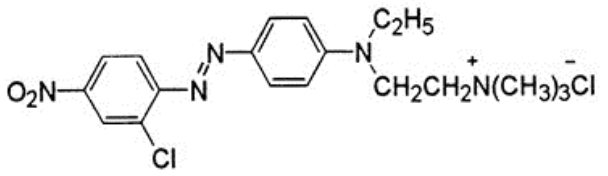

Figure 1B: Basic Red 18.

measured on reflectance spectrophotometer Model Ics-Texicon Ltd. The percentage dye exhaustion (E%) was calculated according to equation 1:

$$E\% = [A_o - A_f / A_o] \times 100 \quad (1)$$

Where A_o and A_f are the absorbance of the dyebath before and after dyeing, respectively, at λ_{max} of the dye (470 nm). The absorbance was measured on a Shimadzu UV-2401 PC UV/Vis spectrophotometer.

The concentration of the dye in the fiber (mg/g) was determined using equation 2:

$$D_f = (D_o - D_s) V / W \quad (2)$$

Where D_f is the dye concentration in fiber (mg/g), D_0 and D_s are the initial and equilibrium concentration of dye in the dye bath (mg/L), respectively, V is the volume of dyebath (L) and W is the weight of fiber (g). The concentrations of dye solution were determined after reference to the respective calibration curve using Lambert-Beer law.

The extent of dye fixation ratio of Basic blue on polyester fabric was determined by measuring K/S values of the dyed samples before and after soaping using equation 3:

$$F\% = \frac{(K/S)\,\text{after soaping}}{(K/S)\,\text{before soaping}} \times 100 \quad (3)$$

From the result of (E %) and (F %), the total dye fixation (T), was calculated using equation 4 [22]:

$$T\% = (E\% \times F\%) / 100 \quad (4)$$

Half dyeing time: After equilibrium, every dyed sample was removed and extracted by DMF as described above to determine the dye concentration in the fiber by employing a Shimadzu spectrophotometer [23]. The half dyeing period ($t_{(1/2)}$min), that is the period needed for the fabric to take up half of the amount of dye at equilibrium, is approximated by plotting the dye uptake (the dye concentration in the fiber mg/g) versus dyeing period, and ($t_{(1/2)}$min) is recognized from the corresponding curves.

Specific dye rate constant (K): The specific dyeing rate constant (K) can be further estimated by using equation (5) [23]:

$$K = 0.5C_\infty - (d / t_{1/2})^{1/2} \quad (5)$$

Where: C_∞ is the percentage of the dye absorbed onto the sample at equilibrium condition, d is the fiber diameter in cm.

Apparent diffusion coefficient (D): A recognized weight of fabric was dyed for a prolonged time (90 min.). C_∞ was determined. Extra dyeing was given for a short period (15 min.) and Ct was comparably determined. The value C_t/C_∞ was then calculated from the apparent diffusion coefficient (D) that might be calculated established on Hill's equation in equation (6) [24].

$$D = (C_t / C\infty) / t \times d^2 \times 100 \quad (6)$$

Affinity and heat of dyeing (-Δμ° and ΔH): A 3% Owf was performed on untreated and treated polyester fabric, at pH 5 for 30 min and 60 min. dyed examples were treated with 80 ml of distilled water in a stopper flask at 80°C for 2 hr. The supplementary samples were comparably treated at 60°C for 4 hr. At the end of the prescribed time, the samples were instantly removed and rinsed countless periods alongside chilly distilled water [25].

The amount of the dye in the adsorption and rinsing solution were spectro-photometrically determined. The partition coefficient (k) of the dye is determined from equation (7):

K= Concentration of dye on fabric (mmg/ kg fabric)/ Concentration of dye in bath(mg/l) **(7)**

The affinity (-Δμ°) is determined according to equation (8):

$$-\Delta\mu^o = 2.3RT \log k (\text{cal.mol}^{-1}) \quad (8)$$

Where R is the gas constant and T is the absolute temperature. The heat of dyeing (ΔH) is further determined from equation (9):

$$\Delta H = [\Delta\mu_1^o / T_1 - \Delta\mu_2^o / T_2] / [\,1/T_1 - 1/T_2\,] \quad (9)$$

Wettability: The wettability was evaluated by measuring the wetting time according to the AATCC method (1). A drop of water is allowed to fall from a fixed height on to the surface of polyester fabric under examination. The time that has been measured and taken as wetting time and the result were the average values of four reading [26].

