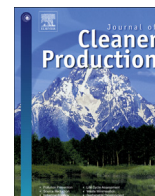




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Controlled eco-friendly shrink-resist finishing of wool using bromelain

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ABSTRACT

Shrink-resist finishing is imperative to introduce dimensional stability and characteristic handle for wider acceptance of woollen garments. Conventionally used oxidative anti-felting treatments using sodium hypochlorite, gaseous chlorine, dichloroisocyanuric acid etc. release chlorine leading to high concentration of absorbable organic halides in waste water; application of resin results in stiffer handle because of spot welding effect. In contrast, enzymatic processing using protease is a sustainable alternative to substitute eco-hazardous conventional processes though diffusion of protease deep inside the wool in alkaline pH is the major concern for substantial weight as well as tensile loss thus limiting its application on wool. Treatment with bromelain even at very mild alkaline pH around 7.5 in absence of salt results in considerable damage in wool structure with consequence of higher weight and tensile loss due to partial/complete removal of cuticles on the surface. In this work, bromelain, a type of proteolytic enzyme, was used along with salt in acidic pH for controlled superficial hydrolysis of wool. A two-step acidic bromelain treatment was opted first using Box–Behnken design keeping in view minimum area shrinkage succeeded by optimization of the treatment parameters to achieve least area-shrinkage, weight loss, strength loss along with improved subjective softness. Controlled adsorption of bromelain at the outer surface of wool in presence of salt at pH ~6 was observed yielding desirable anti-felting behaviour with minimum weight and strength loss. In contrast to conventional chlorine based technologies, acidic bromelain may be used in presence of sodium chloride to yield machine washable wool possessing minimum loss in weight and tensile strength both at industrial level.

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1. Introduction

Wool, composed mainly of keratin protein is a multifunctional natural fibre used for manufacturing apparels. Felting is the limiting behaviour of woollen textiles that affect its dimensional stability and influence suitability to the consumer by deciding the fitting, comfort and appearance after repeated launderings. Shrinkage resulting from felting of wool is primarily because of directional frictional effect through overlapping of cells (scales) in cuticle (surface layer) during mechanical interaction, for example during washing etc (Fig. 1).

Conventional processes in practice for shrink-resist finishing involves either use of oxidizing agents like hypochlorites to attack the scales of the cuticles (subtractive treatment) or covering the scales with a film made up of suitable resins (additive treatment). Combined commercial process like chlorine-hercosett consists of

chlorine pretreatment followed by application of resin polyamide-epichlorohydrin (Udakhe et al., 2011). Chlorination leads to environmental hazard such as release of absorbable organic halides in high concentration to the effluents, excess water consumption etc. (Dominguez et al., 1980). Additive treatment has its own drawback of deterioration in fabric handle and felting along seams and folds leading to breakdown of polymer at seam points (Chen et al., 2000).

Alternative processes to substitute eco-hazardous chlorination to save eco-system is the need of hour. Owing to such concerns, global drive to explore sustainable technologies, environmental friendly processes using biocatalysts i.e. enzymes have emerged as attractive alternatives to conventional textile chemical processing (Jegannathan and Nielson, 2013). Improvement in shrink-resistance, dyeing, handle and comfort properties may be imparted with proteolytic enzymes when the latter hydrolyze the proteins present in the cuticles of wool reducing directional frictional effect in otherwise scaly wool (Bishop et al., 1998; Das and Ramaswamy, 2006; Moncrieff, 1953; Riva et al., 2002). However application of proteases may produce machine washable wool (Woolmark specifications); excessive weight as well as strength loss are the limiting

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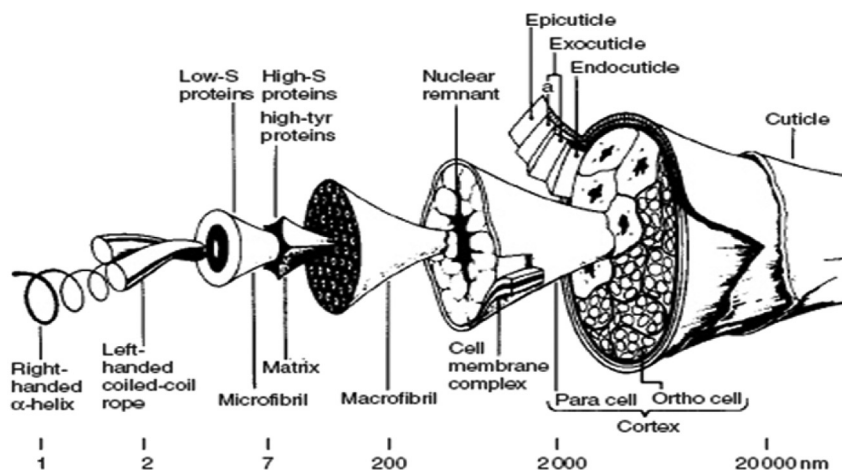


Fig. 1. Cross-sectional view of wool fibre (Hearle, 2002).

