Indian Journal of Fibre & Textile Research
Vol. 51, June 2026, pp. 187-198
DOI: 10.56042/ijftr.v51i2.20674

Sustainable and energy-efficient dyeing of esterase-treated polyester with reactive dyes

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Received 15 June 2025; revised received and accepted 13 January 2026

The conventional high-temperature, high-pressure (HTHP) dyeing of polyester with disperse dyes is energy-intensive (~1580 kcal/kg fabric), time-consuming, and requires specialized machinery while producing harmful, dye-laden effluents. The issue is more pronounced with polyester cotton blends, as polyester requires disperse dyeing at high temperature while cotton requires reactive dyes, both in aqueous media, necessitating separate or sequential dyeing steps that increase water, energy, and chemical consumption. This study presents a cleaner, energy-efficient alternative through the enzymatic surface modification of polyester using Esterase enzyme. The treatment introduces reactive -OH and -COOH groups, enabling dyeing with cold-brand reactive dyes, which are water-soluble, self-hydrolyzing, and require much less energy (~201 kcal/kg fabric). Treatment parameters, enzyme concentration, time, temperature, and pH, were optimized, and FTIR analysis confirmed successful functionalization. The modified polyester showed enhanced absorbency, reduced wicking height and contact angle, and better dye affinity without compromising fabric strength. Dyeing at 40-45 °C achieved 60-70% energy savings over the HTHP process. This method also eliminates the need for high-pressure machinery, reduces process complexity, and lowers effluent load due to the environmentally benign nature of reactive dyes. The dyed fabrics exhibited uniform shades, good color fastness, and moderate K/S values. Overall, this strategy supports sustainable, eco-efficient textile processing.

Keywords: Effluent treatment, Energy consumption, Esterase, Polyester, Reactive dyeing, Sustainability.

1 Introduction

Polyester, being hydrophobic, is typically dyed with disperse dyes at high temperatures (around 130 ± 2 °C) using the High Temperature High Pressure (HTHP) method in jet or beam dyeing machines. This method ensures uniform shade development but is highly resource-intensive, requiring substantial thermal energy, prolonged processing times, and expensive specialized equipment. The HTHP method alone consumes approximately 120 kcal/mol of polyester and necessitates extended exposure to high temperatures for efficient dye fixation. Additional operational challenges include fabric creasing, jute wrapping for beam preparation, and lengthy pre- and post-treatment procedures, all contributing to increased energy consumption, costs, effluent generation, and labour intensity[1].

Dyeing polyester-cotton blends typically involves two separate stages: polyester is dyed first in rope form using disperse dyes, followed by the reactive dyeing of cotton in open width. This two-step process increases water and energy consumption and introduces logistical complexity. Furthermore, disperse dyes are insoluble and non-biodegradable; when released into wastewater, they contribute to environmental pollution and increase the effluent load[1]. These challenges emphasize the need for a low-temperature, eco-friendly dyeing method that reduces energy use and wastewater generation while simplifying processing[1].

A sustainable solution involves modifying the surface of polyester to enable dyeing at lower temperatures using cold-brand reactive dyes. These dyes are water-soluble, self-hydrolyzing, and more biodegradable, resulting in cleaner effluent profiles. This reduces energy demands, eliminates the need for high-pressure equipment, and allows for single-bath dyeing of polyester-cotton blends, conserving water, chemicals, and time[1].

Enzyme-based surface modification offers a more environmentally friendly alternative. Enzymatic treatments operate under mild conditions, require less energy and water, and are compatible with existing

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textile processes. Enzymes are already widely used in textile finishing, such as amylases for desizing[2], pectinases and lipases for scouring[3], peroxidases for bleaching[4], and cellulases for biopolishing[5].

For polyester, hydrolytic enzymes such as cutinases, esterases, lipases, and proteases can improve surface wettability and reactivity. These enzymes can break down the accessible ester bonds on the surface of PET, forming polar hydroxyl and carboxyl groups, imparting ionic properties on surface of polyester and enhance its wettability and reactivity[6].

Several studies have explored the enzymatic hydrolysis of polyester using enzymes such as *Thermobifida cellulosilytica*[7,8], *Fusarium solani pisi*[9] and *Fusarium oxysporum*[10] cutinases; Esterases from T. Fusca[11], *Thermomyces lanuginosus* and *Beauveria brongniartii*[12]; and microbial lipases[13,14]. These enzymes selectively hydrolyze ester bonds on the polyester surface without significantly weakening the fabric[15]. Enzymatic treatments rely on careful optimization of parameters like enzyme concentration, temperature, time, and pH. This facilitates low-temperature reactive dyeing of polyester with reduced cost and environmental impact[16]. Further innovations in reactive dyeing, especially for cellulose, have shown reduced water consumption and dye usage[17]. Although cold-brand reactive dyes may suffer from partial hydrolysis, which affects wash fastness, this can be addressed by precise process control[18]. Enzymatic pretreatment also enables single-bath dyeing of polyester-cotton blends, eliminating the need for a two-step process.

Earlier studies mostly explored whether enzymatic treatments could improve hydrophilicity of polyester and allow dyeing with reactive dyes[19,20]. In comparison, Esterase-based modification offers a promising approach where enzymatic processing and dyeing can be carried out at much lower temperatures than conventional high-temperature, high-pressure (HTHP) disperse dyeing. This not only reduces energy use and cost but also eliminates the need for specialised machinery. Unlike methods such as plasma treatment or hydrophilic coatings, which remain costly and confined to the lab, this process is simpler and more practical and can be easily commercialised. The study systematically examines key factors, time, temperature, pH, and enzyme concentration, and shows how enzyme dosage affects shade depth and colour fastness with dichlorotriazinyl reactive dyes.

In this study, polyester fabric was treated enzymatically using esterase under optimized conditions determined by a Box-Behnken design. FTIR spectroscopy was used to characterize introduced functional groups, which were quantified via volumetric titration. Other properties were evaluated, including absorbency, contact angle, wicking height, moisture regain, tensile strength, weight loss measurements, and thermal behaviour. Surface response plots were used to assess the influence of treatment variables. The treated polyester was dyed with cold-brand reactive dyes and evaluated for color strength and fastness properties. The energy consumption was also compared for the conventional HTHP method and this new method.

