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# Effect of enzyme treatment on wool fabric properties and dimensional stability

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In this study, the merino wool woven fabric has been treated with commercially available enzymes, i.e. transglutaminase, lipase, laccase and protease, at various concentrations (0.5–2.0% over the weight of fabric) to impart desirable shrink resistance without deterioration of the fabric properties. Protease enzyme treated wool fabric shows least area shrinkage (3.0%) followed by laccase enzyme (4.3%), lipase enzyme (4.9%) and transglutaminase enzyme (7.9%) treated fabrics, as compared to 13.3% of the untreated (blank) fabric. The specific reaction mechanism of various enzymes that cause a structural change and dimensional stability are also discussed. The tensile strength, extension-at-break, yellowness and whiteness indices of the enzyme treated fabrics are found comparable with the blank fabric, while frictional and handle properties are significantly improved. The enzyme process to impart shrink resistance to wool fabric is found sustainable, easy to scale up and due to comparable mechanical, frictional, handle, whiteness and yellowness properties, there is a potential of an industrial adaption.

**Keywords:** Anti-shrink property, Cuticle scales, Dimensional stability, Enzyme treatment, Surface modification, Wool fabric

## 1 Introduction

Wool fabric is known for its luxury and unique properties, such as breathability, excellent thermal insulation, flame retardancy, thermoregulation and comfort properties[1,2]. However, poor dimensional stability is one of the major limitations of wool fabrics. The poor dimensional stability is caused by the progressive fibre entanglement in the wool fabric after repeated launderings[1,3–5]. The fabric shrinks during washing due to felting, which is unique to wool. The felting is a directional friction effect at the cuticle layer of fibre microstructure. Wool fibre microstructure comprised of cuticle, cortex, and medulla. The cuticle is a hydrophobic outer surface, consists of overlapping scales. During laundering, scales get interlocked with each other, resulting in irreversible shrinkage to the fabric[4].

The range of treatments has been investigated to make the wool fabric machine washable and shrink resistant. These treatments include oxidation, chlorination, enzyme action, radiation, polymer coating and plasma treatment[6]. Among all, the combination of chlorination and polymer coating (chlorine-Hercosett) treatment has been more effective, cheapest and energy-efficient. However, the process affects handle properties of wool fabric and it releases absorbable organic halides (AOX) into the environment[7–9]. Alternate sustainable treatments, such as UV-irradiation, ozone oxidation, and enzyme treatments, have also been tried[6]. UV-irradiation and ozone treatment can cause severe damage to the wool fibre and yellowing of the fibre is another drawback. Despite slow speed, enzyme treatment looks promising, especially to retain the original properties of the wool fabric.

Enzymes have been remarkably accepted in diverse sectors including textiles owing to their substrate specificity and green chemistry[10–12]. Enzymes are biodegradable natural macromolecules and its treatment to textiles is pollution-free[3,13]. Wool specific enzymes preferentially attack the disulphide bonds which impart hydrophilicity and shrink resistance to wool fabrics[14]. Protease enzyme treated wool fabric showed good shrink resistance without much loss of strength[15]. Enzymes like transglutaminase, laccase and protease have been studied for shrink resistance[6,16–20]. However, the reaction mechanism differs from enzyme to enzyme. Very limited information is available on enzyme-specific action on wool fabric at similar processing conditions. The effect of enzyme treatment on tensile strength,

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friction, colour indices and handle properties of the wool fabric has not been studied in detail.

In this study, the woollen fabric has been treated with transglutaminase, lipase, laccase and protease enzymes at three different concentration levels 0.5, 1 and 2% (over the weight of the fabric) using similar processing conditions. The enzymes are *p*H specific. The *p*H level for transglutaminase, lipase, laccase and protease enzymes are 7.0, 8.5, 4.5 and 8.5 respectively. The shrink resistance at each level is measured and compared with the blank fabric (wet treated without enzyme). The concentration of enzymes at which the least shrinkage is obtained (2% for transglutaminase and laccase whereas 1% for lipase and protease enzymes) has been considered for further experimentation. The surface morphology of the enzyme treated fabric is studied using Field Emission Scanning Electron Microscopy (FE-SEM). The effect of enzymes on moisture, tensile, friction and bending properties of wool fabric is determined. The effect on yellowness and whiteness index of the fabric due to enzyme treatment is also studied and compared with the scoured fabric.

