MATERIALS SCIENCE

FLAMEPROOFING PROPERTIES
OF PHOSPHORUS-CONTAINING
POLYESTER MATERIALS

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The flameproofing indexes of polyester materials made of polymers containing 2-methyl-2,5-dioxo-1-oxo-2-
phospholane added during copolycondensation were investigated. It was found that such process indexes as
the effective melt viscosity and molecular weight of the polymer determine the flameproofing indexes of the
materials obtained.

The fire safety of materials can be ensured by developing new, effective methods of reducing the combustibility of
polymers, including poly(ethylene terephthalate). Polyester fibres and yarns occupy the leading positions among chemical
fibres with respect to production and consumption volumes and are widely used both in pure form and blended with other
fibres where problems of fire safety are extremely pressing: as decorative and upholstery materials, tulle drapery articles, work
clothes, etc. The study of thermal and thermooxidative degradation of polyester materials in the presence of flame retardants
(FR) of different composition showed that phosphorus-containing FR should be used to reduce their combustibility. These FR
slow thermooxidative degradation in the temperature range from the onset of intensive decomposition of the modified polymer,
decrease the intensity of separation of hot volatile products of degradation, and increase the carbonizability of the polymer [1].

Three directions can be distinguished in modification of poly(ethylene terephthalate) to reduce the combustibility
[2, 3]: addition of FR to the polymer melt; chemical modification; surface treatment of the finished fabric. The first two
methods are the most effective.

Addition of FR to the polymer melt allows using ordinary polymer processing technology and ensures stability of the
flameproofing effect in repeated washings. The difficulty of selecting the FR prevents wide use of the method, since it must
retain thermal stability up to 300°C, be easily dosed, melt during processing of the polymer, or have a high degree of dispersion.

Chemical modification consists of reaction of the FR with the functional groups of the polymer during synthesis, i.e., the
FR is added to the polycondensation reaction in different stages. This method of modification of the polymer is used on the
industrial scale in Germany, which manufactures a flameproof fibre under the trade name of Trevira CS. 2-Methyl-2,5-dioxo-
1-oxo-2-phospholane is used as the FR in production of Trevira CS [4]. For a 0.6-0.8% phosphorus content in the copolymer,
the oxygen index of the material attains 28-29%.

However, incorporation of FR in the polymer chain can result in structural and chemical inhomogeneity of the polymer
and increase the number of defects. The resulting change in the properties of the polymer, including the melting point and melt
viscosity, not only complicate further processing of the modified polymer, but can also affect the flameproofing indexes of the
materials obtained.

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TABLE 1. Characteristics of Samples of Polyester Fabrics

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Surface density, g/cm²</th>
<th>P content, %</th>
<th>OI, %</th>
<th>Inflammability (GOST 50810-95)</th>
<th>CR, % at 350 °C</th>
<th>CR, % at 400 °C</th>
<th>ηe, Pa sec</th>
<th>MW</th>
<th>DP</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>295</td>
<td>0.85</td>
<td>27.1</td>
<td>HF</td>
<td>75.0</td>
<td>16.5</td>
<td>1657</td>
<td>20830</td>
<td>105</td>
</tr>
<tr>
<td>2</td>
<td>295</td>
<td>0.86</td>
<td>30.8</td>
<td>DI</td>
<td>76.0</td>
<td>18.0</td>
<td>2100</td>
<td>23630</td>
<td>120</td>
</tr>
<tr>
<td>Initial</td>
<td>295</td>
<td>–</td>
<td>20.0</td>
<td>HF</td>
<td>80.2</td>
<td>16.0</td>
<td>–</td>
<td>26800</td>
<td>135</td>
</tr>
</tbody>
</table>

Notation: OI - oxygen index; CR - carbonized residue; ηe — effective viscosity; MW — molecular weight; DP — degree of polymerization; HF — highly flammable, DI — difficultly ignitable fabrics.

![IR spectra of phosphorus-containing polyester](image1)

Fig. 1. IR spectra of phosphorus-containing polyester: 1) sample 1; 2) sample 2.

![DSC curves of samples](image2)

Fig. 2. DSC curves of samples 1 (a), 2 (b), and unmodified polyester (c).

We investigated the effect of 2-methyl-2,5-dioxo-1-oxo-2-phospholane incorporated in the polymer chain in copolycondensation on the flameproofing and process indexes of polyester. The fabric samples selected for the study were characterized by the same surface density and phosphorus content but differed in the molecular weight of the polymers used to manufacture them and the effective melt viscosity (Table 1).

The structure of the samples of flameproof polyester fabrics was investigated by IR spectroscopy. The survey spectra were made on a Specord M80 in the λ frequency region from 4000 to 400 cm⁻¹ (Fig. 1). The spectrum of sample 1 had an absorption band in the region of λ 1550 cm⁻¹ characteristic of stretching vibrations of a carboxyl group. The intensity of the absorption band in the spectrum of sample 2 was much lower. As a consequence, sample 2 was made of a polymer containing a smaller amount of terminal carboxyl groups and was thus characterized by a higher molecular weight in comparison to sample 1.

Differential scanning calorimetry (DSC) was used to study the thermophysical characteristics of the fabric samples. The studies were conducted on a DSC Q 10 differential scanning calorimeter from TA Instruments. The polymeric materials