STUDIES ON ANTIBACTERIAL FINISHING OF COTTON TEXTILES USING NEEM EXTRACT

by

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Submitted

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dedicated to my parents. . . . . .

CERTIFICATE

This is to certify that the thesis entitled "Studies on Antibacterial Finishing of Cotton Textiles using Neem Extract" being submitted by **Mrs. Roli Purwar,** to the Indian Institute of Technology, Delhi, for the award of the degree of **Doctor of Philosophy** in the department of Textile Technology, is a record of bonafide research work carried out by her. Mrs. Purwar has worked under my guidance and supervision and fulfilled all the requirements for the submission of the thesis.

The results contained in this thesis have not been submitted, in part or full, to any other university or Institute for the award of any degree or diploma.

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ABSTRACT

The use of natural antimicrobial agents for bioactive textiles is an emerging area of research. Neem (*Azadirachta indica*), an evergreen tree of India, is one of the promising sources of biologically active compounds chemically known as limonoids which are bitter in taste. The most important quality of neem extract and its active compounds is that they are skin friendly and less toxic to warm-blooded animals like human. The aim of this work is to explore the possibility of using neem extract as an antibacterial finishing agent for cotton fabric.

Neem seed and bark extract were prepared in methanol using solvent extraction method. The isolation of major limonoids of neem seed and bark extract i.e. azadirachtin A, nimbin and salanin was performed using column chromatography. The identification of compounds was done by using thin layer chromatography (TLC), infrared spectroscopy (IR) and ^{1}H nuclear magnetic resonances (NMR) techniques. The characterization of neem seed and bark extracts in terms of azadirachtin A, nimbin and salannin has been carried out using high performance liquid chromatography (HPLC).

The antibacterial activity of neem extracts and the isolated limonoids was investigated qualitatively by using disc diffusion and parallel streak methods. Both seed and bark extract showed antibacterial activity against gram positive bacteria i.e. *Staphylococcus aureus, Bacillus subtilis* as well as gram negative bacteria i.e. *Escherichia coli*. The minimum inhibitory concentration for seed and bark extract are 8-11mg/ml and 3.3-10 mg/ml respectively. The mechanism of bactericidal action of neem seed and bark extract active compounds on *Staphylococcus aureus* was

investigated using transmission electron microscopy (TEM). Both neem seed and bark extract treated cells show the rupture of cell wall and the loss of cytoplasmic content. The integrity of cell membrane of *Staphylococcus aureus* was studied by determining the release of material absorbing at 260nm. A significant loss of 260nm absorbing material in *Staphylococcus aureus* suspension treated with neem seed and bark extract suggests that nucleic acids are lost because of damage of cell wall. The time for damage of cells was lower in case of bark extract (20 min) as compared to seed extract (40 min).

The neem seed and bark extract were integrated on the cotton fabric substrate along with non formaldehyde, glyoxal/glycol based crosslinking agent. The fabrics were cured using two different curing methods conventional and microwave for crosslinking. The major advantage of using microwave curing method is that it reduces the time of processing as well as retains the mechanical properties of the crosslinked fabric.

The curing conditions of microwave were optimized on the basis of power and time. Low microwave power ~200 W and 1 min curing time was sufficient to impart high crease recovery property to the fabric. Comparisons were made for conventionally and microwave cured cotton fabrics treated with glyoxal/glycol. Crosslinking efficiency in conventionally and microwave cured samples were evaluated on the basis of increase in crease recovery angle and retention of tensile strength and tearing strength after crosslinking. Conventional cured samples showed higher crosslink efficiency than microwave cured samples. FTIR and TGA results also support the higher crosslinking efficiency in conventional curing technique.

The attachment of neem seed and bark extract on to the fabric was confirmed by using IR analysis. Major peaks at 1724 / 1750cm^{-1}(ester and acetate), 1586 / 1582 cm^{-1} and 878 cm^{-1}(β substituted furan ring), 1261cm^{-1} (ether linkage), 1071 / 1025 cm^{-1} (oxide/ether function) for neem seed and bark extract treated fabric after washing clearly indicates that neem seed and bark extract are chemically attached on to the cotton fabric. The range of wt. add-on percent for unwashed samples is 11-22 wt% and for washed samples it decreased to 1-8 wt%.

The antibacterial activity of finished fabric was evaluated qualitatively using parallel streak method and quantitatively using modified colony counting method in which the percent reduction in number of bacterial colonies in treated sample as compared to the untreated sample gives the antibacterial activity of the fabric. The unwashed samples were subjected to parallel streak method to evaluate the antibacterial activity. In general, it was found that as the concentration of neem seed and bark extract increases, the zone of inhibition increases but at still higher concentration it starts decreasing. The washed samples were subjected to the modified colony counting method. It is found that as the concentration of extract on to the fabric increases, the antibacterial activity increases. The concentration (10%w/v) of both the extracts i.e. seed and bark showed high (>94%-99%) antibacterial activity in conventional as well as microwave cured samples as compared to 8-42% antibacterial activity for control samples.

Antibacterial properties of neem seed and bark extract treated fabric and the bacterial growth on the fabric was further examined by scanning electron microscopy (SEM). SEM micrographs, clearly indicates that the neem seed and bark extract act as an effective bactericidal agent on the fabric

The durability of neem seed and bark extract treated fabric was evaluated in term of its retention of antibacterial activity on washing. It was found that the seed extract retains the antibacterial activity up to the five machine washes which is equivalent to approximately 25 home launderings. However, there was sharp decrement in antibacterial activity of bark extract treated fabric as the washing progressed. The semi durable nature of the antibacterial neem extract based finish is attributed to the gradual loss of the crosslinking effect of glyoxal/glycol system after ten washes.

The neem seed and bark extract treated samples were tested for various fabric properties like, appearance, tensile strength, crease recovery angle, tearing strength, bending length etc. The bark extract treated samples were wood colored but the seed extract treated fabric shows no significant change in appearance. Crease recovery angle of neem extract treated fabric slightly decreases but there is no significant change in tensile strength of the treated fabric as compared to the control sample. However, neem finished fabric show improved tearing strength as compared to the control sample.

This study thus reveals that neem seed and bark extracts have a good potential to be used as an ecofriendly and effective agent for antibacterial finishing of cotton based textiles.

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