## 2 Materials and Methods

### 2.1 Materials

Merino wool fabric was procured from the local market in Ludhiana, India. The woven fabric was made up of 19 µm wool. The fabric specifications were as follows: basis weight 146 g/m$^2$, ends/inch (EPI) 52, picks/inch (PPI) 54, warp yarn count 46 tex, weft yarn count 21 tex, and fabric cover factor 19.56. Transglutaminase (100 IU/g), enzyme was sourced from Aum Enzymes, Gujarat, India. Filozyme Lipase (40000 units/g), and Filozyme protease (60000 units/g) enzymes were procured from Om Biosciences, Ahmedabad, India. EBzyme Laccase enzyme (2000EBU/g) was purchased from Enzyme Bioscience Pvt. Ltd. Kim, Gujarat, India. Sodium carbonate (Merck, assay 99.5%), glacial acetic acid (Merck, assay 99%), sodium hydroxide (Merck, assay 98%) and Ultravon JU (wetting agent), were used as received without any further purification. Wool specific detergent was used for dimensional stability test.

### 2.2 Methods

Wool fabric was scoured using 1.0% sodium carbonate and 2.0% Ultravon JU (on the weight of fabric) for 30 min at 55°C to remove wax and other impurities. The scoured fabric was used as a control and reference. The physical properties, like areal density, thickness and thread density of the scoured fabric were recorded.

#### *2.2.1 Enzyme Treatment*

Scoured wool fabric samples were treated with 0.5%, 1.0% and 2.0% (on the weight of fabric) of transglutaminase, lipase, laccase and protease enzymes respectively. Table 1 shows the acronyms used for different concentrations of various enzymes and *p*H conditions for the specific enzyme. The treatments were carried out in an Infrared Beaker Dyeing Machine (Texcare) at 55°C for 60 min. All enzyme treatments to the fabric were performed using a constant material-to-liquor ratio of 1:30. After the treatment, enzymes were deactivated by immersing the treated samples in hot water at 80–85°C for 8–10 min. Finally, the samples were rinsed with cold water and dried in an oven. A blank treatment was conducted where the scoured fabric was treated with similar conditions but without any enzyme. This would nullify the effect of aqueous treatment and help in understanding the role of enzymes.

#### *2.2.2 Characterization*

The surface morphologies of the blank and enzyme treated fabric samples were examined using Field Emission Scanning Electron Microscopy (FE-SEM) (Nova Nano FE-SEM 450) with suitable magnification. The fabric samples were sputter-coated with gold using a sputter coater (Quorum Q15OTES) before analysis. The moisture content of the samples was determined following ASTM D1576-13 standard test method. The yellowness and whiteness index of fabric samples were analysed with the aid of the computer colour matching

Table 1 — Details of enzyme treatment

| Enzyme | Fabric code | Concentration, % | *p*H |
|---|---|---|---|
| Without enzyme | Blank | – | 7.0 |
| Transglutaminase | TG1 | 0.5 | 7.0 |
| | TG2 | 1.0 | |
| | TG3 | 2.0 | |
| Lipase | LP1 | 0.5 | 8.5 |
| | LP2 | 1.0 | |
| | LP3 | 2.0 | |
| Laccase | LC1 | 0.5 | 4.5 |
| | LC2 | 1.0 | |
| | LC3 | 2.0 | |
| Protease | PR1 | 0.5 | 8.5 |
| | PR2 | 1.0 | |
| | PR3 | 2.0 | |

system (Konica Minolta - model- D 5006774) enabled with JAY PAK software. Instron enabled with Bluehill3 software was used to measure tensile properties (ASTM D638) and frictional properties (ASTM D3108). Bending length was measured using a stiffness tester AKR Precision Instruments (ASTM D1388). The flexural rigidity and bending modulus were determined using the following equations:

$$\text{Flexural rigidity } (G) = w \times c^3 \times 9.81 \times 10^{-6} \quad \ldots (1)$$

where $G$ is the flexural rigidity (mN.mm); $w$, the fabric weight per unit area (g/m$^2$); and $c$, the bending length (mm)

$$\text{Bending modulus } (q) = \frac{12G}{t^3} \quad \ldots (2)$$

where $q$ is the bending modulus (kN/m$^2$); and $t$, the fabric thickness (mm).

All tensile strength, frictional, and bending properties were statistically analysed using the ANOVA.

