Role of Biotechnology in the Treatment of Polyester Fabric

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Abstract Poly (ethylene terephthalate) fibre [PET] is the commonly used fibre for majority of end-use applications, however, the desire for improved textile properties such as wettability or hydrophilicity are increasing. Biotechnology can be defined as the application of scientific and engineering to the processing of materials by biological agents to provide goods and services. The environmental issues associated with the textile processing are not new. Currently and in the years to come, besides lower cost of operation, improved durability, wear comfort and development of new attributes for textiles, the new criteria for judging the new processes is ecology. This paves the way for biotechnology. This article throws light on the applications of enzymes for the treatment of polyester fabrics.

Keywords PET · Hydrophilicity · Biotechnology

Introduction

Green chemistry using biotechnology has joined incredible importance in the textile wet processing industry. The search for new, efficient and eco-friendly alternatives have increased interest in using green catalysts i.e., enzymes. Developments in genetic engineering bring about improvements in the stability, specificity, economy as well as overall application potential of industrial enzymes in textile finishing. Over the past four to five decades not only is the demand for poly (ethylene terephthalate) [PET] textile fibres

increasing, but also the desire for improved textile properties such as wettability or hydrophilicity. Furthermore, effects like better dyeability with water soluble dyes or surface functionalization for special purposes like coupling of flame retardants are desirable from the perspective of the industrial textile industry. Besides textiles, enzymatic modification of synthetic materials has immense potential for specialty applications such as in medical devices and electronics [1, 2].

In the recent years there have been major research efforts to find enzymatic technologies to meet the demands of industry. These innovative technologies show the advantage of lower energy consumption and avoid the use of harsh chemicals. When compared to chemical surface treatment enzymes are preferred for their fast reaction rate, highly specific in action at mild conditions, safe and easy to control [3, 4]. The particular benefits offered by enzymes are specificity, mild conditions and reduced waste. It may be possible by choosing the right enzyme, to control which products are produced and unwanted side reactions are minimized due to the specificity of enzymes that appear in the waste stream. The plant using enzymatic reactions can be built and operated at much lower capital and energy costs. Enzyme-based processes tend to have lower treatment costs. Enzymes, however, are biodegradable, and since they usually are dosed at 0.1–1.0% of the substrate, the contribution of the enzyme to the BOD in the waste stream is negligible [5].

Enzymes

Enzymes are versatile biocatalysts, capable of catalyzing diverse and unique reactions that are highly specific in their catalytic mechanism. Enzymes are complex protein ferments secreted by living organisms (bacteria, fungus, animals and plants). The proteins are composed of amino-acids

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with a variety of side chains ranging from non-polar aliphatic and aromatic to acidic, basic and neutral polar groups [6].

Mode of Action

Enzymes, due to their 3-dimensional complex structures have so called ‘active sites’, hollow spaces or columns containing binding and catalyzing groups, which have the ability to bind certain substrates in lock and key fashion, as well as to facilitate all ranges of micro environments for all catalysis. The actual nature of the reaction in the enzyme’s active sites is determined by the enzyme class, and can be described as follows:

Table 1 Enzymes having textile applications

Sr. no.	Enzyme class	Types of reaction catalyzed by
1.	Hydrolases	Hydrolysis of a molecule and degradation in same cases
2.	Lyases	Non hydrolytic cleavage of molecules
3.	Transferases	Transfer of a group from one molecule to another
4.	Oxidoreductases	Oxidation or reduction of a molecule
5.	Isomerases	Conversion of one isomer to another
6.	Ligases	Joining of two molecules

Table 2 Enzyme type: hydrolases [8] and oxidoreductases [9]

