AUTEX Research Journal, Vol. 20, No 3, September 2020, DOI 10.2478/aut-2019-0030 © AUTEX

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A STUDY ON IMPROVING DYEABILITY OF POLYESTER FABRIC USING LIPASE ENZYME

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Abstract:

Enzymatic hydrolysis on synthetic fibers enhances the hydrophilicity and solves the concerns regarding the environmental issues of textile industry. Lipase hydrolyses ester linkages in polyethylene terephthalate and produces polar hydroxyl and carboxylic groups. The study aims to identify and investigate the effect of enzyme treatment on weight loss and surface modification of polyester fabrics. Also the functional groups present before and after treatment and the effect of enzyme treatment on the improvement of dye uptake are studied. The test indicates that enzymatic process creates less surface damage, weight loss and improved moisture regain, dye uptake, and shear properties.

Keywords:

Enzymatic hydrolysis, polyethylene terephthalate, weight loss, shear properties

1. Introduction

Polyethylene terephthalate (PET) is one of the most commonly used synthetic fiber [1]. Merits of PET include high strength, high stretch resistance, washability, wrinkle resistance, and abrasion resistance [2, 3]. However, PET has undesirable properties such as pilling, static, and lack of dyeability associated with its hydrophobic nature. PET fiber has a low moisture regain of about 0.4% [4]. The most conventional and industrial way to modify polyester fabrics is alkaline treatment [5–7], but alkaline treatment affects the strength of polyester fabrics. A recent alternative is the use of enzymes in surface modification [8–10]. Studies about enzymatic treatment of polyester have been focused on biodegradation of aliphatic polyester using a lipase and biological synthesis of polymer with enzymes [11, 12]. Only a few studies have been reported regarding enzymatic modification of PET fabric [13]. Improvement in hydrophilicity of polyesters by hydrolysis of ester bonds has been reported [14]. The applicable enzymes used are lipases and polyesterases. Lipase is known to hydrolyze water-insoluble esters or triglycerides composed of long-chain fatty acids [15]. Thus, the application and studies about lipases in textile processing have been focused on detergent. If enzymes can hydrolyze ester linkage in PET fabrics, polar hydroxyl (–OH) and carboxyl (–COOH) groups will be formed on the surface of PET fabrics. As a result, moisture regains and wettability will improve due to the forming of hydrophilic groups on PET fabrics. Carboxyl and hydroxyl groups on PET fabrics can be evaluated through dyeability of disperse dyes. Hydrolysis of the ester bond creates carboxylic and hydroxyl groups within the polyester fabrics [16]. Under optimum conditions, lipase enzymes of different sources significantly improve the hydrophilicity and dyeability of PET-treated fabrics [17]. Enzymes active on PET substrates include various cutinases, lipases, and esterases. The enzymatic modification of PET[5] implies the limited hydrolysis of backbone ester bonds, which generates new free hydroxyl and carboxyl groups at the polymer surface, thus leading to increased hydrophilicity of the PET substrate, as shown in Figure 1.

1.1. Aminolysis

Several studies have assessed the effects of amine interaction with polyester. Early studies assessed the aminolysis of polyester as a means of examining fiber structure without maintaining the integrity of the polymer. The degradation

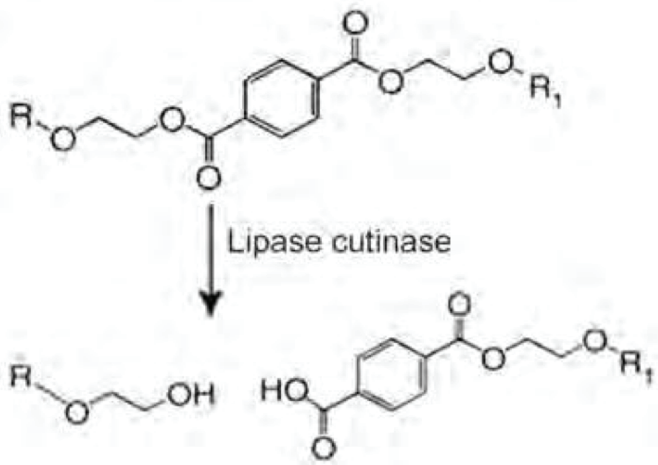

Figure 1. Enzymatic hydrolysis of polyethylene terephthalate (PET)