#### *2.2.3 Shrinkage Measurement*

The dimensional stabilities of scoured, blank and enzyme treated fabric samples were studied using a launderometer. The fabric sample of 15×15 cm was marked 12×12 cm inside the sample with a water-resistant fabric pen. The marked samples have undergone one relaxation cycle and three felting cycles in the launderometer to simulate the standard method ISO 6330 using Wascator[21]. In the relaxation cycle, fabrics were immersed in 1 g/L detergent without any agitation for 60 min at 40°C temperature. During each felting cycle, samples were washed in a launderometer with 0.3 g/L detergent for 60 min at 40°C, rinsed and flat dried in an oven. After three felting cycles, area shrinkage was calculated using the following equation; the shrinkage values were statistically analysed using ANOVA:

$$\text{Area shrinkage } (\%) = \frac{\text{OM}-\text{FM}}{\text{OM}} \times 100 \quad \ldots (3)$$

where OM is the original measurement (cm) of the marked square (Σ warp and weft length); and FM, the final measurement (cm) of the marked square (Σ warp and weft length) after washing for one relaxation cycle followed by three felting cycles.

## 3 Results and Discussion

### 3.1 Effect of Enzymes on Dimensional Stability

The four enzymes, viz transglutaminase, lipase, laccase and protease have been selected for the study. Transglutaminase cause cross-linking by the formation of a carboxylamide groups of peptide bound glutamine in wool keratin[22]. Lipase and protease belong to hydrolases class and cause hydrolytic cleavage of bonds[23]. Laccase has been used in the degradation of waste, colour and lignin[24]. It can participate in the cross-linking of monomers.

All enzymes are *p*H, temperature and time sensitive. Hence, specific *p*H, temperature and concentration range are chosen for each enzyme (Table 1) based on the literature review[6]. The treatment time is kept constant at 60 min for each enzyme. Three concentrations of each enzyme, viz 0.5%, 1.0%, and 2.0%, have been selected. The enzymes can easily penetrate and damage the wool fibre[6]. This probability is higher with high enzyme concentration. Hence, the enzyme concentration is kept limited to a maximum of 2%. The enzyme treated samples are given one relaxation cycle and three felting cycles to determine the shrink resistance or the dimensional stability.

Figure 1 presents the dimensional stability of the various enzyme treated fabrics. The relaxation cycle show comparable and least dimensional change among the fabrics. In the case of protease treated fabrics, negative shrinkage is observed. This phenomenon is also known as hygral expansion and may be due to the possible swelling of enzyme treated wool fibres by water molecules through the modified cuticle. This negative shrinkage indicates that the protease has done the cleavage of surface scales more effectively than other enzymes, which makes the fibre surface relatively hydrophilic.

The blank fabric shows high area shrinkage (12.3%) after three felting cycles (Fig. 1). Overall, the area shrinkage of wool fabric is reduced due to the

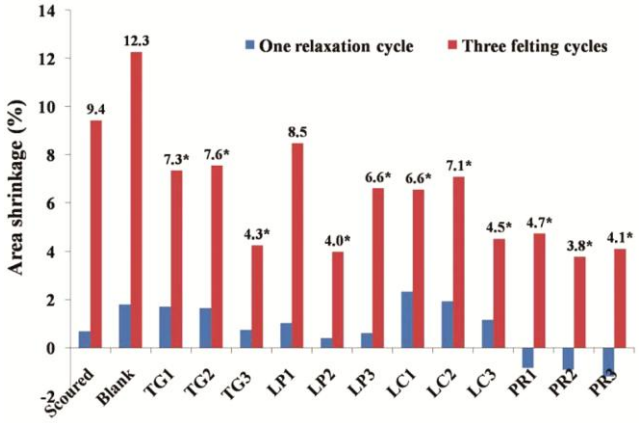


Fig. 1 — Area shrinkage of wool fabrics treated with transglutaminase, lipase, laccase, and protease enzymes in concentration range 0.5–2% (*significant at 5% level of significance)

enzyme treatment. All these protein-specific enzymes, under suitable conditions, may cleave the surface scales of wool, thereby reducing the probability of felting during washing. The surface scale cleavage may be due to the partial disulfide bond breakage by the enzyme attack on the fibre surface[7,25,26].