Moisture regain: Moisture regain was performed according to the standard ASTM method 2654-76 (2). It was calculated according to the following equation [27]:

$$\text{Moisture regain}\% = \frac{(W_1 - W_2)}{W_2} \times 100$$

Where W_1: weight of sample (g) after saturation in the stander humidity atmosphere

W_2: Constant weight of dry sample

Antistatic property measurement: Static electricity of treated and untreated polyester fabrics was measured using electricity collect type potentiometer model KS-525 (Kasuga Denki, Inc., Japan). The antistatic property measurements were carried out according to Test Method of specified requirements of antistatic textile FTTS-FA-2009.

Tensile properties: The tensile properties of untreated as well as enzyme-treated fabrics were evaluated using Instron Textile Tester (USA) according to ASTM D 76.

Carboxylic content: The carboxylic content of untreated as well as treated polyester fabric was estimated by measuring the amount of alkali combined with the polymeric material as follows [28]:

(a) The sample was soaked in 2% hydrochloric acid for 3 hours to 4 hours with occasional shaking. The sample was filtered and washed several times with ethanol/water mixture (60-40) until chloride ions are free. Then the sample was filtered and dried.

(b) The dry sample (0.5 g) was precisely weighed and introduced in 250 mL Erlenmeyer flask, followed by 50 mL 0.1 N sodium hydroxide solution containing 5% sodium chloride. The flask was stoppered and allowed to stand overnight with occasional shaking. The content of the flask was back-titrated with 0.05N hydrochloric acid using phenolphthalein as indicator. Blank titration was carried out on an untreated sample, and the carboxyl content of the sample was determined as follows:

$$\text{Carboxyl content} = \frac{(X-Y)}{W} \times 100 \text{meq} / 100 \text{g fibre}$$

Where

X: volume of HCl solution used in blank titration,

Y : volume of HCl solution used in back titration,

N_A: normality of HCl solution, and

W : weight of sample (in gram).

Result and Discussion

Effect of enzyme concentration

The effect of enzyme (lipolase 100L-EX and lipase type II) concentration on the colour intensity of enzyme-treated polyester fabrics followed by dyeing with Basic Red 18, and Reactive Red 120, was investigated Table 1.

Data of this table reveals that treatment of polyester fabrics with lipase enzyme resulted in enhanced dyeability of polyester fabrics with reactive and basic dyes to various extents depending on the enzyme concentration. The two used lipase enzymes have similar effect on the dyeability of polyester fabrics. The colour strength of the dyed fabrics increased as the concentration of enzyme increased from 0.5% to 1.0% (o.w.f.). Further increase in the enzyme concentration has no or limited increase on the K/S value of the dyed samples.

These results can be interpreted in terms of the fact that treatment of polyester fabrics with lipase enzymes resulted in partial hydrolysis of ester linkages to form carboxylic and hydroxyl groups. The enhanced dyeability of polyester fabrics with basic dyes can be rationalized by virtue of the created anionic carboxylic groups along the polyester macro-molecules. On the other hand, creation of aliphatic hydroxyl groups along the polyester macromolecules will be appropriate targeted niches for the reactive dye molecules.

Effect of treatment time

The effect of treatment time of polyester fabrics with lipase enzymes on their dyeability with basic and reactive dyes was studied. Results of this investigation, summarized in Table 2 imply that 60 min of incubation of polyester fabric with lipolase 100L-EX or lipase type II is enough to impart maximum improvement in its dyeability with basic and reactive dyes.