effects on polyester of a monofunctional amine versus alkaline hydrolysis have been studied. These studies, which again involved high levels of fiber degradation, demonstrated that alkaline hydrolysis has a more substantial effect on fiber weight without extensive strength loss. In contrast, aminolysis had less effect on fiber weight but decreased fiber strength, indicative of a reaction within the polymer structure rather than simply at the surface. It was later demonstrated that bifunctional amine compounds could be reacted with the polymer with minimal loss in strength while generating amine groups at the fiber surface. The early stages of the reaction were largely confined to the fiber surface and the resulting fiber had modified wetting properties and improved adhesion with the matrix when used in composites [18].

2. Materials and methods

In this study, 100% polyester fabric plain weave was used to know the effect of enzyme treatment on the structural modifications. Polyethylene terephthalate, PET, heat set fabric having a surface mass of 82 g/m^2 was used for this study; textured multifilament yarns in both warp and weft directions. Yarn consisted of delustered fibers with trilobal cross-section in warp and circular cross-section in weft. Fabric and enzyme specifications are discussed in Tables 1 and 2 and labels are given in Table 3. The fabrics are subjected to washing to eliminate the presence of impurities and finishes. Fabrics are treated with enzymes of varying concentrations, treatment time, and temperatures. The fabrics selected for the experiments are subjected to alkali and lipase treatment with different concentrations, namely 5%, 10%, and 15%. The fabrics are dyed with disperse dye C.I. Disperse Blue 284 with a shade level of 5% on weight of the fabric. The treated fabrics are dyed using disperse dye with high-temperature high-pressure dyeing machine. The fabric weave is a plain weave and the chemicals used in this work were laboratory grade reagents.

2.1. Pretreatment of polyester fabric

Polyester fabric was immersed in 10 gpl HCl at 40°C and treated for 1 hour at the same temperature with material to liquor ratio of 1:50, to get rid of the added impurities [19].

2.2. Sodium hydroxide treatment

The pretreated PET fabrics were subjected to various concentrations of sodium hydroxide treatment at boil for 1 hour and 2 hours of treatment time.

2.3. Lipase enzyme treatment

Each fabric sample was cut into specific dimensions and weighed ~1 g. Depending on pH, temperature, concentration, and treatment time, the PET fabrics were treated with lipase in Tris buffer solution, using a liquor ratio of 80:1. All the lipase treatments were performed at 150 rpm using a shaking water bath. The enzyme inactivation was performed at 80°C for 10 minutes. The lipase-treated samples were thoroughly washed with water and dried at room temperature. Then, weight loss, scanning electron microscope (SEM) micrographs, water vapor permeability, Fourier transform infrared spectroscopy (FTIR), Color analysis, and low-stress mechanical properties of lipase-treated PET fabrics were measured.

2.4. Dyeing

Enzyme-treated as well as sodium hydroxide–treated polyester fabrics were dyed using 0.5% (owf) dianix navy S2G (disperse dye), C.I. Disperse Blue 284 and 1 g/L dispersing agent in a HTHP dyeing machine. The dyeing was carried out at a temperature of 130°C and at pH 5 (adjusted by acetic acid). The dyed samples were washed with hot water, soaped, and dried.

3. Analytical methods

3.1. Weight loss evaluation

The weight loss (WL) is expressed as relative WL according to the equation:

$$WL=100*(W1 – W2)/W1$$

where W1 and W2 are the weights of the samples before and after treatment, respectively.

3.2. Wicking height measurement

The wicking heights of the treated fabrics are tested as per AATCC Test Method 197 to evaluate the specimens to transport liquid along them. The distance at a given time is measured.

3.3. Absorbency measurement

The water absorbency of treated fabrics is tested as per AATCC Test Method 79 to know the time taken by a drop of water placed on the fabric surface to completely absorb.