All the enzymes are found significantly effective ($p < 0.05$) for shrink resistance of wool fabric as compared to the blank fabric (wet treated without enzyme). Transglutaminase and laccase at 2% (TG3 and LC3 respectively) whereas lipase and protease at 1% (LP2 and PR2 respectively) are found better (< 5% area shrinkage) among all the combinations of enzyme concentrations. Therefore, these four enzymes and their respective concentrations are repeated in the bulk process and considered for further characterization.

### 3.2 Effect of Enzymes on Fabric Physical Properties

Table 2 shows the effect of various enzymes on the physical properties of the fabric. All the wet treatments invariably increase the basis weight, thickness and thread density as compared to the scoured fabric (163 g/m² basis weight and 0.6 mm thickness). The increment (maximum 176 g/m² basis weight and 0.7 mm thickness) is found statistically significant ($p<0.05$). The significant difference in physical properties between the blank and the enzyme treated fabrics indicates that the later causes a felting like action under alkaline *p*H conditions. Among enzymes, laccase and protease do show the highest basis weight and thickness increment. This is probably due to favourable felting like conditions (mainly alkaline *p*H and mechanical agitation during the enzyme treatment), which may interlock the scales of wool fibre surface and reduce the inter-fibre spacing, resulting in higher basis weight and thickness.

### 3.3 Characterization

#### *3.3.1 FE-SEM Analysis*

The effect of enzyme treatment can be better seen at the fibre surface. Hence, the fibres from the enzyme treated fabric are extracted and analysed under FE-SEM. Figure 2 shows the FE-SEM images of blank and different enzyme treated fibres. On the surface of

Table 2 — Effect of enzymes treatment on physical, moisture and surface appearance properties of wool fabric

| Fabric | Weight, g/m$^2$ | Thickness, mm | Moisture content, % | Yellowness index (E313 2 deg/C) | Whiteness index (Hunter 10 deg/D65) |
|---|---|---|---|---|---|
| Scoured | 163.0 | 0.6 | 13.4 | 18.8 | 50.8 |
| Blank | 167.2 | 0.7 | 13.0 | 17.6 | 52.9 |
| TG3 | 168.4 | 0.7 | 13.1 | 19.7 | 49.2 |
| LP2 | 174.0* | 0.7 | 13.0 | 19.5 | 48.7 |
| LC3 | 172.3* | 0.7 | 13.7 | 19.8 | 49.4 |
| PR2 | 175.9* | 0.7* | 13.4 | 18.6 | 50.9 |

*Significant at 5% level of significance.

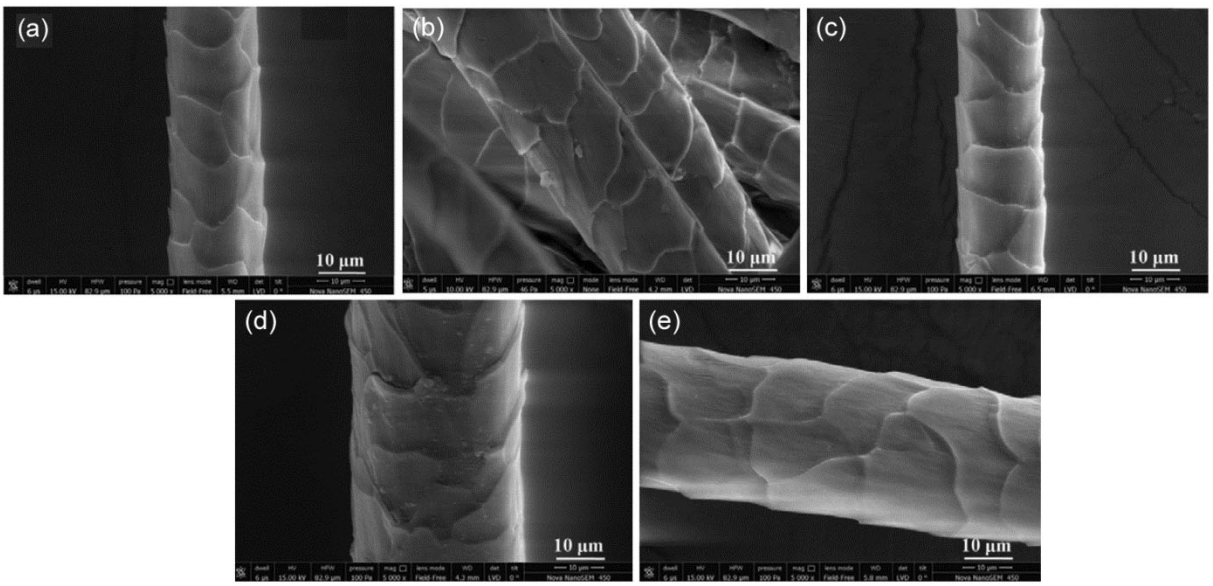


Fig. 2 — FE-SEM images of wool fibres (a) blank, (b) TG3, (c) LP2, (d) LC3, and (e) PR2