The proposed method offers a viable route to low-temperature, energy-efficient dyeing that aligns with sustainable manufacturing goals. It reduces environmental impact, optimizes resource utilization, and supports the development of circular, eco-friendly textile processes.

2 Materials and Methods

2.1 Material

In this study 100% PET fabric, ready for dyeing, with EPI: 110 and PPI: 80, 80^s warp, 70^s weft, and an areal density of 60 GSM was used.

Esterase enzyme (isolated from Bacillus SPP, activity: 1,00,000 U/g, EC 3.1.1.1) was procured from Om Biosciences, Ahmedabad.

Acetic acid, sodium carbonate (Na_2CO_3), hydrochloric acid (HCl), and sodium chloride (NaCl) were procured from SDFCL, Mumbai. Lissapol (non-ionic surfactant) was procured from ICI.

Fivecold-brand reactive dyes, viz**. P**rocion Magenta MB (Reactive Violet 13), Procion Red M5B (Reactive Red 2), Procion Yellow M3R (Reactive Orange 37), Procion Orange M2R (Reactive Orange 4), and Procion Blue MR (Reactive Blue 4) were used in this study.

2.2 EsteraseTreatment of Polyester

Polyester was treated with Esterase at various concentrations against specific times, temperatures, and pH. After the treatment, the bath was heated at 80°C for 10 min to inactivate the enzymes, followed by a thorough wash with cold water and drying.

A trial-and-error technique was used initially to determine working parameters for the modification of polyester using neutral Esterase to analyze their influence on the production of functional groups. In this investigation, Esterase concentration was initially taken as 0.5%, 1.0%, and 1.5%; the treatment period was 60, 120, and 180 min; temperature was varied at 40°C, 60°C, and 80°C; and pH was examined at 5, 7, and 9. During this phase, each parameter was adjusted separately while the others remained constant.

Based on the initial trial's results, the next step was statistical optimization using the Box-Behnken design. For this purpose, four key factors influencing enzymatic treatment, time, enzyme concentration, pH, and temperature, were selected; each was studied at three levels (low, medium, and high), resulting in a 4^3 Box Behnken Design.

2.3 Dyeing of Polyester

Esterase-treated polyester specimens were dyed with five different cold-brand reactive dyes, and the surface color strength (K/S) was determined using computer color matching (Datacolor Spectrum 200-1511 model, USA). The dyed specimens were then tested for color fastness, including wash (ISO 105-C06), light (AATCC 16-2004), rubbing (AATCC 8-2007), and sublimation fastness (ISO 105:P01, at 170 °C)[21].

2.4 Tests Conducted

2.4.1 FTIR

A Fourier Transform Infrared Spectroscopy (FTIR) study was carried out on the enzymatically hydrolysed polyester specimens using a Bruker Alpha II (USA) to investigate the development of functional groups.

2.4.2 Quantitative Estimation of Reactive Groups

2.4.2.1 Estimation of –COOH groups

The unmodified (control) and modified polyester specimens were soaked in a 2% (w/v) dilute HCl solution to remove any primary contaminants, then thoroughly rinsed with an ethanol-water solution to remove leftover acid before drying.

A 0.5g dry specimen was immersed in a 0.1M NaOH solution containing 5% NaCl for 2 hours at 60°C. The leftover NaOH solution was then titrated with standardized 0.05M HCl, with phenolphthalein as an indicator. The amount of acid required to neutralize the excess alkali was determined[22]. The carboxyl group content of the material was estimated using the difference in acid volume between untreated and treated specimens, used to neutralize, the leftover NaOH solution. The difference in NaOH concentration represents the amount of NaOH that reacted with the -COOH groups on the polyester fabric. The carboxyl content is calculated as follows:

$$\text{Carboxyl content} = (X - Y) / W \ \text{mmol/g} \qquad \ldots(1)$$

where 'X' and 'Y' denote carboxyl content in the Esterase-treated polyester specimen and the untreated polyester specimen; W: weight of polyester fabric (g).

2.4.2.2 Estimation of-OH Groups

Polyester specimens were immersed in a 4% (w/v) weak NaOH solution for 1 hour at room temperature to activate the -OH groups, followed by thorough washing and drying.A 0.5g fabric specimen was treated with 0.2M acetic acid at 60°C for 2 h. Afterward, the leftover acetic acid solution was titrated with a standardized 0.2M NaOH solution. The amount of acetic acid left in the residual solution was estimated. The difference between the starting concentration of acetic acid and the residual solution gives an estimate of how much acetic acid reacted with the specimen[23]. The hydroxyl content of the polyester specimens was measured as follows:

$$\text{Hydroxyl content} = (X - Y) / W \ \text{mmol/g} \qquad \ldots(2)$$

where X' and 'Y' denote hydroxyl content in Esterase-treated polyester and untreated polyesterspecimens; W: weight of polyester fabric (g).

2.4.3 Other Tests

The hydrophilicity of the polyester specimens was assessed using several methods, including wicking rate[24] (the height traversed by water within 2 min, according to AATCC Test Method 197), absorbency[25] (the time required for water droplets to be absorbed on the fabric surface, according to AATCC Test Method 79), and contact angle[26], which was measured with a Drop Shape Analyzer (DSA25). Moisture regain of the untreated and Esterase-treated polyester specimens was measured following the ASTM D629-99 standard[13]. The tensile strength of each untreated and treated polyester specimen was measured using a Universal Testing Machine (UTM) in accordance with ASTM D5035 standards, and the oven-dry weight of the fabric specimens was also measured to analyze the change in weight due to hydrolysis.

Differential Scanning Calorimetry was carried out on a Mettler Toledo DSC-823 to analyse the melting and crystallisation behaviour of polyester specimens. The rate of heating, initial temperature, and final temperature were set as 10 °C/ min, 30 °C, and 300 °C, respectively[27].