Sr. no.	Enzyme name	Substrate attacked	Textile application
1.	Amylase	Starch	Starch de-sizing
2.	Cellulase	Cellulose	(i) Stone-washing, bio-polishing (ii) Bio-finishing for handle modification (iii) Carbonization of wool
3.	Pectinase	Pectin	Bioscour replacing caustic
4.	Proteases	Protein molecules or peptide bonds.	(i) Degumming of silk (ii) Bioantifelting of wool.
5.	Lipases	Fat and oils	Improve hydrophilicity of PET in place of alkaline hydrolysis
6.	Laccase	Color chromophore and pigments	(i) Discoloration of coloured effluent (ii) Bio-bleaching of lignin containing fibres like kenaf and jute. (iii) Bio-bleaching of indigo in denim for various effects
7.	Peroxidases	Color chromophore and pigments	Bio-bleaching of wood pulp
8.	Glucose oxidases	Color chromophore and pigments	In situ generation of H ₂ O ₂ and bio-bleaching of cotton.
9.	Azoreductase	Color chromophore and pigments	Discoloration of azo dyes effluent
10.	Peroxidase ostreatus	Color chromophore and pigments	Discoloration or removal of basic dye from effluent.
11.	Ligninase	Color chromophore and pigments	Removal of metal ions and toxins from effluent

The binding and the catalytic groups of an enzyme E, combine with the substrate S, to form an intermediate-enzyme complex ES, and within this complex a series of atomic and electronic rearrangements occur followed by the releasing of the enzyme along with one or more product on substrate decomposition [7].

Enzyme Performance

Factors affecting the performance of an enzyme catalyzed reaction are its specific pH, bath temperature, incubation period, substrate binding specificity, presence and absence of inhibitors such as metals, ionic detergents, CH₂O—based products or activates e.g., Vitamins, ascorbic acid, Ca²⁺ etc.

Enzymes Having Textile Applications

The current applications in the textile industry involve mainly hydrolases and to some extent oxidoreductases. The Table 1 and 2 exemplify such textile applications.

Microbial enzymes are often more useful then enzymes derived from plants or animals because of the great variety of catalytic activities available, the high yields possible, ease of genetic manipulation, regular supply due to absence of seasonal fluctuations and rapid growth of micro organisms on inexpensive media. Microbial enzymes are also more stable than their corresponding plant and animal enzymes and their production is more convenient and safer [10]. Only about 2% of the world’s microorganisms have been tested as enzyme sources. Bacterial strains are

generally more used as they offer higher activities compared to yeasts [11] and tend to have neutral or alkaline pH optima and are often thermo-stable. Genetic and environmental manipulation to increase the yield of cells [12], to increase the enzyme activity of the cells by making the enzyme of interest constitutive, or by inducing it or to produce altered enzymes [13], may be employed easily using microbial cells because of their short generation times, and since screening procedures for the desired characteristic are easier.

Use of Lipases In Textile Industry

Lipases, particularly of microbial origin constitute an important group of bio-technologically valuable enzymes mainly because of their versatility and ease of mass production. Lipases play important roles in the textile industry as mentioned here in:-

- 1) *De-sizing of Textile*: Amylases are used to remove starch-based size for improved and uniform wet processing. Incorporation of lipase along with amylase in de-sizing bath results in complete removal of lubricants, shortening the time of de-sizing as well as giving better quality with respect to uniformity and hand [14].
- 2) *Bio-scouring*: Non-cellulosic impurities such as fats, waxes, proteins, pectins, natural colourants, minerals etc. are found to a large extent in the primary walls of plant cells. Bio-scouring is based on the idea of specially targeting the non-cellulosic impurities like waxes, fats, oils, pectinic, substances with appropriate enzymes. Use of mixed enzyme like pectinases (for pectin) and lipases (for fats) can be very effective and environmentally friendly treatment for attaining better wettability, without adversely affecting the mechanical properties of treated substrate [15].
- 3) Lipase treatment of wool fabric enables reactive dyeing of wool under mild conditions with increasing rate of dyeing and extent of exhaustion, thereby achieving energy saving and reducing pollution impact [16].
- 4) Lipase catalyzed polymerization provided an eco-friendly process for synthesis of biodegradable polyester from reaction of poly-anhydride derivatives with glycols in water-toluene or supercritical carbon-dioxide. The reaction behaviour depends on the starting materials and reaction media [17].
- 5) Pretreatment of polyester with lipase results in an improvement in its wettability, water penetration, absorbency as well as disperse dye uptake, while the fabric strength was retained [18].