blank wool fibre, sharp cuticle scales can be observed clearly [Fig. 2(a)], while the sharpness of the scales is reduced due to enzyme treatment [Fig. 2(b)–(e)]. This result is in line with earlier reports[27–29].

#### *3.3.2 Moisture Content*

Table 2 depicts moisture properties in the blank and enzyme treated fabric. The blank fabric has a moisture content of 13.0%. These values are comparable with the standard moisture content of wool (13.8%). The enzyme treated fabrics show no significant difference ($p>0.05$) in the moisture content. However, Soun *et al.*[29], and Chakraborty and Madan[30] reported that the enzyme treatment causes an increase in moisture content, which is due to the attack of enzymes on the cuticle scales on the wool surface which are hydrophobic. It may be inferred that, in this study, the enzyme concentration, *p*H and time cause minimal damage to the cuticle layer of the wool fibre surface, resulting in comparable moisture content.

#### *3.3.3 Yellowness and Whiteness Index*

Table 2 also shows the yellowness and whiteness index of scoured, blank and enzyme treated wool fabric. Both yellowness and whiteness indices of enzyme treated fabrics are found comparable with that of scoured and blank fabric. This infers that the enzyme treatment does not adversely affect the whiteness. The enzyme treatment does not cause yellowing to the fabric which is a major advantage over the conventional shrink resistance process.

#### *3.3.4 Tensile Properties*

Figure 3 shows the tensile properties of scoured, blank and enzyme treated woollen fabric. It can be seen that the tensile strength [Fig. 3(a)] of the fabric does not change significantly ($p>0.05$) due to the enzyme treatment. All fabrics show comparable tensile strength (11 MPa). This result can be linked with the comparable moisture content of the enzyme treated fabric due to the minimal damage of surface scales. Chakraborty and Madan[30], Mojsov[31], Kotlinska and Lipp-Symonowicz[32] reported that the high tensile strength loss is due to high enzyme concentration and prolonged duration of treatment. The tensile extension [Fig. 3(b)] of PR2 (15.7%) and LP2 (17.1%) reduce significantly in comparison with the blank sample (19.0%). This is due to the alkaline *p*H (8.5), which favours felting and reduces the extension. It means LP2 and PR2 enzymes are modifying the fibre surface as compared to TG3 and LC3. However, this surface modification has limited magnitude, due to which the tensile strength remains comparable with that of the blank.

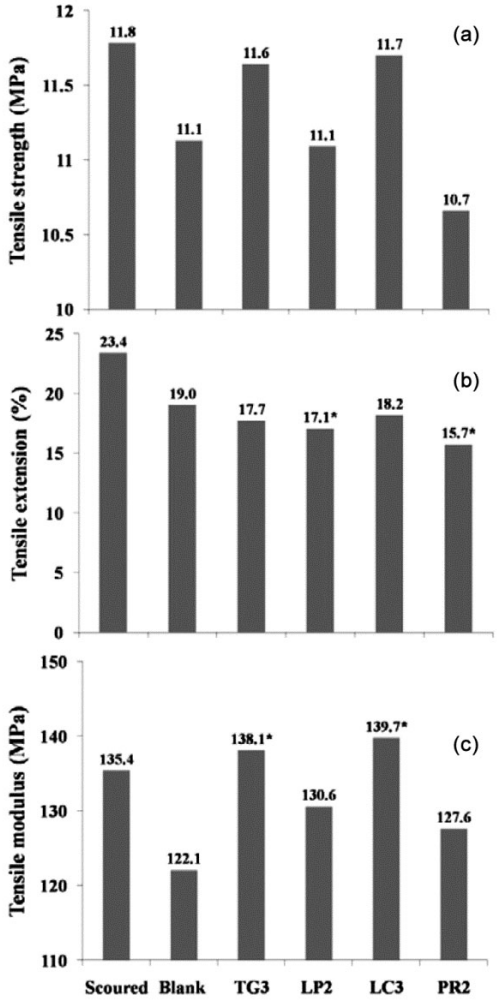


Fig. 3 — Effect of enzyme treatment on woolen fabric (a) tensile strength, (b) tensile extension, and (c) tensile modulus (*significant at 5% level of significance)