2.4.4 Energy and Water Consumption Analysis

In jet dyeing, polyester is dyed with disperse dyes by heating the liquor to around 130 °C, then cooling it to 80 °C, and reheating it again to 90-100 °C for reduction clearing of dark shades, followed by soaping and washing. These repeated heating and cooling stages significantly increase energy consumption. In contrast, jigger dyeing with cold-brand reactive dyes generally requires no extra heating since the necessary dyeing temperature (40-45 °C) is close to ambient conditions in processing areas. Here, thermal energy is mainly used only during the soaping step to raise the temperature to boiling. For polyester-cotton blends, the polyester component is dyed first in a jet dyeing machine, then the cotton component is dyed in a jigger, along with reduction clearing, soaping, and washing, which makes the overall process longer and more energy-intensive. As a result, the energy demand of jigger dyeing is considerably lower than that of jet dyeing.

When water consumption is considered, polyester-cotton blends require the highest quantity, as water is used extensively at multiple stages such as dyeing, reduction clearing, soaping, and repeated washing cycles for both the polyester and cotton components. In the case of polyester dyeing alone, the total water requirement is comparatively lower, but still considerable, because large bath volumes are needed for high-temperature dyeing, cooling, reduction clearing, and subsequent washing. For cotton dyeing, water consumption is relatively less than polyester dyeing, as the main requirement is for dye application, fixation, soaping, and washing. However, the cumulative consumption in polyester-cotton blends is highest because both fibres must be processed separately, multiplying the number of wet processing steps. By contrast, enzyme-treated polyester-cotton blends require significantly less water, since enzymatic modification allows dyeing of both polyester and cotton components in a single bath with cold-brand reactive dyes. This eliminates the need for separate dye baths, high-temperature stages, and multiple soaping and washing cycles, thereby reducing overall water usage substantially.

For comparison, the energy requirement for both machines is evaluated for the same liquor volume, i.e., 100 L (~100 kg), keeping the process parameters the same as those for the processes, i.e., disperse dyeing, reactive dyeing, and enzymatic treatment[28,29]. The energy consumed was then calculated mathematically using the equations given below[28]:

Thermal Energy required to heat the bath

$$Q_{heat} = m \cdot C_p \cdot \Delta T / \eta \quad \ldots(3)$$

Thermal Energy required to hold the bath at the required temperature:

$$Q_{hold} = U \cdot A \cdot (T_{bath} - T_{amb}) \cdot t / \eta \quad \ldots(4)$$

Total energy consumed: $Q_{total} = Q_{heat} + Q_{hold}$ ...(5)

Where, m is mass of water (kg), $C_p$is specific heat of water ($kJ \cdot kg^{-1} \cdot K^{-1}$), $\Delta T = (T_f - T_i)$ is temperature rise (K), η is system efficiency, U is overall heat-transfer coefficient ($kW \cdot m^{-2} \cdot K^{-1}$), A is heat-exchange surface area (m^2), T_{bath} is bath set temperature (°C), T_{amb} is ambient temperature (°C), and t is holding time (h).

Although the exact thermal energy requirement may vary due to the presence of auxiliaries in the bath, the calculated values using the above equations closely approximate the actual energy consumption. This is because water is the universal solvent in all dye baths and is present in the highest proportion compared to other auxiliaries, making its thermal properties the dominant factor.

3 Results and Discussion

3.1 Results of Initial Trial

An initial trial was carried out using a hit and trial approach, where treated specimens were evaluated to observe if there were any changes in the functional groups found on the polyester.

FTIR analysis assures the development of -COOH and -OH groups on the surface of Esterase-treated polyester (Fig. 1). Wide and intense bands at 3678.29 cm^{-1} and 3279.55 cm^{-1} are characteristic of -OH stretching vibrations, depicted in Fig. 1. They are characteristic of free hydroxyl groups that are not present in untreated polyester.

A very strong and sharp peak at 1713.75 cm^{-1}, as shown in Fig. 1, is attributed to the -CO stretching vibration of ester groups, a typical feature of polyethylene terephthalate (PET) polyester. The presence of the peak, where it appears along with significant -OH stretching, also suggests, to some extent, the partial hydrolysis of ester linkages, resulting in the development of carboxylic acid (-COOH) groups on the surface. The peak at