Effect of dyeing conditions

Further investigations were conducted to assign the effect of dye concentration, dyeing temperature, and dyeing time on the dyeability of enzyme-treated as well as untreated polyester fabrics with basic dyes. Due to the similar effects of both lipolase 100L-EX and lipase type II, only these studies were conducted on those fabrics pretreated with lipolase 100L-EX.

Results of these studies are tabulated in Tables 2-5. Data of these tables demonstrates that the K/S values of lipase-treated polyester fabrics are always much higher than the corresponding untreated polyester fabric using the same dye concentration. Moreover, as the dye concentration increased from 0.5% to 3.0% (o.w.f.), the K/S value of the dyed fabric was enhanced from 0.9 to 3.3. Further increase in the dye concentration to 3.5% (o.w.f.) has no appreciable effect on the colour strength of the dyed fabrics.

Dyeing kinetics

The rate of reaction is expressed as the change in reaction concentration with time. Therefore, monitoring the change in dye exhaustion with time leads to an assessment of the dyeing kinetics for certain process.

Exhaustion time of treated as well as untreated polyester fabric dyed with Basic blue at 60°C and 80°C are shown in Figure 1. In all case, the behavior of the dyeing isotherm indicates early saturation, irrespective of the fabric treatment or the temperature used. The data in Figure 2 can be analyzed by using equation 5:

$$A_t - A_f / A_0 - A_f = Qe^{-kt} \quad (10)$$

Where K is the kinetic constant proportional to the diffusion coefficient, Q Is the coefficient dependent on equilibrium exhaustion, A_t is the absorbance of the dye bath at time t, A_0 is the initial absorbance, A_f is the final absorbance and t is the dyeing time. This formula is applicable for middle and final stage of dyeing and takes into consideration the first term of the infinite sum of general solution for describing the diffusion into the fiber.

Taking the logarithm of equation 5 would lead to equation 6 and since A_f is known so $A_t - A_f$ can be calculated.

$$E\% = [A_o - A_f / A_o] \times 100 \quad (11)$$

A plot of $\ln(A_t - A_f / A_0 - A_f)$ vs. time is expected to be linear with a slop of ΔH. The values of the dyeing rate constant are lasted in Table 6 and Figure 2.

Half dyeing time, dyeing rate constant (k), and Diffusion coefficient (D): The rate of reaction is expressed as the change in reactant concentration with time. Therefore, monitoring change in dye exhaustion with time leads to an assessment of the dyeing kinetics for a certain process. Time exhaustion isotherms of dyed pre-treated polyester fabric with 3% owf basic dye, are shown in Table 7. The result shows that in all cases, the behaviors of the dyeing isotherms indicate main saturation irrespective of the enzyme treatment

Standard affinity: It is a measure of dye tendency to move from the solution to the fiber when it is in its standard state in each phase. From Table 8 it can be seen that the standard affinity values of the pre-treated polyester fiber more than the untreated one. The result in this table also indicates that the values at 60°C are lower than those obtained at 80°C.

Enzyme concentration (% o.w.f.)	K/S			
	Lipolase 100L-EX		Lipase type II	
	Reactive Red 120	Basic Red 18	Reactive Red 120	Basic Red 18
Untreated	2.2	0.1	2.2	0.1
0.5	2.8	1.3	2.9	1.5
1.0	3.7	1.9	3.5	2.0
2.0	3.4	1.6	3.3	1.9
3.0	3.4	1.1	3.4	1.9
4.0	3.4	0.8	3.3	1.8

Table 1: Effect of treatment of polyester fabrics with two different lipase enzymes on the colour strength of enzyme-treated fabrics and dyed with reactive or basic dye (Treatment condition: L.R. 1:40, treatment time 60 min at 40°C, pH 8 for lipolase 100 L- EX and pH 7.4 fir lipase type II).