Tensile modulus [Fig. 3(c)] is an overall representation of the tensile behaviour. The modulus of TG3 (138.1 MPa) and LC3 (139.7 MPa) are found significantly higher than that of the blank fabric (122.1 MPa). The result is due to the reduced extensibility of the enzyme treated samples at comparable tensile strength. Although LP2 (130.6 MPa) and PR2 (127.6 MPa) record higher modulus, it is not statistically significant, because both tensile strength and extension are reduced.

#### *3.3.5 Frictional Properties*

Table 3 represents the dynamic and static coefficient of friction for enzyme treated fabrics. Both friction coefficients are found to be significantly increased after wet treatment. This result is due to the increase in the

Table 3 — Effect of enzyme treatment on frictional and bending properties of wool fabric

| Fabric | Coefficient of friction | | Bending length mm | Flexural rigidity mN.mm | Bending modulus kN/m$^2$ |
|---|---|---|---|---|---|
| | Dynamic | Static | | | |
| Scoured | 0.7 | 0.8 | 20.8 | 14.4 | 692.4 |
| Blank | 0.8 | 0.8 | 23.5 | 20.9 | 1005.3 |
| TG3 | 0.8* | 0.8 | 21.4 | 15.7* | 752.8* |
| LP2 | 0.8* | 0.8* | 23.0 | 19.5 | 937.4 |
| LC3 | 0.8* | 0.8* | 22.2 | 17.7 | 848.8 |
| PR2 | 0.8* | 0.8* | 22.7 | 18.8 | 903.2 |

*Significant at 5% level of significance.

basis weight and thickness during the aqueous treatment. The blank fabric nullifies the effect of wet treatment. Interestingly, when friction values of the blank fabric are compared with the enzyme treated fabrics, they are found to be reduced. It infers that the enzymes may have cleaved the fibre surface by partial hydrolysis and reduce the sharpness of scales. This change allows the fibres to slide over each other without interlocking of the scales. Among enzymes, LC3, LP2 and PR2 are found to have maximum surface cleavage mainly due to the acidic and alkaline *p*H conditions (for later two) respectively. The softening and smoothening of protruding scales by the enzyme action are in line with the recent report of Wang *et al.*[16].

#### *3.3.6 Handle Associated Properties*

The handle associated properties of the fabric are bending length, flexural rigidity and bending modulus. Table 3 shows the bending properties for scoured, blank and enzyme treated wool fabrics. The bending length seems to be increased due to the enzyme treatment. However, it may be due to the intensive water treatment under agitation, which results in partial felting and eventually increase the stiffness of the fabrics. The aqueous treatment of the blank fabric causes a significant increase (from 20.8 mm to 23.5 mm) in the bending length. While the enzyme actions tend to do the opposite. As compared to the blank fabric, all enzyme treated fabrics show lower bending length. This further confirms the observation of partial removal of scales by the enzyme action which helps in enhancing the smoothness and handle of the enzyme treated fabric. The flexural rigidity and bending modulus results go hand in hand with that of the bending length. This result infers that the enzyme treatment reduces the stiffness of the fabric[4] and improves the handle of the fabric without deteriorating the mechanical properties of the fabric.

#### *3.3.7 Shrink Resistance*

To validate the shrink resistance results obtained earlier, wool fabrics were again treated with selected enzyme concentration and compared with the blank and scoured fabrics (Fig. 4). The results of Figs 1 and 4 are found like-wise, which validates the findings of shrink resistance caused by enzyme treatment. The negative shrinkage during relaxation cycle in case of protease and laccase is due to the easy entry of water molecules which causes hygral expansion, as discussed before in Fig. 1. The scoured fabric exhibits high area shrinkage (11.0%), while the blank (wet treated without enzyme) fabric show maximum shrinkage (13.3%). This result may be due to the agitation of fabric in hot water at 55°C for 60 min. The mechanical action and high temperature cause the wool fabric to shrink. However, when the fabrics are treated with enzymes under the same conditions, the shrinkage of fabric is significantly reduced ($p<0.05$). Among the various enzymes, minimum area shrinkage is observed in PR2 (3.0%) treated fabric sample. It may be due to the partial hydrolysis of cuticle scales by enzymes at the surface of the wool fibre[18]. The dimensional stability results of PR2 can be aligned with the lowest coefficient of friction. Protease hydrolyses the cuticle scales of the wool and reduces the inter-fibre friction[33]. The shrinkage