3.4. SEM analysis

The enzyme-treated polyester and blended samples are analyzed through SEM to know the effect of enzyme treatment on surface etching. The surface morphology of polyester fabrics was observed in SEM (JOEL JSM-6360).

3.5. FTIR analysis

FTIR tests were carried out to reveal the additional functional groups present due to alkaline and enzymatic hydrolysis. The attenuated total reflectance–Fourier transform infrared spectroscopy (ATR–FTIR) measurements were carried out on polyester fabrics (control and treated) using an infrared spectroscopy. Nicolet IR200 FT-IR system with a single-reflection attenuated total reflectance attachment was used. Solid samples are prepared by shaving material off of the part that is thin enough to obtain a good FTIR spectrum. The x-axis

Table 1. Fabrics specifications

Fabric type	Weave	Linear density	Ends/inch	Picks/inch	Thickness (mm)	GSM
Polyester (100%)	Plain	75*77Denier	120	94	0.18	82

Table 2. Enzyme specifications

Name of the enzyme	Activity	Form	Manufacturer
Lipolase 100L-EX	100,000 U/g	Liquid	Novozyme

Table 3. Labels and treatments

S. No.	Treatment	Label
1	Untreated polyester fabric (100%)	PU
2	Alkaline-treated polyester fabric	PA
3	Lipase-treated polyester fabric	PL
4	Dyed alkaline treated	PAD
5	Dyed lipase–treated fabric	PLD

is wavenumber (cm−1), which is the inverse of wavelength (cm). The y-axis is absorbance normalized on a scale of 0 to 1, where 0 denotes no absorption and 1 denotes maximum absorption.

3.6. Water vapor permeability measurement

The water vapor transmission rates of treated fabrics are tested as per ASTM D6701-16 using water vapor permeability tester.

3.7. Color measurements

The relative color strength (*K/S*) of dyed fabrics was measured using SS5100A spectrophotometer premier color scan by the light reflectance technique at a wave length 400−700 nm. Reflectance values were measured and the relative color strength (*K/S*) was calculated using Kubelka–Munk equation. *K/S* defines a relationship between spectral reflectance *I* of sample and its absorption (*K*) and scattering (*S*) characteristics.

3.8. Evaluation of low-stress mechanical properties

The Kawabata Evaluation System of Fabric (KESF) tester is used to measure the low-stress mechanical properties of alkaline- and lipase-treated fabrics.

3.9. Statistical analysis

To understand the significance of various test results, statistical parameters are analyzed using SYSTAT Software. The statistical parameters like t-test, correlation coefficient, and regression equation were derived.

4. Results and discussions

4.1. Influence of lipase treatment on weight loss of polyester fabrics

It is understood from Tables 4 and 5 that the increase in concentration of enzyme causes increase in weight loss in both the cases. The weight loss is significantly lower for lipase-treated samples than alkaline-treated, in similar concentrations (t value=2.963; p value=0.098).

There is a high degree of positive correlation between enzyme concentration and weight loss in both the cases, but the lipase treatment shows lesser ($0.955<0.981$). The multiple R of 0.981 indicated in Table 6 shows linear relationship, and F ratio of 25.075 indicate that samples treated at different concentrations have significant weight loss.

4.2. Effect on absorbency

Table 7 values indicate that lipase-treated samples are having higher absorbency characteristics compared with the untreated and alkaline-treated fabrics.

4.3. Effect of lipase treatment on surface characteristics of polyester fabrics

The enzyme-treated polyester and blended samples are analyzed through SEM to know the effect of enzyme treatment

Table 4. Effect of lipase treatment on weight loss of polyester fabrics

Label	Fabric type	5%	10%	15%
PA	Alkaline-treated weight loss (%)	7.14%	12%	22%
PL	Lipase-treated weight loss (%)	2%	2.6%	4.6%

Table 5. Statistical analysis

Fabric	Mean of PA	Mean of PL	Mean difference	95% confidence interval (CI)	Standard deviation (SD) of difference	t value	Degrees of freedom (df)	p-value
Polyester	13.713	3.067	10.647	−4.815 to 26.109	6.224	2.963	19	0.098

on the surface etching. The SEM photographs of the alkaline- and lipase-treated samples are magnified (5000×) to understand the effect of treatments, respectively. The extent of damage is shown in Figure 2.

