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Shrinkproofing of wool using benign methanolic potassium hydroxide and sericin biopolymer

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In this research work, wool fabrics are pretreated with methanolic potassium hydroxide and subsequently treated with the biopolymer sericin to impart shrink-proofing. The effects of key process parameters, namely the concentration of potassium hydroxide and the concentration of sericin, on the area shrinkage of wool are examined. The results indicate that wool pretreated with 0.02 M methanolic potassium hydroxide followed by 15 g/L sericin shows the least area shrinkage of 6%, compared with untreated wool. The fabrics are characterised using scanning electron microscopy, tensile strength testing, bending length measurement and yellowness index evaluation. The findings demonstrate that controlled hydrolysis of wool fibres, followed by sericin coating, provides a satisfactory anti-felting effect. The colour of the wool changes to off-white without adversely affecting handle, tensile strength or elongation. The proposed shrink-proofing method proves effective, as it is chlorine-free and employs benign potassium hydroxide along with sericin reclaimed from silk waste, offering a safer alternative to conventional chemical-based polymers.

Keywords: Antifelting, Biopolymer coating, Machine-washable wool, Sericin, Silk waste

1 Introduction

Wool is a superior hair fibre obtained from sheep, which is known for its warmth, stretchability, resilience and inherent flame retardancy. Despite these advantages, wool suffers from a major drawback: its tendency to felt when exposed to washing or mechanical action in the presence of moisture, leading to fibre entanglement and irreversible shrinkage[1]. Felting shrinkage arises primarily from two characteristics of wool: its sharp, overlapping scale structure and its hydrophobic surface. The directional differences in friction caused by the scales generate a Differential Frictional Effect (DFE), while hydrophobicity results from the presence of a covalently bound lipid layer, chiefly 18-methyleicosanoic acid (18-MEA), linked through thioester bonds[2-3]. Effective shrink-proofing, therefore, requires the partial removal or modification of this lipid layer, followed by deposition of a suitable polymer to smooth or mask the fibre scales.

The most widely adopted industrial process, the Chlorine–Hercosett method, achieves excellent felt resistance through a two-step treatment: oxidation with dichloroisocyanuric acid (DCCA) followed by application of a polymer such as Synthapret BAP[4]. However, this process has notable disadvantages, including limited durability, fibre yellowing, altered dyeability and the release of toxic adsorbable organic halides (AOX), posing environmental and health concerns[5]. These issues have stimulated the search for more sustainable alternatives. Numerous physical, chemical and biological approaches—including plasma, ultraviolet irradiation, enzymatic treatments and ionic liquids—have therefore been explored[6-7].

Methanolic potassium hydroxide (MPH) treatment has emerged as a promising alternative, as it hydrolyses ester and thioester bonds on the wool surface, reduces aliphatic lipid content and improves wettability[8]. However, MPH alone does not achieve the commercial machine-washability requirement of <8% felting shrinkage, necessitating additional polymer deposition. Replacing synthetic polymers such as Synthapret BAP with biopolymers offers an opportunity to reduce environmental impact.

Among biopolymers, sericin has garnered significant interest due to its biodegradability, biocompatibility, and multifunctional properties, including moisture absorption, antioxidant, antimicrobial, and coagulating activities[7]. Sericin constitutes the adhesive coating surrounding silk fibroin filaments in *Bombyx mori* cocoons and is

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typically removed during degumming[9]. An estimated 50,000 tonnes of sericin could be recovered annually from global silk-processing wastewaters, where its high oxygen demand poses an environmental burden[10]. Recovery and use of the same from wastewater can reduce this effluent load and lead to significant economic and social benefits.

Few studies have reported the surface modification of wool using potassium hydroxide[11-13], and none, to the best of our knowledge, have examined a combined MPH pretreatment followed by sericin application. In this study, wool fabrics are pretreated with benign methanolic potassium hydroxide and subsequently treated with sericin extracted from silk waste to achieve eco-friendly shrink-proofing. The effects of MPH and sericin concentrations on felting shrinkage are evaluated. Surface morphology is studied using scanning electron microscopy (SEM), while mechanical properties, handle characteristics, and yellowness index are assessed and compared with those of untreated wool.