Polyester Fabric Modification by Some Lipases

Potentially a great variety of enzymes can be used to modify the surface of polyethylene terephthalate. Among these the most important are the esterases, lipases and cutinases. The enzymatic hydrolysis of polyester linkages produces polar hydroxyl and carboxylic groups on the surface [19, 20].

Heish and Gram [21] also reported the production of hydroxyl and carboxylic groups due to the hydrolysis of ester linkages in PET and that the enzymatic surface hydrolysis has the advantage of maintaining mechanical stability because the enzyme cannot penetrate the fiber and hence is restricted to reacting on the surface only, thereby increasing the fabric surface wettability. They studied the ability of six hydrolyzing enzymes to improve the hydrophilicity of several polyester fabrics including sulfonated polyester and microdenier polyester fabrics. Five of the six lipases significantly improved the water wetting and absorbent properties of regular polyester fabrics, and they improve water wetting and water retention more than alkaline hydrolysis.

For instance a 10 min reaction (1 g/l, pH 8.0, 35°C) reduced the water wetting contact angle of regular PET from 75.8 to 38.4° and increased water retention from 0.22 to 1.06 µl/mg. Alkaline hydrolysis of PET fabric under the optimal conditions (3 N NaOH at 55°C for 2 h) produced a water contact angle of 65 to 0° and a water retention value of 0.32 µl/mg. Improved water wettability was accompanied by full strength retention compared to the significantly reduced strength and mass from alkaline hydrolysis. The wetting and absorbent properties of sulphonated polyester and microdenier polyester fabrics are also improved by lipase. Hsieh and Gram also optimized the reaction conditions of two of the lipases they worked with. The enzyme reactions were effective under moderate conditions, including a shorter reaction time (10 min), at ambient temperature (25°C) and without a buffer.

Improved oily stain resistance, wettability, dyeability, high cationic dye binding, removal of polyester size, de-pilling and reduced fibre luster of poly (ethylene terephthalate) fabrics treated with polyesterase—a serine esterase was reported by Yoon et al. [2]. Oxidative enzymes such as laccases have also been shown to hydrophilize the PET surface [22] although oxidative modification with laccases would be especially interesting since functionalization could be achieved without cleavage of the polymer. There is no detailed mechanistic or application related data available.

However, there are a number of reports on the hydrolysis of synthetic aliphatic polyesters while the aromatic polyesters seem to be more recalcitrant to microbial enzymatic attack [23–28]. Walter et al. [24] studied the

enzymatic degradation of a model polyester i.e., lipase obtained from *Rhizopus deleamar*. They standardized an enzyme assay for the use of insoluble substrates which gave reproducible data. Ester bond cleavage was measured with respect to time comparison of ester cleavage and weight loss which indicated that oligomers with an average length of 5–6 monomers are released from polymer bulk. In some of his early studies performed by Tokiwa and Suzuki, reports are made on lipases and esterases that can hydrolyze polyesters. Poly(ethylene adipate), poly(caprolactone), poly(cyclohexylene dimethyl trimethyl isophthalate) and poly(2,2-dimethyl trimethylene isophthalate) were used as substrates on which the effect of enzyme preparations from *Achromobacter species*, *Candida cylindracea*, *Geotrichum candidum*, *Rhizopus arrhizus*, *Rhizopus deleamar* and hog liver esterase was studied. The hydrolysis of polyesters was determined by measuring the water-soluble total organic carbon (TOC) in the reaction mixture. *Rh. arrhizus* and *Rh. deleamar* lipases showed especially strong activity [29].

