ABSTRACT

Wool is commonly treated with alkaline protease enzyme to improve handle and shrink resistance. The limitations associated with the usage of this enzyme on wool are excessive weight and strength losses and pH sensitive enzyme activity etc. Wool is generally stable if processed in acidic conditions rather than alkaline conditions due to its isoelectric point in the pH range of 4.5-5.5. Based on the above facts, this study explores the possibility of using an acid protease enzyme to improve handle and shrink resistance of wool. The acid protease enzyme is applied on the acid and the alkaline peroxide bleached wool fabrics using optimized process parameters and the results are compared with alkaline protease enzyme treated wool fabric. The acid protease treated fabrics show 30% higher softness than alkaline protease treated fabric with 72%, 62% and 16% lower weight loss, strength loss and alkali solubility respectively. However, the whiteness index and shrink resistance values of acid protease treated wool is 44% and 113% lower compared to the alkaline protease treated wool. In order to improve the above properties, alkaline peroxide bleaching process prior to acid protease treatment has been proposed. The action of the enzymes on wool and handle of the treated fabrics have been characterized using SEM, FT-IR spectra and KES-F method.

The outer cuticle layer of wool is damaged during different chemical processing treatments like bleaching and enzyme treatments,
opening a way for microorganisms to attack the cortex, the main fibre portion of wool. Hence, in this research, an attempt has been made to study the microbial resistance nature of wool fabrics pretreated with acid hydrogen peroxide, alkaline hydrogen peroxide, acid protease and alkaline protease using AATCC 100 method. The results show that the acid peroxide bleached fabric (ACB) shows 74% and 64% reduction in the bacterial count for *Staphylococcus aureus* and *Escherichia coli* compared to negligible or nil reduction percentage in the case of scoured, alkaline peroxide bleached (ALB), acid protease treated (ACB-ACP), alkaline protease treated (ALB-ALP) and alkaline peroxide-citric acid treated fabrics. The incorporation of citric acid, which is a known antibiotic agent against the several strains of bacteria on the wool fibre during bleaching, is the reason for reduction in microorganism percentage in the acid peroxide bleached fabric. Solid state $^{13}$C nuclei NMR spectra is used to study the bond formation between wool and citric acid in the acid peroxide bleached and other pretreated fabrics. Based on the above facts, it is inferred that bleached and enzyme treated wool materials need antimicrobial finishing treatment due to the damage to wool cuticle during such treatments. Hence, in this work, attempt has been made to develop antimicrobial finishing process for wool based on nano silver and natural dyes.

Polyvinyl pyrrolidone (PVP) coated silver nanopowder containing 100 ppm of silver has been synthesized by sono-chemical method comprising sonication and reduction with trisodium citrate followed by spray drying. The synthesized silver nanopowder has been characterized by UV-Visible spectra, Atomic absorption spectra, TEM and EDAX. The results indicate the
presence of silver particle with 50-60 nm size in the synthesized powder. The silver nanopowder has been applied on wool to impart antimicrobial efficacy by exhaustion method. The treated wool fabric shows a clear zone of inhibition in the agar diffusion test and 100% and 98% reduction of microorganism in AATCC 100 test against *Staphylococcus aureus* and *Escherichia coli*.

The PVP coated silver nanopowder has been applied on alkaline peroxide, acid peroxide, alkaline protease and acid protease pretreated materials to study the effect of such pretreatments on the antimicrobial efficacy and uptake of silver. The results show that the acid peroxide bleached and the acid protease pretreated wool fabrics absorb 27% and 8% higher silver and exhibit increased antimicrobial efficacy compared to their alkaline counterparts.

The aqueous leaf extracts of the five different deciduous plants, namely, silver oak, flame of forest, tanner’s senna, wattle and serviceberry have been used on their own and in combination with aluminium sulphate, stannous chloride and ferrous sulphate to dye wool by a simultaneous mordanting technique. The washing and light fastness of the developed shades are moderate to good. Based on the CIE 2000 spectral colour coordinate values ($K/S$, $\Delta L$, $\Delta a$, $\Delta b$ and $\Delta E$), the developed shades have been classified into four groups viz. yellow/brown, yellow, orange and dark grey. The use of aluminium sulphate gives medium shades ($K/S = 8.24$) while the stannous chloride and ferrous sulphate mordants provide deep shades ($K/S = 30.5$). Statistical analyses of colour coordinates of the dyed materials
have shown that only the type of mordant and not the dye source significantly influenced the development of colour on wool. Hence, it is theoretically possible to use five selected leaves as a single mixture to produce four different colours on wool.

The above-developed natural dyeing process using plant leaf extracts containing tannin has been used to give both dyeing and antimicrobial finishing treatments to wool pretreated with acid and alkaline peroxide and protease enzymes. The acid protease treated and the alkaline protease treated samples show 10% and 17% higher dye uptake respectively over their corresponding bleached samples. The antimicrobial efficacy test results show that all the wool samples dyed without the use of mordant irrespective of the dye sources show a clear zone of inhibition against Staphylococcus aureus. Only, the dyed wool samples pretreated with the acid peroxide and the acid protease show a clear zone of inhibition against Escherichia coli irrespective of the dye sources. The antimicrobial efficacy of the natural dyed wool samples is due to the enzyme and metal ion binding capacity of tannins, which are present in the selected dye sources. The samples dyed with the use of mordants show low and nil antimicrobial efficacies against Staphylococcus aureus and Escherichia coli compared to the dyed samples without mordant due to the loss of enzyme and metal ion binding capacity of tannins present in the dyed samples during mordanting process.