concern (Jovancic et al., 1998). Small protease molecules of nearly 13–20 kDa molecular weights are generally able to penetrate into the wool. As the protease diffuses into the membranes between the cells, it rapidly disrupts cell membrane complex and damages wool if reaction is prolonged further (Okada et al., 2008). Native proteases generally attack at highly swollen continuous phase of cell membrane complex (CMC) underneath the cuticle, allowing enzyme penetration between cuticular and cortical cells (Heine and Hocker, 1995). Lenting et al. (2006) used alkaline bleaching pretreatment in presence of salt to control the enzymatic process. Raja and Thilagavathi (2010) however suggested use of acidic pretreatment conditions before protease action. Treatment of wool with modified enzyme (having bigger size than native protein) was reported to control the diffusion of protease inside wool structure (Cavaco-Paulo and Silva, 2003; Schroeder et al., 2004; Silva et al., 2004). Post-treatment of protease treated wool with protein cross-linking enzyme was suggested to overcome the weight and tensile strength loss occurred during protease action (Cardamone, 2007; Cortez et al., 2004; Du et al., 2007). Recently, use of immobilized protease (having advantage of controlled process and reusability) was also mentioned in this context (Shen et al., 2007; Smith et al., 2008).

In general, alkaline proteases are used for shrink-resist treatment though wool remains stable if processed in acidic conditions rather than in alkaline. The acidic and basic groups of wool remain in equal concentrations during pH range of 4.5–5 (Capablanca and Watt, 1986; Sookne and Harris, 1939). At this juncture, one key approach to prevent damage of wool may be identification of proteases, which can work preferably in wide range of acidic conditions while proteolytic activity significantly remain focused at the surface of wool. Another approach may be use of some retarding agent like salt during protease treatment itself for restricted diffusion of enzyme at the interior of wool. Lesser swelling of wool in presence of salt also imposes restriction to diffusion of enzymes at the wool interior. Studies show synergetic effect of proteolytic enzyme action in acidic pH and restricted diffusion at the interior of wool while influence of salt in protease treatment was not examined (Bradbury et al., 1963).

Stem bromelain (EC 3.4.22.32) isolated from the stem of the pineapple plant is a thiol protease with a molecular mass of 23.8 kDa (Vanhoof and Cooreman, 1997). Its activity becomes best in the pH range of 4–11 and at 25–70 °C as per the substrate (Suh et al., 1992). Bromelain possesses low substrate specificity and may be used for uniform treatment, but delicate control of reaction is the major limitation (Silvestre et al., 2012).

The aim of this work is to study the potential and controlled treatment of stem bromelain enzyme on scoured wool in presence of salt in acidic pH to achieve desired shrink-resist property without significant loss in its weight and tensile strength both.

2. Experimental

Woven scoured wool fabric (warp: 2/52 Nm, weft: 1/30 Nm, ends per inch: 84, picks per inch: 54, grams per square metre: 224) was used in the study. Wool fabric for experimentation as well as industrial trial was kindly provided by OCM India Ltd., Amritsar (India). Stem Bromelain, i.e. EC 3.4.22.32 (Excellent Biotechnologies, India), non-ionic surfactant Sandoclean PCJ (Clariant), casein, BSA, trichloroacetic acid, folin ciocalteu (Hi media) and other analytical grade chemicals (SDFCL, India) were used. Bromelain activity was assessed with UV–Vis spectrophotometer (Perkin Elmer) while taking casein as substrate (Xue et al., 2010). Area shrinkage and tensile strength were evaluated as per Woolmark TM31 and ASTM D5034-1995 respectively. Subjective softness ratings of treated fabrics were given by five experts having knowledge on the evaluation of fabric handle. Rating ‘5’ was considered as very good and ‘1’ as very poor. The coefficient of variation (CV %) and ANOVA probability value for the subjective ratings given by judges was determined (Raja and Thilagavathi, 2010). Chemical change analysis was done with the help of FTIR spectrometer (Perkin Elmer) using KBr pellet method. Microscopic evaluation at fibre surface was done with SEM (Jeol-JSM-6610LV).

2.1. Influence of different process parameters on activity of bromelain

Bromelain activity at varying pH, temperature and salt concentration were determined. To measure bromelain activity, 0.25 ml of casein (1%) was treated with 0.3 ml bromelain solution in 0.1 M sodium phosphate buffer, pH 6.8 at 37 °C for 15 min and the reaction was arrested by the addition of 0.25 ml of 5% trichloroacetic acid (TCA) (Murachi et al., 1964). The resulting soluble peptides were then quantified by using Bovine serum albumin (BSA) as the standard (Lowry et al., 1951). For comparison, an appropriate blank solution was also prepared. One unit of bromelain activity was considered the amount that resulted in change of 0.001 absorbance units at 670 nm per min. Bromelain activity was assessed with respect to different pH (4–11), temperature (30–100 °C) and NaCl (0–1.5%, w/v).

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