2 Materials and Methods

2.1 Materials

Loosely woven 100% pure merino wool fabric, typically used for high-fashion stoles and scarves, was procured from Shingora Textiles, Ludhiana. The fabric is a 2/2 matt weave with a warp/weft count of 23.5/18.02 tex, thickness of 0.33 mm, ends per inch/picks per inch (EPI × PPI) of 46 × 44, and an areal density of 86.3 g/m².

Potassium hydroxide (KOH; assay ≥ 85.0%) and methanol (assay ≥ 99.8%) were obtained from Fisher Scientific Chemicals, Mumbai. Deionised water was used for preparing all solutions. Lissapol N, a non-ionic surfactant, was used during shrinkage testing.

2.2 Methods

2.2.1 Extraction of Sericin from Silk Waste

Sericin liquor was prepared by extracting silk waste in deionised water at 100 °C for 60 min using an infrared dyeing machine (DLS 7000, Daelim Scarlet, Korea) at a material-to-liquor ratio (MLR) of 1:30, following the procedure reported earlier[14]. Silk waste (the part of the silk fibre which is left over after reeling of silk from the cocoon) procured from the Central Silk Technological and Research Institute (CSTRI), Bangalore, was cleaned manually and used directly after extraction without further purification.

2.2.2 Pretreatment of Wool with Methanolic KOH (MPH)

Wool fabric samples were treated with a solution containing a mixture of methanol and KOH (MPH) at varying KOH concentrations (0, 0.01, 0.015, 0.02, 0.03, 0.05, 0.08, 0.1 and 0.2 M) at 40 ˚C for 20 min, in an infrared dyeing machine (DLS – 7000, Daelim Starlet, Korea) equipped with six medium-wave IR heating tubes. The MLR was maintained at 1:25. Following treatment, the fabrics were thoroughly rinsed with deionised water and air-dried.

2.2.3 Post-Treatment of MPH-Treated Wool with Sericin

The MPH-treated wool samples were padded with sericin liquor at room temperature of 35°C using a 3-dip/3-nip sequence on a laboratory pneumatic padding mangle set to achieve 60% expression. The sericin bath pH was adjusted to 4. To examine the influence of polymer add-on, sericin concentration varied across six levels: 0, 2.5, 5, 10, 15 and 20 g/L. After padding, the treated fabrics were dried, washed with Lissapol N solution (10 min at 40 °C), rinsed, and dried.

2.2.4 Shrinkage Testing

Shrinkage behaviour was evaluated using a previously established method[15]. Each sample was subjected to one relaxation wash followed by three felting washes in a launderometer (R.B. Electronics, Mumbai). The launderometer operated at 40 ± 2 rpm using stainless steel beakers (SS-316) with a 500 mL capacity, a diameter of 7.5 mm, and a length of 12.5 cm, with a revolution speed of 40 ± 2 rpm.

Relaxation wash: 1 g/L Lissapol N, MLR 1:30, 40 °C, 1 h

Felting washes: 0.3 g/L Lissapol N, MLR 1:30, 40 °C, three cycles of 1 h each

2.2.5 Measurement of Shrinkage

Fabric specimens (12 × 12 cm) were marked in warp and weft directions. Dimensions were recorded before washing and after one relaxation and three felting washes. Total area shrinkage was taken as the final shrinkage obtained after one relaxation and three felting washes. The mean of three samples was taken.

$$\text{Area shrinkage (\%)} = \frac{\text{FM - OM}}{\text{OM}} \times 100 \qquad \ldots (1)$$

where *OM* is the original measurement of marked warp and weft length dimensions (cm); *FM*, final measurement after washing.

2.2.6 Scanning Electron Microscopy (SEM)

Surface morphology of untreated and treated wool fibres was studied using a ZEISS EVO 50 scanning electron microscope. Samples were sputter-coated with gold (~100 Å) and imaged at 20 kV using an SE detector at a resolution of ~3 nm.