Treatment time (min)	K/S			
	Lipolase 100L-EX		Lipase type II	
	Reactive Red 120	Basic Red 18	Reactive Red 120	Basic Red 18
0	2.2	0.1	2.2	0.1
15	2.5	0.1	2.3	0.3
30	3.0	0.7	2.7	1.1
45	3.7	1.2	3.1	1.7
60	3.7	1.9	3.5	2.0
75	3.8	2.0	3.6	2.2
90	3.9	2.1	3.6	2.2

Table 2: Effect of treatment time on the colour strength of enzyme-treated as well as untreated polyester fabrics dyed with reactive or basic dye (Treatment condition: L.R. 1:40, enzyme concentration 1 % (o.w.f.) at 40°C, pH 8 for lipolase 100 L-EX and pH 7.4 fir lipase type II).

Dye concentration (%, o.w.f.)	K/S	
	Basic Red 18	
	Treated fabric	Untreated fabric
0.5	0.9	0.04
1.0	1.9	0.1
1.5	2.2	0.2
2.0	2.8	0.3
2.5	3.1	0.3
3.0	3.3	0.3
3.5	3.3	0.3

Table 3: Effect of dye concentration on the colour strength of enzyme-treated as well as untreated polyester fabrics dyed with basic dye (Treatment conditions: L.R. 1:40, enzyme concentration 1% (o.w.f.), treatment time 60 min at 40°C, pH 8). Dyeing condition: 30 dyeing time, pH 5 at the boiling; the liquor ratio is 1:100.

Dyeing time (min)	K/S	
	Basic Red 18	
	Treated fabric	Untreated fabric
5	1.6	0.09
10	1.9	0.1
15	2.4	0.2
30	3.3	0.3
45	3.9	0.5
60	4.3	0.8
120	4.6	0.9
180	4.6	1.1
240	4.6	1.1

Table 4: Effect of dyeing time on the colour strength of enzyme-treated as well as untreated polyester fabrics dyed with basic dye (Treatment condition: L.R. 1:40, enzyme concentration 1% (o.w.f.), treatment time 60 min at 40°C, and pH 8). Dyeing conditions: 3% shade, pH 5 at the boiling; the liquor ratio is 1:100.

This may be due to the fact that the dyeing operation is an exothermic process.

Heat of dyeing (ΔH): Heat of dyeing was calculated and the values are listed in Table 8. Enthalpy was found to have a negative value indicating that the dyeing process is an exothermic one.

Effect of lipase treatment on the physico-mechanical properties of polyester: The effect of treatment of polyester with lipase type II or lipolase 100L-EX, on some of its properties, was investigated. Results of this table, summarized in Table 9, indicate that treatment of polyester fabric with the said enzymes has no adverse effect on their mechanical properties; namely tensile strength and elongation at break. On the other hand, the wettability and moisture regain of the enzyme-treated fabrics were enhanced remarkably compared to the untreated. This can be attributed to the creation of hydrophilic niches, namely carboxylic and hydroxyl groups, along polyester macromolecules as a result of hydrolytic effect of lipase enzymes in aqueous medium. This hypothesis was supported by the reduced electrostatic charge and higher carboxylic content of the enzyme-treated fabrics relative to their counteracted analogue.

Conclusion

The ester linkages along the polyester macromolecules were found to be suitable candidates for ennzymatically-catalyzed hydrolytic reactions viai two commercially produceed lipase enzymes, namely lipase type II and lipolase 100L-EX. Hydrolysis of the ester bond creats carboxylic and hydroxyl groups within the polyester fabrics. Consequently, the enzyme-treated samples exhibited unusual affinity towards reactive dyes via the induced hydroxyl groups, as well as towards basic dye through the created carboxylic groups. This finding would impart suitable conditions for dyeing of polyester fabrics with those normally dyed with reacive, such as cotton and viscose fabrics, or basic dyes such as acrylic fabrics.Treament of polyester fabric with the said enzymes improved its performance attributes, such as reduced electrostatic charge, as well as enhanced wettability and moisture regain. Treatment of polyester with lipase type II and lipolase 100L-EX, under the used experimental cositions, has no deteriorative action on the treated polyester fabrics.