Fischer-Colbrie et al. [30] carried out screening processes for organisms that had a potential to modify the surface of polyethylene terephthalate. The screening was carried out using a mineral medium have PET polymer powder as the sole source of carbon, inoculated with soil sample and incubated at 30°C. After enrichment pure strains were isolated on PET containing media and media containing a PET model substrate i.e., bis (benzoyloxy-ethyl) terephthalate. Out of the screening processes, four bacterial and five fungal strains were isolated. The extracellular enzymes from these isolates showed a good activity of the model substrate. All the enzyme preparations showed esterase activity on *p*-nitrophenyl acetate while no activity was found on *p*-nitrophenyl decanoate or *p*-nitrophenyl palmitate. The enzyme preparations from the isolates were used for enzymatic treatment of polyester fabrics and the treated fabrics were tested for changes by the drop test and rising height measurements, both which are measurements of hydrophilicity of PET fabrics. Enzyme preparations of some of the isolates showed on significant change in the hydrophilicity of the fabric.

The use of esterases (closely related to lipases) to improve the ability of a polyester fabric to uptake chemical compounds, such as cationic compounds, fabric finishing compositions, dyes, antistatic compounds, anti-staining compounds, antimicrobial compounds, antiperspirant compounds and/or deodorant compounds is reported [31].

Vertommen et al. confirmed the hydrolysis of poly(ethylene terephthalate) using cutinase from *Fusarium solani pisi* and lipase A from *Candida antartica* on PET. The extent of hydrolysis was detected by measuring the amount of soluble degradation products in solution using reversed phase HPLC. The cutinase from *F. solani pisi* had significant hydrolytic activity towards amorphous regions

of PET. No hydrolysis activity was, however, demonstrated by Lipase A from *C. antartica* [32].

The effect of three different lipases was studied on polyester fabric. The lipases were from *Penicillium roquefortii*, *Candida cylindracea* and *Porcine pancrease*. The treatment was carried out for varying times at 33°C and pH 7.2. The enzyme treatment causes significant improvement in water penetration and the absorbent properties of regular polyester fabric without causing major damage to the mechanical properties of the fabric. SEM studies showed modification of the surface of the treated fibres [33].

Mark et al. noted on increase in the hydrophilicity of PET fibres after treatment with hydrolases from *Thermomonospora fusca* and *Fusarium solani pisi*. Water sorption and dyeing increased. Reflectance spectrometry showed stepwise peeling of fibres by enzyme treatment comparable to alkali treatment [34]. Similarly Gouda et al. [35] studied the production of extracellular hydrolase, from the thermophilic actinomycete, *Th. fusca* in the presence of random aliphatic aromatic co-polyester from 1,4-butanediol, terephthalic acid, adipic acid, with around 40–50 molecular percent of terephthalic acid, in a synthetic medium with pectin and ammonium chloride as the N-Source.

Cultivation of isolates of *Thermomonospora fusca* in media containing polyethylene terephthalate yarns and ‘suberin’ a plant polyester composed of aliphatic and aromatic moieties, induced the production of *p*-nitrophenyl butyrate hydrolyzing enzymes. Incubation of these enzyme preparations with polyethylene terephthalate yarn resulted in an increase in the absorbance of reaction mixture at 240 nm indicating the release of terephthalic acid or its esters catalyzed by the enzyme. Dyeing of the enzyme treated PET fabrics with a reactive dye C I Reactive Red 2 indicated an increase in hydroxyl groups at the fibre surface due to enzyme treatment [36].

Commercial lipase and laboratory produced lipase enzyme from *Penicillium* sp. *OILI* was used to treat 100% polyester fabric. The treated fabric showed better sorption and dyeing properties. The water contact angle is the same for the treated and the control fabric. Moisture regain and vapour permeability of the treated fabric good. Dyeing studies showed better exhaustion of the dye from the dyeing bath. Since enzymes are natural agents acting under mild conditions changing the surface structures of highly hydrophobic polyester fibers, similar processing offers a potential possibility for the application of such enzyme systems in bio-degradation of polyester products [37].