2.2.7 Yellowness Index

Yellowness index (YI) was measured according to ASTM 313 using Colour Eye 7000 A, a computer colour matching spectrophotometer (Gretag Macbeth, USA). Four readings were taken per sample at different positions and averaged.

2.2.8 Tensile Strength and Elongation

Tensile strength and elongation were measured using an Instron tensile tester according to the ravelled strip method (ASTM D5035-90). Ten replicates in warp and weft directions were tested and averaged.

2.2.9 Bending Length

Bending length was measured using a cantilever-type stiffness tester, in accordance with the ASTM D 1388–14 standard.

3 Results and Discussion

3.1 Effect of Concentration of MPH on Total Shrinkage

Figure 1 shows the effect of MPH on the area shrinkage of wool. It is observed that the untreated sample exhibits 43.33% shrinkage after one relaxation and three felting cycles. This value reduces to 37.3% after treatment with 0.01M MPH. A further decrease is observed at 0.015 M (35.6%) and 0.02 M (33%). Increasing the concentration to 0.03–0.06 M results in comparable shrinkage values with no significant improvement. At 0.1 M, shrinkage decreases marginally to 30.67%. However, at 0.2 M an order-of-magnitude increase the shrinkage reduces substantially to 23.66%.

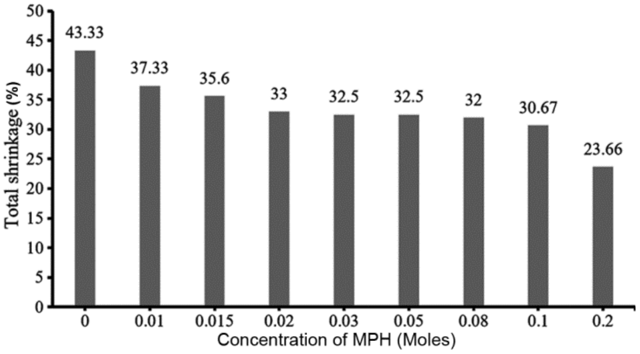

Fig. 1 — Effect of concentration of MPH on total shrinkage

These effects arise because the wool surface is covered by a thin covalently bound lipid layer, connecting cysteine residues via thioester or ester bonds, which renders the wool surface highly hydrophobic[16]. MPH hydrolyses these bonds and reduces the aliphatic lipid content[17], thereby enhancing fibre wettability and reducing felting shrinkage. However, the results demonstrate that MPH treatment alone cannot achieve the machine-washability requirement of <8% shrinkage, indicating the need for a second treatment with a polymer to mask fibre scales and enhance antifelting effect.

3.2 Effect of Concentration of MPH on YI

The colour of wool is another important characteristic when assessing its suitability for a particular end use. Figure 2 indicates that the YI increases from 7.89 (untreated wool) to 13.5 at 0.1M MPH. Yellowness progressively increases with MPH concentration. According to the International Wool Textile Organisation (IWTO)[18], yellowness values between 9.5-10.5 are considered "slightly creamy", 14.5-16 "quite yellow", and above 16 "heavily stained yellow". So, MPH-treated wool acquires a creamy yellow colour. Such yellowing aligns with trends reported for chlorine- and alkali-based treatments[19]. Based on shrinkage (33%, Fig. 1) and a YI of 11.8, 0.02M MPH is identified as the optimum concentration for subsequent sericin application.

3.3 Effect of Concentration of MPH on Tensile Strength and Elongation

While MPH effectively facilitates lipid removal and degradation of cuticle scales, the lipid removal is nonspecific in nature, resulting in the extraction of cortex cells of the wool fibres[8]. So, it is expected that it may have affected the strength of wool fibres. Figure 3 shows that tensile strength remains largely