4.4. Effect of lipase treatment on moisture regain

FTIR tests reveal the additional functional groups due to alkaline and enzymatic hydrolysis. The addition of hydroxyl groups in polyester is responsible for the hydrophilicity. The following comparative FTIR spectrum reveals the modification in the functional chains, which are responsible for the property changes.

Figures 3–6 and Table 8 indicates that additional groups, such as aromatic rings, alkynes, esters, hydrogen bonds, and carboxylic acids, are present in treated fabrics. The stretching in the region of 3600–3200 is responsible for the hydrophilicity of lipase- and alkaline-hydrolyzed polyester. The high peaks from 1700 cm^{-1} to 600 cm^{-1} indicate the original signals, such as characteristic spectra of stretching vibration band of C=O at 1760 cm^{-1} and C–O–C stretching vibration band at 1097

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Table 6. Correlation and regression analysis

Fabric	Pearson correlation coefficient	Multiple R	Squared multiple R	Adjusted squared multiple R	t value	p value	F ratio
Polyester	PE-0.955 PA-0.981	0.981	0.962	0.923	5.007	0.125	25.075

Table 7. Influence of lipase treatment on absorbency of polyester

Label	Fabric type	Wicking height (Cm)	Drop test (seconds)
PU	Untreated fabric	2.5	4.8
PA	Alkaline-treated fabric	5.9	3.5
PL	Enzyme-treated fabric	6	2.8
PAD	Dyed alkaline treated	6.0	3.4
PLD	Dyed lipase–treated fabric	7	3.0

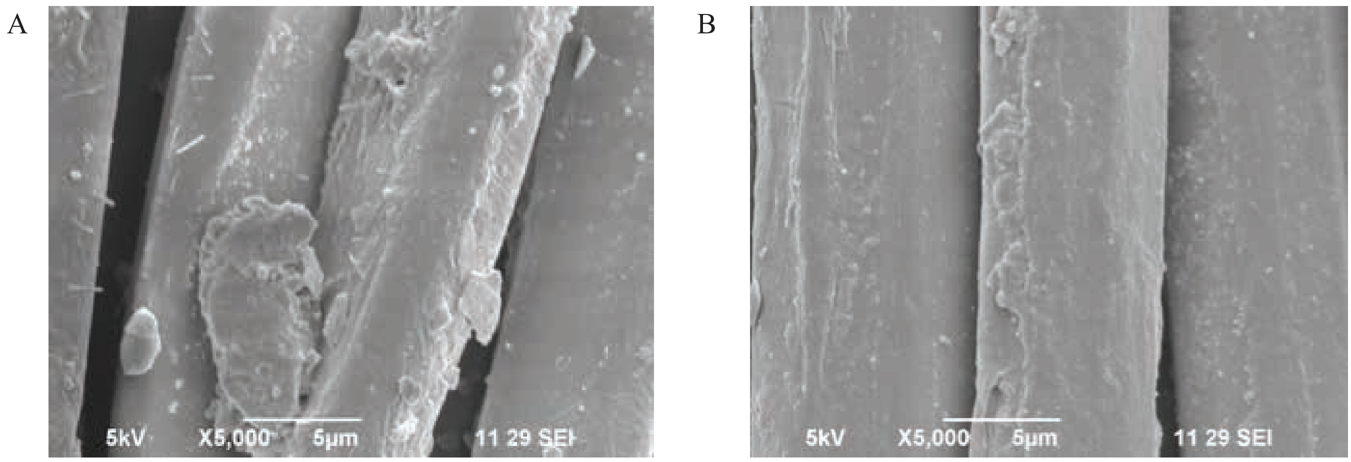

Figure 2. Scanning electron microscope (SEM) images of A (alkaline-treated) and B (lipase-treated) polyester fabric