The use of cutinase B from *Humicola insolens* and Laccase from *Streptomyces Coelicolor* (ScL) for increasing the hydrophilicity of polyethylene terephthalate fabric was explored. A Launderometer and 100% polyethylene terephthalate sample was used to mimic the industry process. The wettability was found by drop test. Cutinase B + 0.1%

non-ionic surfactant Triton X 100, imparted durable hydrophilicity to the polyethylene fabric when applied at 55°C, at pH-8 for 2 h at 5 µl/ml dose. ScL combined with a mediator Denilite II Assist and Triton X-100 noticeably increased polyester hydrophilicity at dosage above 0.048 LAMV/MI [38].

Kim and Song used lipase from porcine pancreas on polyethylene terephthalate fabrics and optimized the condition for increasing the hydrophilicity of PET fabric by lipase treatment. A pH-7.5, temperature-40°C, time-90 min and concentration of 6.25 g/l were optimum conditions. The enzyme activity was evaluated by the number of carboxylic group using titration method. SEM studies on the fabric showed voids and cracks which are responsible for the water-related properties [39].

In another study performed by the same workers with the aim of increasing the moisture regain of PET fabric using lipase treatment, nine lipase sources were used. The effect of lipase activator and non-ionic surfactant on moisture regain of PET fabric was studied as well as a comparison was done with alkali treatment of PET fabric. SEM studies of alkali treated PET showed large pits, however, the moisture regain does not improve much [0.568 + 0.08]. But with lipase treatment moisture regain increases up to 2.4 times [1.272 + 0.05] and SEM studies show a moderate etching of the fabric. The k/s values of the lipase treated samples increased confirming that the –COOH and –OH groups are produced [40].

In a study performed by Wavhal, a novel treatment was given to polyester fabrics in a microbial consortium under anaerobic conditions which brought about significant surface etching under anaerobic conditions with improved moisture regain. The microbial consortium was maintained under anaerobic conditions and lipid rich substrates were added to the microbial consortium in order to enrich the growth of lipolytic organisms which are expected to release lipases that can act on ester linkages of the polyester fabric. The treatment with and without chemical pretreatments with ethylamine indicated that pretreatment gave optimum results. The moisture regain and the ‘total hand value’ as found by the Kawabata evaluation System have increased from 0.2% of control to 1.38% and from 2.8 of control to 4.0, respectively. The improvement in hydrophilicity was also shown in terms of increase in wicking height and soil release properties. The dyeing characteristics with disperse dyes, basic dyes and natural dyes show an increment in the k/s values. Dyeing with the basic dye, Basic blue is four times more than the control. SEM photographs show that the fabric surface has changed with pits and pores which increased the surface area for absorption of water molecules. With anaerobic treatment the tensile strength is almost the same, however, with the combined treatment it is reduced. The microbial consortium containing a cocktail

of enzymes proved successful in bringing out surface etching of the fabric. The process is simple, cost effective, worked at room temperature, and did not require pH and temperature controls. It being an eco-friendly process and did not add any toxic effluents to the ecosystem. The scale-up trials were possible, economical and gave reproducible results [41].

In a study performed by Lee and Song enzymatic hydrolysis of polyethylene terephthalate fabric was carried out by using lipase and cutinase. The optimum conditions for enzyme reaction were determined. Lipase worked at a pH of 4.2, temperature of 50°C and time 90 min while for cutinase the optimum reaction conditions were pH-9.0, temperature 50°C. Both the enzyme reactions reduced the water contact angle significantly [42].

Ebert et al. [43] used lipase from *Thermonospora lanuginosus* and cutinase from *Thermomonospora fusca* and *Fusarium solani* to hydrolyze polyethylene terephthalate. Action of lipase in the presence of Triton-X-100 resulted in the formation of novel polar groups that enhanced the dyeability to as much as 130% for cutinase and 300% in the case of lipase. In a study performed by Donelli et al. [44] enzymatic surface modification of polyethylene terephthalate was carried out and studies were carried out to determine the water contact angle, FTIR and fluorescence spectroscopy.

Khoddami et al. [45] studied the enzymatic hydrolysis of polyester fabrics using different time, temperature and concentrations of enzyme. The FTIR studies showed increase in hydroxyl groups. Also moisture regain of treated fabric increased whereas weight loss, tensile strength and thickness changes were negligible.