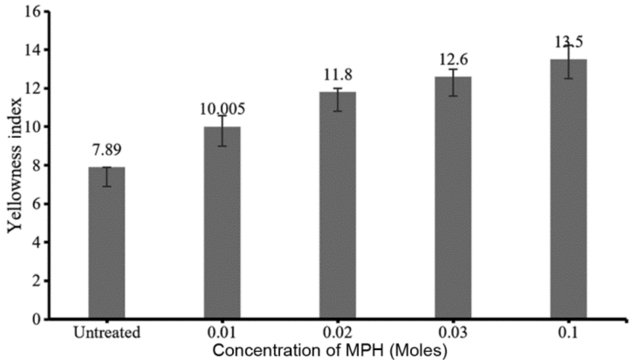

Fig. 2 — Effect of concentration of MPH on yellowness index

unchanged for 0.01–0.1 M MPH, with a slight increase in some cases. However, beyond 0.2 M, severe fibre degradation occurs, and fabrics disintegrate (not shown). It has been previously reported that mild concentrations of MPH lead to higher degradation of the lipid layer on the wool surface, with less hydrolysis of the cortex. However, at higher concentrations, apart from cleaving ester bonds, disulfide and isopeptide cross-links in the wool scales also lead to the severe degradation of cortex cells, resulting in the rupture of fibres[4]. Elongation values exhibit an increasing trend with higher MPH concentrations. The cleavage of surface scales reduces fibre interlocking, thereby enhancing the elongation.

3.4 Effect of Concentration of Sericin on Area Shrinkage

Sericin, a high-molecular-weight protein with film-forming properties, must be applied at an optimal concentration to uniformly coat wool fibre scales without rendering the fabric stiff. If it is too less, there will be non-uniform covering of scales. If it is too high, the fabric will become heavy and stiff.

Figure 4 shows that while MPH pre-treatment reduces shrinkage to 33%, post-treatment with sericin significantly enhances anti-felting performance. Concentrations above 2.5 g/L were studied to achieve the target value of 8% shrinkage. It is observed that Shrinkage reduces progressively with increasing sericin concentration, reaching 6% at 15 g/L, which achieves the machine-washability standard. Beyond 15 g/L, shrinkage plateaus. These findings are in agreement with earlier studies that used biopolymers such as chitosan, wheat starch, Arabic gum, and enzyme biopolymer combinations, which also achieve shrinkage of less than 8%, meeting the machine-washable standard[19-20]. Thus, 15 g/L sericin is considered the optimum concentration.

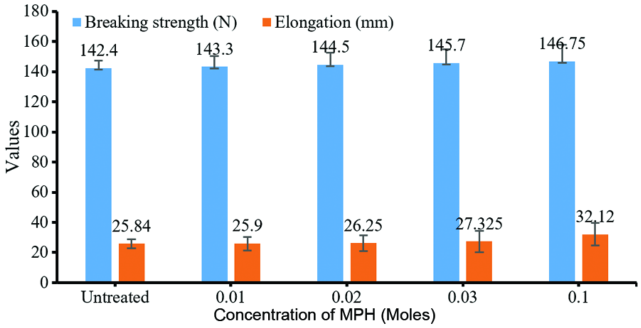

Fig. 3 — Effect of MPH concentration on tensile strength and elongation

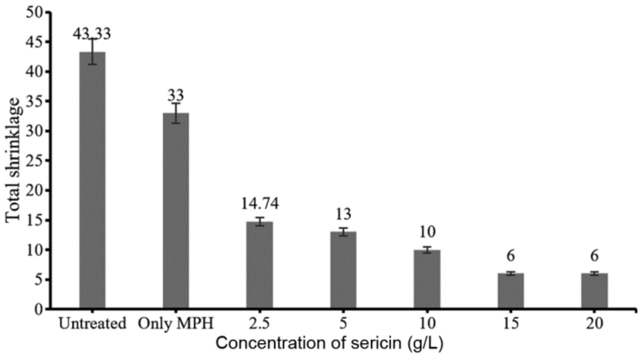

Fig. 4 — Effect of sericin concentration on total shrinkage

3.5 Proposed Reaction Mechanism

Wool fibre surfaces contain thioester linkages anchoring the hydrophobic 18-methyleicosanoic acid (18-MEA) layer. MPH treatment cleaves these linkages via nucleophilic attack, releasing 18-MEA and exposing a more hydrophilic, anionic surface[8] [Fig. 5(a)].

