STUDIES ON DYEABILITY AND ANTIMICROBIAL PROPERTY OF CATIONIC ADDITIVE MODIFIED VISCOSE RAYON FIBRES

by

NILANJANA BAIRAGI

DEPARTMENT OF TEXTILE TECHNOLOGY

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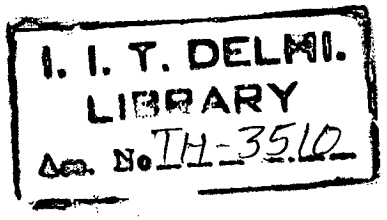
Viscose rayon fibres



CERTIFICATE

This is to certify that the thesis entitled **"STUDIES ON DYEABILITY AND ANTIMICROBIAL PROPERTY OF CATIONIC ADDITIVE MODIFIED VISCOSE RAYON FIBRES"** being submitted by **Ms. Nilanjana Bairagi,** 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. Ms. Nilanjana Bairagi has worked under our guidance and supervision, and fulfilled the requirements for submission of the thesis.

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

(Prof. M. L. GULRAJANI)
Professor,
Department of Textile Technology
IIT Delhi, New Delhi-110 016

(Prof. B. L. Deopura)
Professor,
Department of Textile Technology
IIT Delhi, New Delhi-110 016

This thesis is dedicated to my father (Late Dr. P. Bairagi)

-He loved me enough to grow without his presence

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Nilanjana Bairagi

Nilanjana Bairagi

ABSTRACT

Modified viscose rayon fibres dyeable with reactive dyes (without the addition of electrolyte), acid dyes and metal-complex dyes have been developed. These fibres also exhibit antimicrobial properties. This has been achieved by incorporation of cationic polymers in the cellulose xanthate solution. The cationic polymers used are polydiallyldimethyl ammonium chloride (poly-DMDAAC) and polyamine epichlorohydrin condensate (poly-AECP) based product.

The modified viscose rayon fibres are spun from a solution of viscosity 50-77 sec. The viscosity of the dope increases on incorporation of the cationic polymers. Two sets of modified fibres have been spun in a pilot spinning plant by incorporating 0.2%, 0.4%, 0.6 %, 0.8 % and 1.0 % of poly-DMDAAC and 0.5%, 1 %, 3 % and 5 % of poly-AECP (on the weight of cellulose) in the viscose dope.

FTIR analysis has been carried out to verify the presence of additives in the modified fibres. Additional peaks in the IR spectra confirmed the presence of additive in the cationic modified fibres.

To quantify the additive content in the modified fibres, nitrogen estimation through Kjeldahl method has been done. Further, to evaluate the number of amino groups present in the modified fibres, mono-sulphonic acid dye (CI Acid Red 88) absorption at equilibrium has been estimated. Mono-sulphonic acid dye forms salt linkages with the amino groups of the fibre. Therefore, the amount of mono-sulphonic acid dye taken up is stoichiometrically proportional to the number of cationic sites in the fibre. The nitrogen content of the fibres indicated that the poly-DMDAAC modified fibres retain 65%-80% of the additive and the poly-AECP modified fibres retains 20%-30%, and the remaining additive leaches out of the fibre during coagulation. The nitrogen content is found to have a good correlation with the mono-sulphonic acid dye uptake at equilibrium conditions.

The fibres were characterized using Scanning Electron Microscopy (SEM). The SEM results indicate that there are no changes of the surface properties but the poly-DMDAAC and poly-AECP modified fibres have a thicker skin and a more serrated cross-section than the unmodified fibre. The physical properties of the modified fibres like tensile strength, moisture regain, density and crystallinity have been tested. There is a loss of about 17% in the tensile strength of the poly-DMDAAC incorporated fibres and around 20 % in the poly-AECP incorporated fibres on modification. The total crystallinity index, x-ray crystallinity percentage and the density measurements show a decreasing trend with the increase in the concentration of the additive indicating that poly-DMDAAC as well as poly-AECP cationic modified fibres have lower crystallinity.