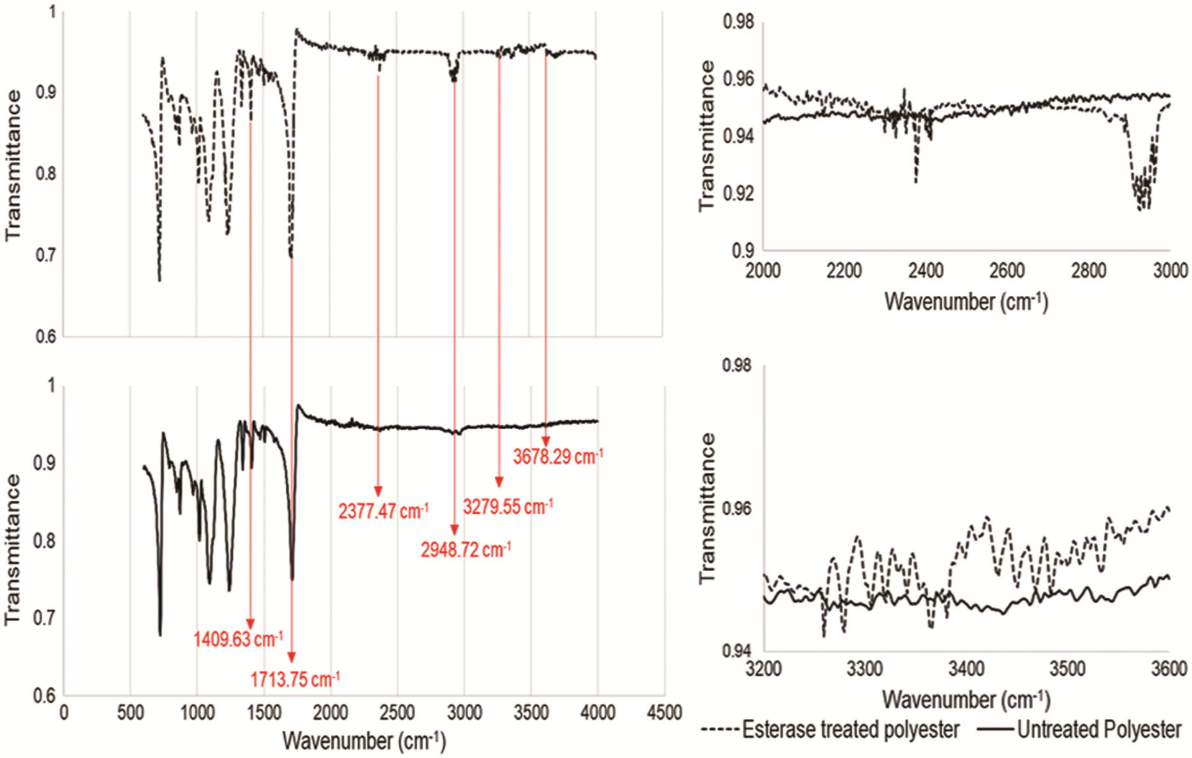

Fig. 1 — FTIR analysis for untreated and Esterase-treated polyester

1409.63 cm^{-1} explains the -OH bending or symmetric -COO stretching.

The 2948.72 cm^{-1} and 2377.47 cm^{-1} bands correspond to aliphatic -CH stretching vibrations and possible overtones or atmospheric CO_2 interference, respectively. The -CH stretching vibrations confirm that the polymer backbone is not damaged, and chemical modifications are localized on the surface.

Number of such reactive groups were assessed by carrying out volumetric titration, and it was discovered that the number of -OH and -COOH groups produced was approximately comparable at a specific concentration of Esterase. The maximum number of -COOH and -OH groups was observed at: Esterase 1.5%, pH~9, at 70 °C, and 120 min in the initial trials of this study.

3.2 Enzymatic Hydrolysis Mechanism

The Esterase, specifically hydrolyses ester bonds, used in this study, is derived from Bacillus spp. and belongs to the Hydrolase (α/β-hydrolase) class, which catalyses the hydrolytic cleavage of the ester bond in polyester[30].

The Esterase follows the classical serine-histidine-aspartic acid catalytic mechanism, where the serine residue initiates cleavage of ester bonds, resulting in the formation of -OH and -COOH groups[31,32]. Since polyester is highly crystalline, the enzyme mainly acts on the more accessible surface regions rather than the inner core of the polymer. This process is generally described as surface erosion, where the enzyme primarily targets ester linkages present in the amorphous domains, mostly on the surface. The overall hydrolysis behaviour of PET by such enzymes can be explained using Michaelis-Menten-type kinetics[33-35], generally used to analyze the bulk reactions of various substrates with enzymes.

Table 1— Factors and levels as input for Box Behnken Design

Factors	Levels		
	-1	0	1
A: Esterase, %	1	1.5	2
B: Temperature, °C	50	60	70
C: Time, min	90	120	150
D: pH	5	7	9

3.3 Effect of Various Process Parameters on Enzymatic Hydrolysis

Keeping in view the results obtained through initial trials, different factors and levels were designed to be fed to the Box Behnken design as shown in Table 1.

The levels for pH and temperature were set to be 9 and 70 ºC, respectively because the enzyme was

Table 2 — Quantification of development of -OH and -COOH groups, absorbency, and wicking height for untreated polyester and Esterase-treated polyester against different runs

Sr. no.	Esterase, %	Temperature, ºC	Time, min	pH	-OH groups, mmol/g	-COOH groups, mmol/g	Absorbency, s	Wicking Height, cm
1	2	60	120	9	14.8	13.97	392	11.2
2	1.5	70	90	7	7.1	6.20	501	8.6
3	1.5	60	90	9	11.3	9.45	432	9.7
4	2	60	120	5	17.86	19.03	335	12.4
5	1.5	50	90	7	5.89	7.10	480	8.9
6	1.5	60	150	9	14.03	15.92	392	11.5
7	1.5	70	120	5	9.92	9.60	440	9.7
8	1.5	60	90	5	7.6	8.60	465	8.7
9	1	60	120	9	10.2	13.26	402	10.8
10	1	70	120	7	16.42	15.92	385	11.8
11	1.5	50	120	9	14.08	12.92	410	10.1
12	1	60	90	7	9.25	10.42	418	10.4
13	1.5	60	120	7	9.98	10.20	406	10
14	1.5	70	150	7	18.08	16.86	362	11.6
15	1	50	120	7	7.2	6.50	490	8.9
16	1.5	50	150	7	14.8	13.50	409	11.5
17	1.5	70	120	9	19.22	21.56	320	12.8
18	2	60	150	7	15.98	17.58	368	11.4
19	2	70	120	7	17.96	19.31	344	12.1
20	1	60	120	5	7.2	5.80	515	7.5
21	2	60	90	7	18.62	16.20	389	11.8
22	1	60	150	7	15.20	14.00	395	11.7
23	2	50	120	7	9.8	9.00	465	9.4
24	1.5	60	150	5	17.44	18.80	356	12.5
25	1.5	50	120	5	7.5	6.20	495	8.2
Untreated	-	-	-	-	0.12	0.08	980	3.2

getting inactivated at pH above 9 and temperature above approximately 80 ºC. Reactive functional groups like -COOH and -OH were found to have been introduced to the polyester fabric during the FTIR analysis. Acid-base back titration techniques were used to quantify these reactive groups. It was discovered that the samples had almost the same concentration of -COOH and -OH groups. The hydrolysis of ester (-COO) groups produces -COOH and -OH groups in an equivalent molar ratio.

The Box-Behnken design was used to create several specimens, and Table 2 shows the outcomes for the various runs.