Sericin contains polypeptides containing 17-18 amino acids, among which hydrophilic amino acids like serine and aspartic acid are common. The pH of sericin is pH 5 to 5.5. It has been demonstrated that sericin exhibits a compact structure at pH 4 as opposed to a comparatively free and branching form at pH 3. At pH 4, the amino groups and carboxylate groups get protonated, making the net charge of the sericin positive [Fig. 5(b). Additionally, sericin's particle size shows less polydispersity at pH 4 than at pH 3. These characteristics work together to make it easier to apply to wool, particularly at pH 4[21]. Therefore, the wool fibre surface after treatment with methanolic KOH results in an anionically charged

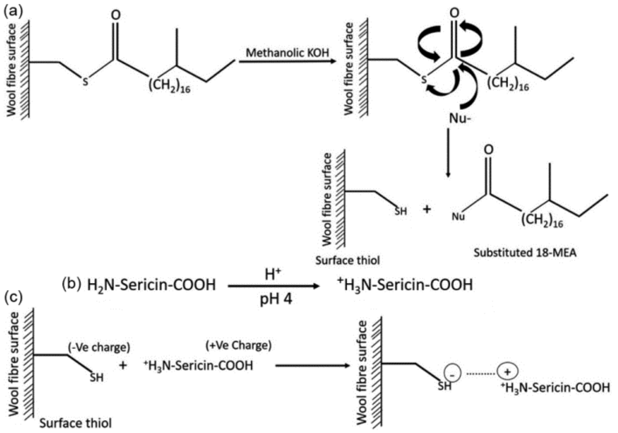

Fig. 5 — Proposed reaction mechanism (a) Nucleophilic cleavage mechanism of thioester bonds linked to 18-MEA[8], (b) protonation of amino and hydroxyl groups of sericin, and (c) ionic binding between wool fibre surface and sericin[21]

surface, which is susceptible to bonding with positively charged sericin through ionic interaction [Fig. 5(c)].

3.6 Effect of Concentration of Sericin on Bending Length

Any textile finishing process can affect the handle properties of the fabric. Merino wool is popular due to its softness next to the skin; however, its stiffness can limit its use in apparel applications. Bending length was performed on two different concentrations of sericin. Figure 6 shows that MPH treatment alone does not affect bending length. However, sericin application increases the bending length slightly, with stiffness becoming noticeable at a concentration of 20 g/L sericin. For the optimum condition of 0.02 M MPH + 15 g/L sericin, the increase in bending length is minimal and does not compromise handle quality. The results correspond with reports on chitosan-treated wool, where an increase in polymer concentration increases bending rigidity, due to the inherent stiffness of the chitosan macromolecule[15].

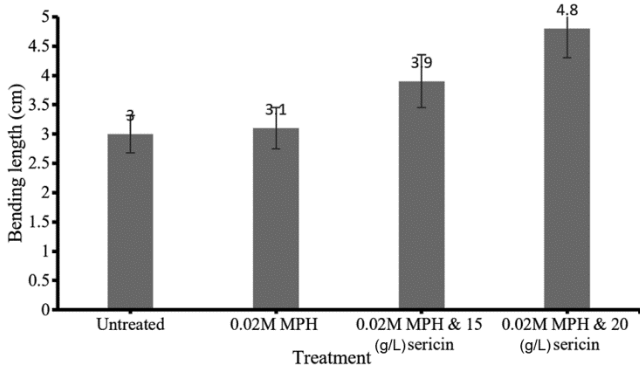

Fig. 6 — Effect of concentration of sericin on bending length

3.7 SEM Analysis

Figure 7 illustrates the morphological changes observed in SEM analysis. Untreated wool shows the characteristic sharp scaly structure, which is responsible for the differential frictional effect and felting shrinkage. However, after treatment with MPH, partial removal of scales is observed, resulting in a cleaner surface. Subsequent sericin treatment results in visible film deposition along fibre edges, indicating effective surface coating. These structural modifications support enhanced anti-felting performance.

