SYNTHESIS, CHARACTERIZATION AND PRODUCTION OF X-RAY OPAQUE AND ANTISTATIC POLYESTER FIBRES

By

RAKESH KOUL

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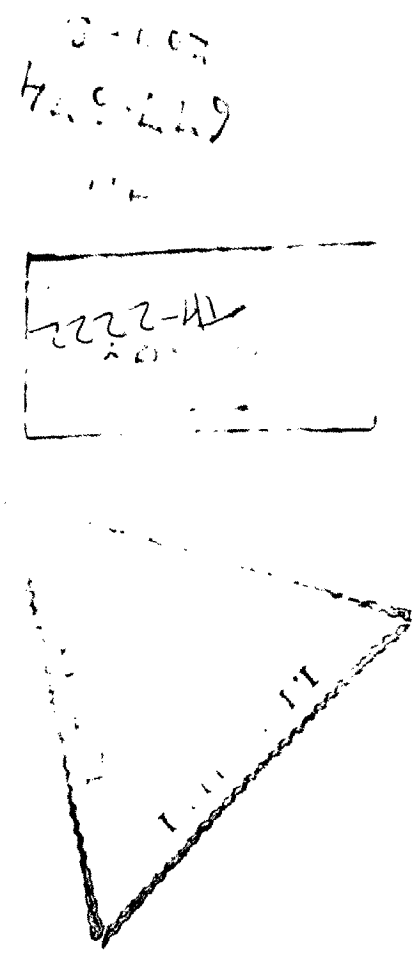

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TO

MY PARENTS

CERTIFICATE

This is to certify that the thesis entitled **"Synthesis Characterization and Production of X-ray Opaque and Antistatic Polyester Fibres"** being submitted by **Shri Rakesh Koul,** 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 him. Shri Rakesh Koul has worked under my guidance and supervision and fulfilled 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.

Prof. (Ms) P. Bajaj
Department of Textile Technology
Indian Institute of Technology
New Delhi-1100016.

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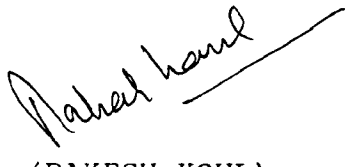

(RAKESH KOUL)

ABSTRACT

Polyester fibres filled with $BaSO_4$ (particle size=0.5 um) have been produced by two routes in order to get X-ray opacity, hygroscopicity and antistatic properties.

Particulate filler, $BaSO_4$ (5-15 wt. %) was added at the time of transesterification alongwith manganese and sodium acetate catalysts. After polycondensation, polyethylene terephthalate [η = 0.57-0.58] produced was melt spun.

In the second route, physical blending of $BaSO_4$ (5-20 wt. %) with commercially available fibre grade PET (η = 0.63) was done using silicone oil (500 CTSK) and titanates, namely neoalkoxy-tri(dioctyl phosphato) titanate (C_1) and neoalkoxy tri(dioctyl pyrophosphato) titanate (C_2) as processing/ blending aids. Melt blending was carried out on a twin screw extruder.

Rheological behaviour of blended PET samples at different shear rates and temperatures (275 to 285^{o}C) was studied and very interesting results on melt viscosity and melt elasticity have been obtained. On the basis of the melt rheology data, melt spinning parameters were set on the spinning rig for producing $BaSO_4$ filled PET fibres.

Differential scanning calorimetry, thermo-gravimetry and hot-stage optical microscopy of $BaSO_4$ filled PET done in order to study the influence of $BaSO_4$ and titanates on the crystallization behaviour and spherulitic growth of PET. From this data, nucleating activity of $BaSO_4$ filler on the crystallization of PET has been demonstrated.

Melt spinning of filled and virgin PET was carried out at 275-285^{o}C under pressure of 18-21 kg/cm^{2} at 400-500 m/min winding speed. Due to the lubricating action of silicone oil and homogeneous dispersion of $BaSO_4$ due to titanates, it was possible to do melt spinning of 15-20% filled PET at a much lower temperature and pressure as compared to neat PET.

As spun filaments were drawn at 90^{o}C and140^{o}C in two stages, and subsequently heat-set at 180^{o}C or 200^{o}C.

Effect of $BaSO_4$ filler on the saponification with 5-10% NaOH solution at 95 ^{o}C for 1 to 2 hr has been studied. Surface morphology of $BaSO_4$ filled PET before and after saponification has been studied using scanning electron microscopy.

Structure of $BaSO_4$ filled PET fibres has been established through DSC, thermo-mechanical analysis, wide angle X-ray diffraction and density measurements.

Addition of $BaSO_4$ to PET fibres improves the antistatic properties as measured through the decay time. The fibres were first charged with Corona for 5 min. With voltage input of 8000 V and then decay time was noted. It was found that by addition of 5% $BaSO_4$ to PET reduces the decay time to 145 sec as compared to neat sample (460 sec).

Dyeing of $BaSO_4$ filled PET fibres with disperse dyes showed higher dye uptake values and dye diffusion coefficient as compared to neat samples. On the whole, by incorporation of $BaSO_4$ (coated with titanates) in PET it is possible to produce X-ray opaque and antistatic PET fibres with higher dye uptake properties.

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