It was found that the highest numbers of -OH and -COOH groups (19.22 mmol/g and 21.56 mmol/g, respectively) developed at an Esterase concentration of 1.5% at 70°C, pH ~9 and 120 min. This treatment produced a wicking height of 12.8 cm and a comparatively improved absorbency time of 320 s, suggesting increased hydrophilicity in the polyester (Table 2, specimen 17). According to the absorbency and wicking height measurements, this change is most likely the result of these functional groups forming on the surface of polyester making the fibre reactive.

ANOVA analysis showed the p-value for Esterase (%), temperature (°C), and time (min), as <0.05, signifying the significance of these parameters, while pH showed its insignificance, which requires its elimination from the model (Table 3). This indicates that pH does not show a significant effect on the extent of development of reactive groups.

The number of reactive groups developed with changes in various parameters can be predicted as per the following regression equation:

After excluding insignificant factors, such as pH, from the regression equation,

$$\text{Reactive groups (mmol/g)} = 11.2 + 8.8A - 0.96B - 0.012C + 0.63D + 9.04A^2 + 0.0070B^2 + 0.002C^2 + 0.273D^2 - 0.053(A\times B) - 0.144(A\times C) - 1.51(A\times D) + 0.00172(B\times C) - 0.0340(B\times D) - 0.0296(C\times D) \quad \ldots(6)$$

The final equation stands as:

$$\text{Reactive groups (mmol/g)} = -21.62 + 4.93A + 0.2452B + 0.0994C \quad \ldots(7)$$

where A: Esterase (%), B: Temperature (°C), C: Time (min), and D: pH.

Fig. 2 shows the degree of reactive functional group development brought about by the interaction of Esterase concentration, time, temperature, and pH.

The rate of increase in formation of reactive groups increased with an increase in Esterase (%), temperature, and time, as shown in Fig. 2(a) and Fig. 2(b). The enzymes work better at a pH 9 as compared to pH 5 and 7 for lower concentration, but for higher concentrations i.e., 2% the enzyme works in almost a similar manner at any pH range from 5-9 as shown in Fig. 2(c).

The number of groups formed increased with an increase in temperature and time, and the rate of increase in the number of groups is found to be higher at higher temperatures (70 °C) [Fig. 2(d)]. At lower temperature (50 °C), the rate of increase with increase in pH is not as significant as found at higher temperature (70 °C), as depicted in Fig. 2(e). For a lower time period (90 min) the rate of development of reactive is found to be more with increase in pH from 5 to 9 but the rate remains almost same for higher time period (150 min) as depicted in Fig. 2(f).

Table 3 — ANOVA test results for Esterase-treated polyester

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Esterase, %	1	72.767	72.767	8.04	0.009
Temperature, °C	1	72.177	72.177	7.98	0.010
Time, min	1	106.624	106.624	11.78	0.002
pH	1	21.628	21.628	2.39	0.136
Error	23	208.118	9.049		
Lack-of-Fit	20	208.118	10.406	*	*
Pure Error	3	0.000	0.000		
Total	27	481.314			

In Table 2 (Sr. 1, 3, and 10), higher enzyme concentrations at pH 9 (alkaline conditions) resulted in fewer functional groups compared to 1% enzyme at neutral pH (pH 7). This shows that the outcome is not simply due to alkaline hydrolysis but rather the combined influence of enzyme concentration, time, temperature, and pH. Moreover, functional group development was observed across both acidic and basic pH ranges, further supporting the enzymatic effect.

3.4 Wicking Height

Table 2 presents the wicking height results of polyester fabrics treated with Esterase under varying concentrations and conditions. An increase in wicking height reflects enhanced hydrophilicity. The Esterase treatment modifies the PET surface by imparting an ionic character, thereby introducing polar groups on the inert polyester surface. This structural modification leads to improved absorbency. Compared to untreated polyester, all Esterase-treated samples exhibited a significant increase in wicking height, as shown in Table 2.

3.5 Absorbency

Absorbency time for all 25 specimens is shown in Table 2. All the treated specimens exhibited shorter

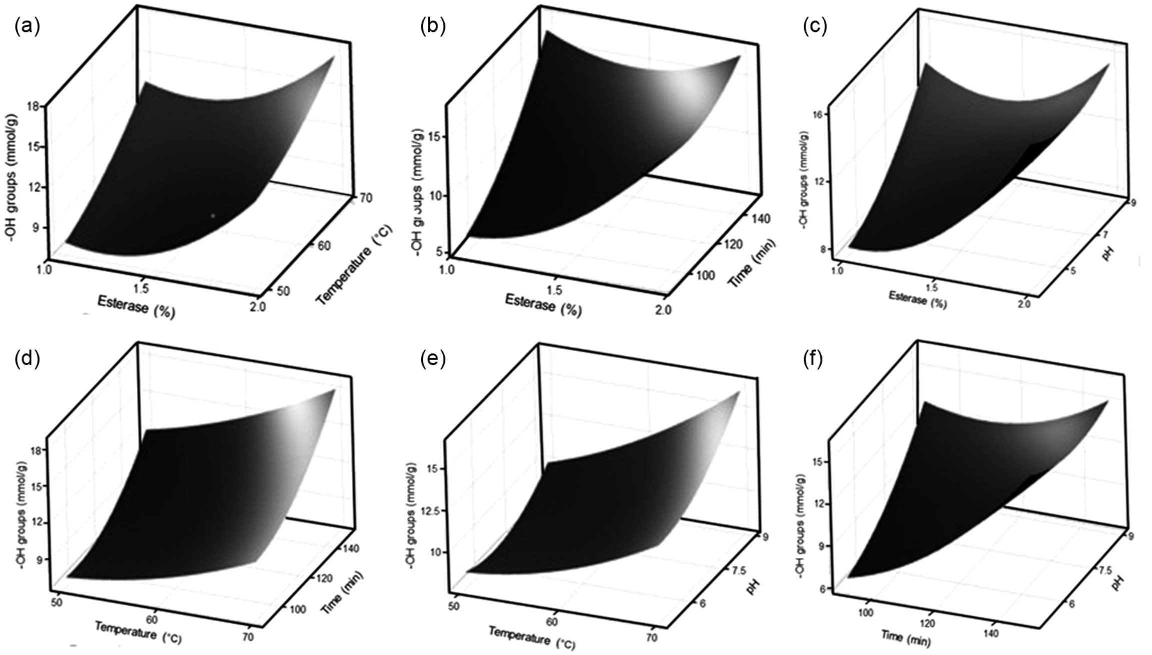

Fig. 2. — Response surface plots showing influence of various parameters on development of reactive groups on Esterase treated polyester (a) Esterase (%) and temperature (°C), (b) Esterase (%) and time (min), (c) Esterase (%) and pH, (d) temperature (°C) and time (min), (e) temperature (°C) and pH, and (f) time (min) and pH

absorption times than untreated polyester (980 s), as shown in Table 2. The higher the number of reactive groups formed on the polyester fabric surface, the superior the absorbency of Esterase-treated polyester.