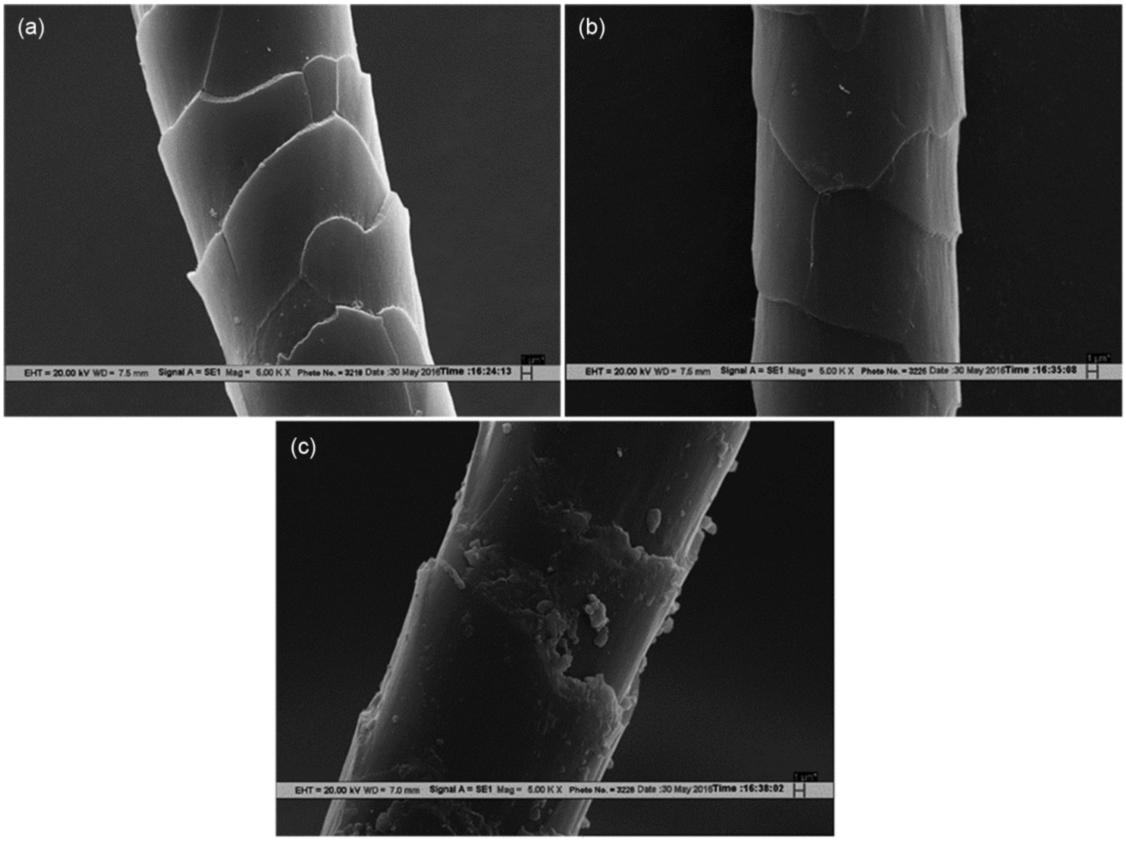

Fig. 7 — SEM images of (a) untreated wool, (b) 0.02M MPH treated wool, and (c) 0.02M MPH-treated wool coated with 15 g/L sericin

4 Conclusion

The present study demonstrates that controlled hydrolysis of wool fibres using methanolic potassium hydroxide, followed by coating with sericin, effectively imparts shrinkproofing to wool. Treatment with 0.02M MPH significantly reduces felting by modifying and partially removing surface scales, while maintaining acceptable colour, tensile strength and handle. The subsequent application of sericin further enhances the antifelting performance, achieving an area shrinkage as low as 6%, which meets the standard for machine-washable wool. The results confirm that sericin at an optimum concentration of 15 g/L forms a uniform, film-forming layer over the fibre surface, promoting ionic interaction with the anionic wool substrate created by MPH pretreatment. This combined treatment improves shrink resistance without adversely affecting the fabric's mechanical properties.

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