

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

By

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DEPARTMENT OF TEXTILE TECHNOLOGY

THESIS SUBMITTED

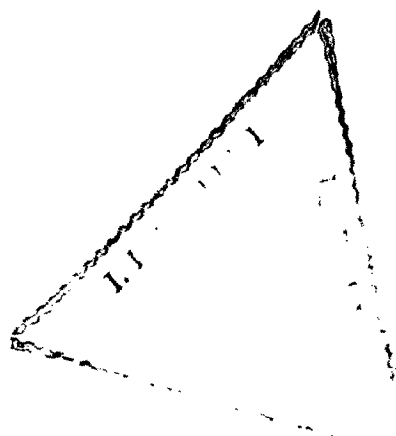
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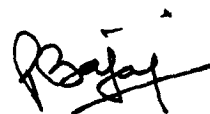
TO

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.



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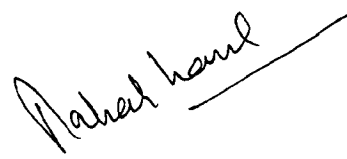
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ABSTRACT

Polyester fibres filled with BaSO_4 (particle size=0.5 μm) 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 [$\eta = 0.57-0.58$] produced was melt spun.

In the second route, physical blending of BaSO_4 (5-20 wt. %) with commercially available fibre grade PET ($\eta = 0.63$) 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°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°C under pressure of 18-21 kg/cm² at 400-500 m/min winding speed. Due to the lubricating action of silicone oil and homogeneous dispersion of BaSO₄ 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°C and 140°C in two stages, and subsequently heat-set at 180°C or 200°C.

Effect of BaSO₄ filler on the saponification with 5-10% NaOH solution at 95°C for 1 to 2 hr has been studied. Surface morphology of BaSO₄ filled PET before and after saponification has been studied using scanning electron microscopy.

Structure of BaSO₄ filled PET fibres has been established through DSC, thermo-mechanical analysis, wide angle X-ray diffraction and density measurements.

Addition of BaSO₄ 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% BaSO₄ to PET reduces the decay time to 145 sec as compared to neat sample (460 sec).

Dyeing of BaSO₄ 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 BaSO₄ (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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