3.6 Moisture Regain

The Esterase-treated polyester specimens exhibit a significantly higher moisture regain compared to the untreated specimens. Among them, specimen 17 shows the highest value, as shown in Fig. 3, which aligns with the data presented in Table 2, indicating the highest number of hydroxyl and carboxyl groups. These newly developed functional groups enhance the fabric’s affinity for water molecules, thereby improving moisture absorption.

Similar behaviour is observed in specimens 4, 19, and 24, further confirming the correlation between the extent of functional group development and the moisture management performance of Esterase-treated polyester fabrics

3.7 Loss in Tensile Strength and Weight

None of the treated specimens showed any sort of significant weight loss, as shown in Fig. 4**,** and strength loss in the warp direction, as shown in Fig. 5, suggesting that the polyester polymer chain had not ruptured, confirming the activity of Esterase on the surface of polyester only.

The tensile strength of the enzymatically treated polyester samples remains comparable to that of the untreated fabric, with no consistent trend of increase or decrease. The small variations observed are primarily due to the intrinsic characteristics of the fabric, including slight yarn irregularities, minor differences in filament orientation resulting from variations in the draw ratio during spinning, and variations in fabric construction. These natural variations account for the narrowly scattered strength values.

3.8 Water Contact Angle

The contact angle formed by a water droplet on the Esterase-treated polyester surface was smaller than on the untreated polyester, indicating that the Esterase-treated polyester's hydrophilicity had improved (Fig. 6). The polyester treated with Esterase (Table 2, specimen 17) had a contact angle of 59.3° [Fig. 6(a)], whereas the untreated polyester had a contact angle of 122.4° [Fig. 6(b)].

3.9 Thermal Characteristics

The Esterase-treated polyester specimen has a lower melting point (249.6°C) in comparison to the untreated polyester (255.1°C), as depicted in Fig. 7(a), indicating surface modifications or partial modifications in polymeric chains rather than a significant degradation in the crystallinity of polyester.

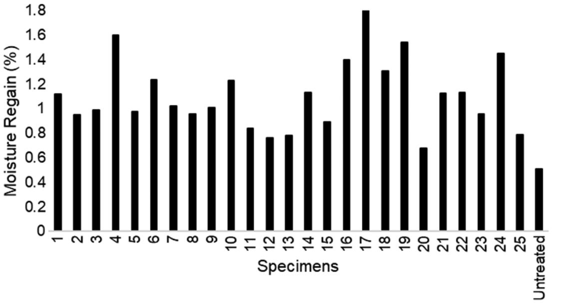

Fig. 3 — Moisture regain of Untreated and Esterase-treated polyester specimens

Fig. 4 — Weight of Untreated and Esterase-treated polyester specimens

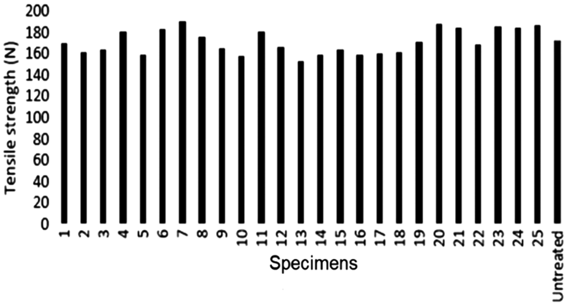

Fig.5 — Tensile strength results for untreated and Esterase-treated polyester specimens

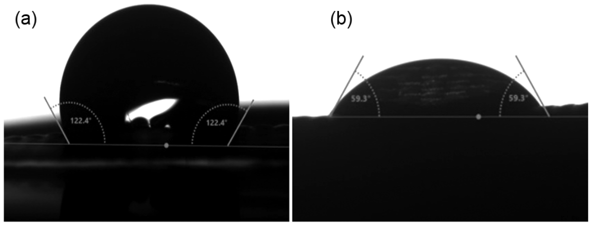

Fig.6 — Contact angle of (a) Untreated polyester, and (b) Esterase-treated polyester

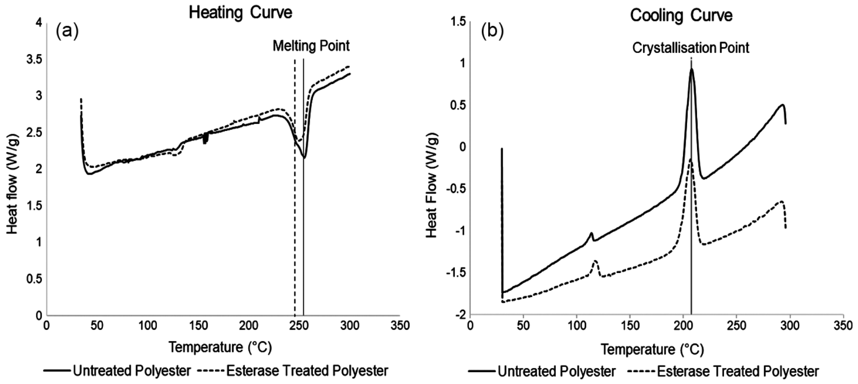

Fig. 7 — Differential Scanning Calorimetry results (a) Melting (heating curve), and (b) Crystallisation (Cooling curve) of Untreated and Esterase-treated polyester specimens