The dyeability of the poly-DMDAAC and poly-AECP were studied using four different reactive dyes having different reactive groups. The effect of electrolyte concentration on the extent of dye exhaustion was studied. The fibres were dyed using 10, 20, 40 and 60 g/l of salt. The dye exhaustion and total dye fixation have been estimated. Dye exhaustion above 90 % is obtained without the addition of electrolyte in fibres modified with 0.8% and 1 % of poly-DMDAAC when dyed with the four selected reactive dyes. The other poly-DMDAAC and poly-AECP modified fibres require around 20–40 g/l of electrolyte to achieve 90 % exhaustion of the reactive dyes investigated. However, the reactive dye exhaustion is higher in all the cationic modified fibres as compared to the unmodified viscose rayon fibre, when dyed without the addition of electrolyte. The cationic sites increases the substantivity of the dye towards the fibres and lower crystallinity of the modified fibres makes it more accessible to the dye molecules. The wash fastness and light fastness of the cationic modified fibres have been evaluated. The modified fibres showed wash fastness properties similar to the unmodified fibre, and it is almost '5' in all the cases. However, the light fastness has been slightly affected in the poly-DMDAAC and poly-AECP

fibres having high concentration of cationic additive. A drop of maximum 1 point has been observed in the light fastness.

The dyeability of the poly-DMDAAC and poly-AECP modified fibres was studied using three different acid dyes and three different metal-complex dyes using three levels of shade (2 %, 4 % and 6 % owf). Due to the presence of the cationic groups, all the poly-DMDAAC and poly-AECP modified viscose rayon fibres dyed with acid and metal-complex dyes with dye exhaustion as high as 99%. The plots of dye exhaustions *vs* the nitrogen content in the fibres indicate that the dye exhaustion increases linearly with the increase in the concentration of additive (nitrogen content) in the fibre. The dye exhaustion and *K/S* of the dyed fibres also show a linear relationship. The modified fibres show change in the hue angle with all the acid and metal-complex dyes indicating tonal changes. Tonal changes have been attributed to the aggregation of dye in the fibre. The increase in light fastness of the metal–complex dyed fibres with the increase in *K/S* has been attributed to the aggregation of dye in the modified viscose rayon fibres. The wash fastness of the modified fibres improves on after-treatment with dye-fixing agent.

The antibacterial activities of the modified fibres were assessed against *S.aureus* and *E.coli* bacteria with respect to contact time. Additionally, the antibacterial activity of the washed fibres and the acid dyed fibres were also tested. Poly-DMDAAC and poly-AECP modified viscose rayon fibres both showed antimicrobial activity against *S.aureus* and *E.coli* bacteria but to different extent. Poly-AECP incorporated viscose rayon fibres showed higher antimicrobial activity than those modified by poly-DMDAAC. This has been attributed to the structural differences in the additives and the efficiency of interaction of the additive molecules with the bacteria on the fibre surface. The acid dyed fibres show a significant decrease in the antimicrobial activity. The antimicrobial activity of the modified fibres gets deactivated on repeated washing of the fibres using detergents. However, reactivation of the antimicrobial activity on rinsing with mildly acidified water has been observed. Absence

of the zone of inhibition and growth of microbes on the fibre surface indicates that the cationic additives are not diffusing out of the fibre and the antimicrobial activity is due to the presence of polycations on the fibre surface.

The present study indicates that on incorporation of poly-DMDAAC and poly-AECP in the viscose dope, quaternary amino groups and amino groups are introduced into the cellulosic fibre. The cationic groups improve the substantivity of the anionic dyes towards the fibre. The modified fibres become dyeable with reactive dyes (without the addition of electrolyte), acid dyes and metal-complex dyes. On addition of cationic polymers the crystallinity of the fibres decreases, which further enhances the accessibility of the dye to the fibre.

The modified fibres have 17-20 % lower tensile strength. However, the incorporated cationic groups impart antimicrobial activity to the fibres.

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