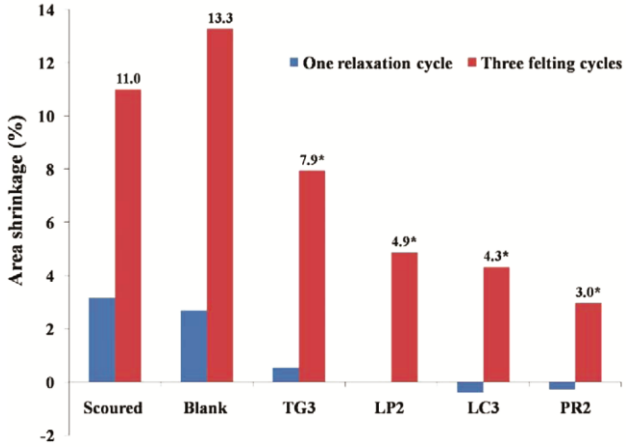


Fig. 4 — Effect of selected enzyme concentration on dimensional stability of wool fabrics (*significant at 5% level of significance)

(3.0%) of the PR2 is found comparable with the multi-step treated fabric (3.1%), as also reported by Wang *et al.*[34], where the fabric is pre-treated with peroxide and cutinase enzyme. Overall, the protease enzyme provides better dimensional stability to wool fabrics as compared to all other enzymes at a similar level of processing conditions.

In the case of other enzymes, lipase shows comparable results with protease. This may be due to the similar *p*H level (8.5) and similar nature of enzymes. Protease and lipase both belong to hydrolases group of enzymes and follow the similar reaction mechanism that is hydrolytic cleavage of bonds. However, since lipase hydrolyses the ester bonds while protease hydrolyses the peptide bonds, the equivalent *p*H level may have a dominating role. While, in case of transglutaminase and laccase, both assist in cross-linking of protein molecules, which may result in better dimensional stability of wool fabric when compared with scoured and blank fabrics.

Protease and lipase at 1% concentration show better shrink resistance than laccase and transglutaminase at 2%. Due to the different nature of all four enzymes, it is difficult to compare the shrink resistance performance straight away. However, it can be linked with a *p*H level of enzymes. Protease and lipase enzyme treatments have alkaline *p*H (8.5), whereas laccase and transglutaminase have acidic (4.5) and neutral (7.0) *p*H respectively. This study suggests that the alkaline *p*H specific enzymes at lower concentration can impart better shrink resistance to wool fabrics than neutral and acidic *p*H specific enzymes.

## 4 Conclusion

Wool fabric is treated with transglutaminase, lipase, laccase and protease enzymes at 0.5, 1.0 and 2.0% concentrations. The felting shrinkage of the 12 fabrics is determined using standard protocols and compared with the scoured fabric. The concentration of each enzyme with best shrink resistance is repeated for validation and to study mechanical, frictional, and bending properties. Transglutaminase (2%), lipase (1%), laccase (2%) and protease (1%) enzymes are found to have less shrinkage, i.e. 7.9%, 4.9%, 4.3% and 3.0% respectively as compared to 13.3% of the untreated fabric. The low concentration of protease enzyme partially hydrolyses the peptide bonds of wool at the cuticle scale and reduces the inter-fibre friction which avoids the interlocking of scales. This enzyme action provides dimensional stability to wool fabric. The alkaline *p*H during the protease and lipase enzyme treatment further favours in achieving dimensional stability. Transglutaminase and laccase enzymes assist in cross-linking of protein molecules, which may result in better dimensional stability of the wool fabric. Tensile properties of enzyme treated fabrics are found to be comparable with the blank fabric. Handle and frictional properties are significantly changed in favour of enzyme treatment. The enzyme treatment neither affects the whiteness index nor causes the yellowing of the fabric. Among the selected enzymes, protease at 1.0% concentration is found to be the best to achieve maximum shrink resistance without significant change in mechanical and handle associated properties. In addition to wool properties retention, the enzyme treatment is sustainable and easy to scale up. Therefore, it has promising potential for industrial adaptation.

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