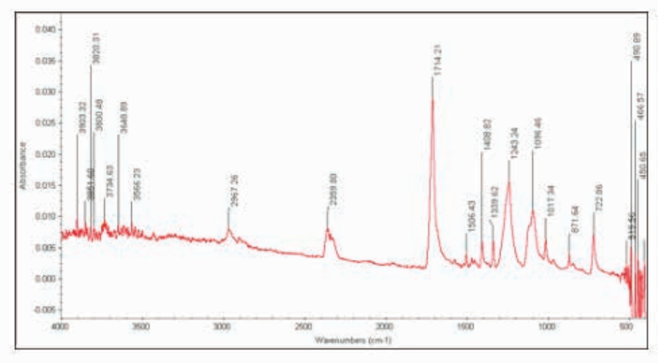

Figure 3. FTIR spectra of untreated polyester

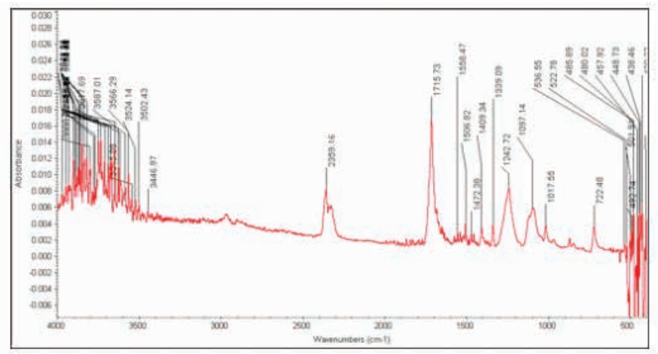

Figure 5. FTIR spectra of lipase-hydrolyzed polyester

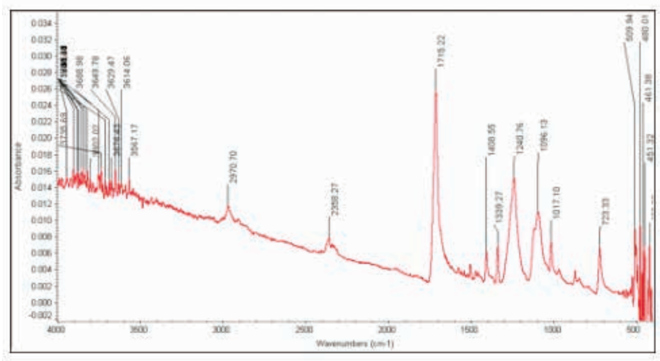

Figure 4. FTIR spectra of alkaline-hydrolyzed polyester

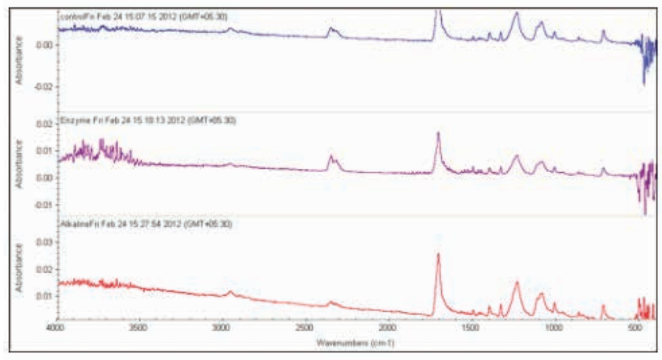

Figure 6. FTIR comparison spectra of untreated and hydrolyzed polyester

Table 8. Interpretation of Fourier Transform Infrared (FTIR) spectra of polyester

Group	Vibration	Type	Range	Inference
Aromatic rings	Ring stretch	Symmetric	1600,1500 stretch	C=C
Alkynes	Bend	Band	700–610(b) bend	C–H
Hydrogen-bonded	Stretch	OH stretch	3600–3200(b) stretch	O–H
Esters	Stretch	C=O	1760–1670(s) stretch	C=O
Carboxylic acids	Stretch	Antisymmetric	3000–2500(b) stretch	COOH