Bruekner et al. [46] compared the alkaline and enzymatic hydrolysis of PET fabrics based on released degradation products assayed by HPLC and changes in surface properties affecting hydrophilicity, cationic dyeing and X-ray photoelectron spectroscopy (XPS).

Enzyme hydrolysis led to enhanced water absorbency and dyeability, whereas to reach similar benefits in hydrophilicity, drastically higher amounts of degradation products were released during alkaline hydrolysis as also indicated by more than 6% weight loss compared to less than 1% after enzymatic treatment. SEM images demonstrated that alkali treatment drastically affected the surface morphology of the polymers resulting in crater-like structures on the fibres while after enzymatic treatment the morphology of the fibre remains unchanged.

The optimization of cultivation conditions for lipase production and the preparation of a specific lipase catalyzing the hydrolysis of polyethylene terephthalate by *Aspergillus oryzae* CCUG338R as well as modification of polyethylene terephthalate fabrics by the enzyme was investigated by Wang et al. [47] The lipase activity

produced by the fungus after the addition of olive oil was not sufficient in changing the properties PET and therefore to induce lipase activity two derivatives of terephthalic acid (TPA) namely diethyl-*p*-phthalate (DP) namely bis (2-hydroxyethyl) terephthalate (BHT) and PET short fibres were used as inducers. The result showed that BHT was the best inducer. The BHT induced extracellular lipase could catalyse the hydrolysis of the PET model substrate. The formation of new carboxyl groups is consistent with the increase in *k*/s values of the dyed PET fabrics after the enzymatic treatment. Treatment with BHT induced lipase resulted in increased moisture regain and weight loss of PET fabric while water contact angle and static half decay time reduced slightly. Thus, hydrophilicity and anti-static ability were improved after treatment.

In a recent study performed by Feuerback et al. enzyme preparation from *Thermobifida fusca* KW3b was used to treat knitted fabrics made from PET. The fabrics were also treated with sodium hydroxide and the results were compared. Both enzyme and alkali treated fabrics showed an increase in reactive dye uptake, vertical wicking height and water absorbance capacity indicating an increased surface hydrophilicity. However, X-ray photoelectron studies (XPS) and energy dispersive spectroscopy (EDS) did not give results on the presence of introduced hydrophilic groups on the surface of fibres. SEM and atomic force microscopy indicated increase in surface roughness of fibres which may contribute to the observed increase in hydrophilicity. However, much longer treatment time (24 h) was required to obtain this effect with the enzyme as compared to chemical treatment (1 h) [48].

Bio-polishing is a finishing process in which a fabric is treated with an enzyme to impart properties such as anti-pilling, softness and smoothness. In a study performed by Stefanie et al. a cutinase was used for bio-polishing of polyester fabrics and can be combined with compatible cellulose to treat polyester and cotton blended fabrics. Two cutinases were investigated and one cellulase were tested separately and in combination with 50%/50%—polyester/cotton blend knit fabric following enzymatic treatment, weight loss, high performance liquid chromatography (HPLC) analysis of the treatment liquor and pilling note were evaluated. An improvement in pilling note for both polyester and polyester blends was demonstrated. Additionally, HPLC analysis of the treatment liquors indicated polyester hydrolysis due to cutinase activity which correlated well with pilling note results [49].

A patent published by Andersen et.al. [50] describes a method to reduce pilling propensity and improve colour clarification by enzymatic treatment on polyester fabrics in the presence of detergents.

Conclusion

Surface wettability of textiles is a key factor in many process techniques and end use characteristics. The enzymatic treatments can impart many desirable properties to the fabric such as, increased moisture regain, soil release properties, wear comfort properties, soft and smoothness, air permeability and increased dye uptake. These innovative technologies besides lower energy consumptions and avoidance of use of harsh chemicals are fast in their reaction rates, highly specific in action at mild conditions and safe and easy to control.

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