Table 4—Shades and K/S values for modified and dyed polyester and cotton specimens

Shade, %	Specimen	Red M5B		Yellow M3R		Magenta MB		Orange M2R		Blue MR	
		Shade	K/S	Shade	K/S	Shade	K/S	Shade	K/S	Shade	K/S
0.5%	Cotton	🟥	1.33	🟨	1.24	🟪	1.35	🟧	1.30	🟦	1.26
	Polyester	🟥	1.03	🟨	0.82	🟪	0.99	🟧	0.95	🟦	0.82
1%	Cotton	🟥	1.93	🟨	2.06	🟪	2.34	🟧	2.45	🟦	2.34
	Polyester	🟥	1.12	🟨	1.35	🟪	1.81	🟧	1.61	🟦	1.41
2%	Cotton	🟥	2.47	🟨	3.97	🟪	4.26	🟧	3.53	🟦	4.26
	Polyester	🟥	1.99	🟨	2.30	🟪	3.05	🟧	2.33	🟦	3.45
3%	Cotton	🟥	4.03	🟨	4.48	🟪	6.26	🟧	5.57	🟦	6.26
	Polyester	🟥	3.26	🟨	3.49	🟪	3.96	🟧	3.26	🟦	3.95
4%	Cotton	🟥	6.31	🟨	5.97	🟪	7.59	🟧	7.57	🟦	7.59
	Polyester	🟥	4.97	🟨	3.82	🟪	4..83	🟧	4.22	🟦	4.62
5%	Cotton	🟥	9.03	🟨	7.31	🟪	8.02	🟧	8.05	🟦	8.02
	Polyester	🟥	6.55	🟨	4.96	🟪	5.98	🟧	4.98	🟦	5.08

A slight decrease in cold crystallisation temperature is observed for Esterase-treated polyester (206.8°C) in comparison to untreated polyester (208°C), as depicted in Fig. 7(b), which indicates a delay in the crystallisation, which also supports that there is no significant change in the internal polymeric structure of the polyester rather the effect of enzymtic treatment limits upto the accessible ester bonds on the surface or in the amorphous regions.

3.10 Dyeing with Reactive Dyes

Five distinct cold-brand reactive dyes were used to dye cotton fabric specimens and 1.5% Esterase-treated polyester (specimen 17, Table 2). Table 4 shows the relative surface color strength (K/S) of the cotton and polyester dyed with these dyes for different shade percentages.

The glass transition temperature of PET (>70 °C) is relevant in disperse dyeing, where structural opening is required to provide adequate opening of the fibre structure to provide a passage for to disperse dye to enter inside followed by cooling for dye entrapment. In contrast, the enzymatic treatment introduces -OH and -COOH functional groups through hydrolysis of ester linkages. The development of these functional groups imparts ionic character to the inert polyester surface. As a result, reactive dyes can form interactions with the ionic polyester surface via covalent bonding (nucleophilic substitution), like reactive dyeing of cotton, allowing them to act as a potential substitute for non-ionic disperse dyes, which are applied in the conventional HTHP method by opening the polyester structure at very high temperature, enabling dyeing at around 40 °C without relying on the bulk glass transition temperature related to fibre opening.

In comparison to the identical shade percentage developed on cotton, it was observed that all the polyester specimens treated with Esterase were noticeably lighter in shade. In contrast to cotton, which has a huge number of -OH groups throughout its structure and can form covalent bonds with a large number of dye molecules, the modified polyester surface had a limited number of -OH groups not sufficient enough to develop comparable shades.

Table 4 shows the comparison of the hues produced with each dye on cotton and polyester treated with Esterase. The surface color strength of Red M5B, Magenta MB, and Blue MR was noticeably higher than that of Yellow M3R and Orange M2R.

Table 4 not only provides an idea of the relative shade depth (K/S) observed but can also be used as a result for shade matching. As shown in Table 4 and Table 5, comparable K/S values and fastness ratings were obtained at similar shade depths (e.g., Procion Red M5B at 2% on polyester vs. 1% on cotton, and at 5% on polyester vs. 4% on cotton). The comparison is made between reactive dyed cotton and polyester, as this provides an idea about the dyeing behaviour of polyester-cotton blends.

3.11 Evaluation of Color fastness

Dyed polyester specimens were evaluated for color fastness grades (Table 5).

Excellent color fastness grades were demonstrated by the modified and colored polyester, and they were nearly identical to those of cotton dyed with five distinct reactive dyes. This implies that the dye and the -OH groups on the surface of the modified polyester have reacted to form the fast shades.

The number of unreacted -OH and -COOH groups on the dyed specimens was assessed to further

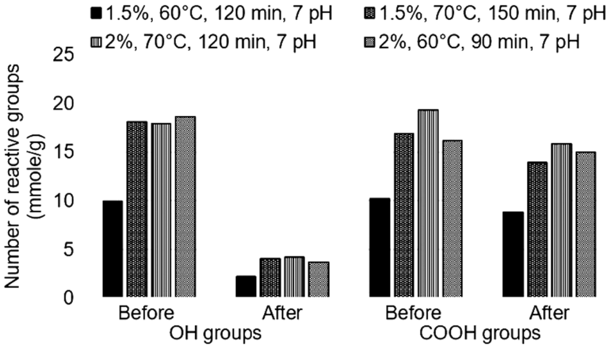

Fig. 8 — Change in the number of reactive groups on dyed polyester after dyeing

Table 5 — Color fastness grades for reactive dyed Esterase-treated polyester

Dyed Specimens	Rating Scale	Rubbing Wet		Rubbing Dry		Light		Wash		Sublimation	
		Cotton	PET	Cotton	PET	Cotton	PET	Cotton	PET	Cotton	PET
Procion Red M5B	Staining on cotton	4	4-5	4	4-5	-	-	4	4	4	3-4
	Change in color	4	4-5	4-5	4-5	6	6-7	4	4-5	4	4
Yellow M3R	Staining on cotton	4	4-5	4-5	4-5	-	-	4-5	4	4-5	4-5
	Change in color	4-5	4-5	4-5	4-5	6	6-7	4	4	4-5	4
Blue MR	Staining on cotton	4	4-5	4-5	4-5	-	-	4	4-5	4-5	3-4
	Change in color	4	4-5	4-5	4-5	6	6-7	4-5	4	4-5	4
Magenta MB	Staining on cotton	4	4-5	4	4-5	-	-	4	4-5	4	3-4
	Change in color	4	4-5	4-5	4-5	6	6-7	4	4	4	4
Orange M2R	Staining on cotton	4	4-5	4	4-5	-	-	4-5	4	4-5	4-5
	Change in color	4-5	4-5	4-5	4-5	6	6-7	4	4-5	4-5	4