Table 9. Water vapor permeability of lipase-treated polyester fabric

Treatment type	Label	Water vapor permeability(g/m2/day)
Untreated polyester fabric (100%)	PU	1260
Alkaline-treated polyester fabric	PA	1660
Lipase-treated polyester fabric	PL	1980
Dyed alkaline treated	PAD	1820
Dyed lipase–treated fabric	PLD	2761

and 1240 cm^{-1}. All these peaks confirm the existence of ester linkage. This is attributed to the carboxylic group (–COOH) introduced on the surface due to hydrolysis of the ester linkage. The stretching is very high in the case of lipase-hydrolyzed polyester, which makes the enzyme treated more hydrophilic. It is also noted that the presence of basic functional groups inherent in the polyester materials.

4.5. Effect of lipase treatment on water vapor permeability of polyester fabrics

From Table 9, it is understood that the alkaline- and lipase-treated polyester fabrics are having improved water vapor permeability compared with the untreated polyester fabrics. It is mainly due to the degradation of chain links and the addition of hydroxyl groups.

Table 10. Polyester fabric K/S values

Type of treatment	Label	Wave length (nm)/K/S value						
		400	450	500	550	600	650	700
Alkaline treated, dyed	PAD	3.904	5.897	4.167	1.503	0.169	0.023	0.014
Lipase treated, dyed	PLD	4.021	5.79	3.685	1.318	0.146	0.018	0.012

Table 11. Effect of lipase treatments on compression and shear properties of polyester

Fabric type	Treatment type	Label	LC	WC (gf cm/cm²)	RC (%)	G (gf/cm.deg)	2HG (g/cm)	2HG5 (g/cm)
100% polyester	Untreated	PU	0.273	0.062	43.55	0.52	0.50	1.79
	Alkaline treated	PA	0.303	0.069	42.03	0.56	0.79	2.24
	Lipasetreated	PL	0.292	0.062	51.61	0.61	0.90	2.17
	Dyed alkaline treated	PAD	0.304	0.070	42.06	0.57	0.78	2.20
	Dyed lipase treated	PLD	0.308	0.064	54.69	0.59	0.66	2.13

LC, linearity of compression thickness; RC, compression resilience; WC, compression energy.

4.6. Effect of enzyme treatment on dye uptake of polyester fabrics

The alkaline-treated, lipase-treated, and dyed fabrics are tested by reflectance type spectrophotometer to know the *K/S* values. These values help understand the difference in luster and color values. The treatment offers better dye uptake that enhances the color values.

From Table 10, it is understood that lipase-treated fabrics shows higher reflectance and lower *K/S* values. Similar trend is observed in dyed fabrics. This clearly shows that treatment enhances the dye uptake of the polyester fabrics.

4.7. Effect of enzyme treatment on the low-stress mechanical properties of polyester

The Kawabata evaluation tests are used for evaluating compression and shear properties in lipase-treated and untreated polyester fabrics, as it reveals the effectiveness of the enzyme treatment.

Table 11 shows that lipase treatment reduces the linearity of compression and compression energy, whereas enhances the compression resilience in polyester fabrics. The enzyme treatment makes the fabric much softer, and it retains its original shape as quickly as possible while applying low stress load. Hence it is possible to infer that lipase treatment makes the polyester fabric with low shear rigidity, which indicates that the fabric will conform to 3-dimensional structures while applying low stress.

5. Conclusions

Alkali treatment offers more surface itching and weight loss, whereas lipase enzyme attacks the polymer chain, which causes mild itching and low weight loss. The treated fabrics have improved water vapor permeability compared with the untreated polyester fabrics. It is mainly due to the degradation of chain links, which ultimately enhances the dye uptake and improves the moisture uptake. Enhanced compression resilience in polyester fabrics makes them much softer and helps to retain its original shape as quickly as possible while applying low stress load, which makes the polyester fabric with low shear rigidity. The statistical result shows that the treatment is significant and effective in modifying the fabric properties.

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