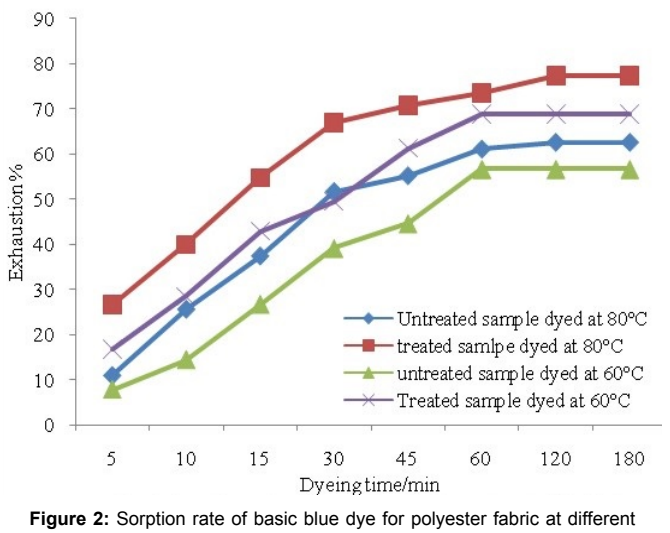

Figure 2: Sorption rate of basic blue dye for polyester fabric at different temperature.

Temperature (˚C)	K/S	
	Basic Red 18	
	Treated fabric	Untreated fabric
30	0.7	0.06
40	0.9	0.09
50	1.6	0.2
60	2.7	0.4
70	3.3	0.6
80	4.2	0.8
90	4.5	0.9
100	4.6	0.9

Table 5: Effect of dyeing temperature on the colour strength of enzyme-treated as well as untreated polyester fabrics dyed with basic dye (Treatment condition: L.R. 1:40, enzyme concentration 1% (o.w.f.), treatment time 60 min at 40˚C, and pH 8). Dyeing conditions: 3% shade, dyeing time 120 min, pH 5; the liquor ratio is 1:100.

Dyeing time/ min	Untreated fabric			Treated fabric		
	E%	F%	T%	E%	F%	T%
5	10.9	33	3.597	26.6	48.1	12.7946
10	25.6	39.4	10.0864	39.9	57.5	22.9425
15	37.4	50.1	18.7374	54.7	66.7	36.4849
30	51.7	62.9	32.5193	66.8	80.5	53.7740
45	55.2	65.7	36.2664	70.7	82.0	57.9740
60	61.1	70.0	42.7700	73.3	83.4	61.2156
120	62.6	71.1	44.5086	77.3	85.7	66.2461
180	62.6	71.1	44.5086	77.3	85.7	66.2461

Table 6: Effect of dyeing time on Exhaustion (E%) and total fixation (T%) of treated and untreated polyester fabric dyed with Basic blue.

Polyester type	Half dyeing time $t_{1/2}$/min.	Dyeing rate constant (k)	Diffusion coefficient (D)
Untreated	60	510.790×10^{-3}	178.538
Treated	30	726.895×10^{-3}	212.863

Table 7: Half dyeing time $t_{½}$ dyeing rate constant (k), and Diffusion coefficient (D) of pre-treated polyester dyed with the Reactive Red 24.

Polyester type	K		$-\Delta\mu$(Kj/ mol)		ΔH(Kj/ mol)
	60˚C	80˚C	60˚C	80˚C	
Untreated	0.00746	0.00333	-3237.358	-3996.905	9421.630
Treated	0.00734	0.00266	-1523.978	-4154.298	42306.170

Table 8: Langmuir sorption parameters and calculated thermodynamic values of the different pre-treated polyester fabrics.

Property	Untreated	lipase Type II	lipolase 100L-EX
Tensile strength	75	69.3	72.5
Elongation (%)	31	32.6	30.8
Wettability (s)	25	17	13
Moisture regain	0.8	3.3	3.9
Electrostatic charge	1.1	0.8	0.7
Carboxylic content (meq./100 g sample)	16.6	28.1	27.5

Table 9: Effect of lipase treatment of polyester on some of its properties.

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