Table 6— Shades and K/S of dyed polyester specimens pretreated with Esterase at different concentrations with cotton dyed with 5% shade

Esterase, %	Red M5B		Yellow M3R		Magenta MB		Orange M2R		Blue MR	
	Shade	K/S	Shade	K/S	Shade	K/S	Shade	K/S	Shade	K/S
5%	🟥	3.12	🟨	1.32	🟪	2.13	🟧	1.24	🟦	1.35
10%	🟥	3.87	🟨	2.65	🟪	3.19	🟧	2.35	🟦	2.82
20%	🟥	4.48	🟨	3.21	🟪	3.85	🟧	3.22	🟦	3.65
30%	🟥	5.33	🟨	3.79	🟪	4.82	🟧	3.96	🟦	4.99
40%	🟥	5.93	🟨	4.82	🟪	5.16	🟧	4.65	🟦	5.24

validate the dye-fibre attachment via the -OH or -COOH groups on modified polyester, as illustrated in Fig. 8. While there was no discernible change in the number of -COOH groups on the polyester surface, it was found that between 70 and 75 % of the -OH groups were consumed during dyeing. Furthermore, -OH groups are more efficient nucleophiles than -COOH groups and reactive dyes create covalent bonds with cotton by nucleophilic substitution or addition processes.

At this stage, it was thought that a different strategy to improve surface color strength might involve developing more -OH groups to polyester treated with Esterase at higher concentrations. Therefore, while maintaining other parameters unchanged, polyester was treated with Esterase at concentrations of 5%, 10%, 20%, 30%, and 40% (specimen 17, Table 2).

The contact angle was measured for Esterase-treated polyester specimens as 39.6°, 31.7°, 29.5°, 29.3° at higher concentrations viz 10%, 20%, 30%, and 40%, respectively.

Without compromising the strength of Esterase-treated polyester, it was found that gradually increasing the Esterase concentration significantly improved the depth of shade (Table 6). The increase in -OH groups with greater Esterase concentrations was credited with this improvement.

3.12 Energy and Water Consumption and Processing Cost Analysis

The conventional dyeing of polyester with disperse dye using the HTHP method in jet dyeing machines consumes almost 664910 kJ of energy to dye 100kg of fabric. But the enzymatic treatment followed by the dyeing with cold brand reactive dye consumes only 418140 kJ of energy for 100kg of fabric. For polyester cotton blends, the energy consumption in the conventional two-bath two-step method was 673326 kJ, but after enzymatic treatment, it was reduced to 280940 kJ.

When water consumption is considered, it is maximum in the conventional two-bath, two-step dyeing process for polyester-cotton blends, followed by polyester dyeing with disperse dyes, then by enzymatic treatment, followed by dyeing with reactive dyes for polyester-cotton blends, and minimum water is consumed for dyeing of cotton with reactive dyes. In terms of effluent treatment, minimum resources are required for effluent from reactive dye baths, as they are self-hydrolysable, whereas disperse dye effluents require more resources for proper treatment.

The cost of the enzyme used in our study was approximately Rs. 500-700 per litre. While this does add to the processing expense, conventional polyester dyeing involves several significant costs, including high thermal energy consumption due to high dyeing temperatures, investment in specialized high-temperature machinery, use of disperse dyes and auxiliaries, effluent treatment costs, and, in the case of polyester-cotton blends, the need for two separate dyeing processes, which further increases time and expense. In contrast, enzymatic treatment followed by reactive dyeing offsets the enzyme cost because the process can be carried out at much lower temperatures with reduced energy consumption, does not require specialized machinery, as it can be carried out in an open bath, and uses reactive dyes that are self-hydrolysable and therefore less demanding on effluent treatment. Moreover, for polyester-cotton blends, the dyeing can be accomplished in a single step, first by pretreating the blend with Esterase to develop functional groups on polyester, followed by dyeing with reactive dye in the same way done for cotton, thus saving time, energy, and cost. Thus, despite the added enzyme expense, the process offers clear economic and environmental viability compared to conventional polyester dyeing.

4 Conclusion

Under optimal esterase concentration, pH, temperature, and time, Esterase treatment develops both -OH and -COOH groups on polyester without compromising tensile strength, as verified by FTIR results demonstrating the successful application of Esterase to enhance the wettability of polyester. Compared to untreated polyester, the treated polyester also exhibited better absorbency and reduced wicking height and contact angle without any significant loss in strength and weight. Mechanical and thermal characterisation verifies that the process is not affecting the bulk or the internal structure of the polyester fabric. A higher concentration of Esterase significantly improved the surface color strength of Esterase-treated polyester, increasing the potential for dyeing polyester with reactive dyes, although the surface color strength of dyed polyester was lower than that of cotton. Additionally, it was noted that the reactive dye groups formed covalent bonds with the -OH groups of the treated polyester. Excellent color fastness was even demonstrated by the Esterase-treated polyester dyed with reactive dyes. Energy consumption was reduced by 50-60% compared to conventional dyeing of polyester with HTHP dyeing machines. Therefore, Esterase treatment of polyester provides a viable alternative to conventional high-temperature, high-pressure dyeing methods using disperse dyes, which require expensive jet dyeing equipment. Further testing is necessary to enhance this novel dyeing technique and make it suitable for commercial application.

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