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POLYCONDENSATION OF POLYETHYLENETEREFTHALATE AT SOLID PHASE

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Abstract
Acceleration of technical progress, increase in the assortment of chemical products, increase in labor productivity and quality of products from plasmas are to a certain extent related to acquisition and use of new types of polymer materials. With the development of air and space mechanics, the need for polymers with high atmospheric tolerance, heat, and long service life is increasing. Today, polyethylene terephthalate is used in the production of films, microorganism and mold resistant synthetic fibers, radio parts, and chemical equipment. This article describes the polycondensation process of polyethylene terephthalate in the solid phase.

Keywords: polyethylene terephthalate, granules, ethylene glycol, carboxyl number, crystallinity, degree of polycondensation, hydroxyethyl groups, polyester.

Introduction

During the crystallization of amorphous polyethylene terephthalate, the polycondensation reaction takes place parallel to the main process, as a result of which the degree of polycondensation of polyester increases from 80-100 to 160 and above. The chemistry and mechanism of the reaction is almost unchanged and remains the same as in the steps of liquid phase polycondensation. As a result of the reaction, the length of polyester macromolecules increases and ethylene glycol is released.

An important difference from the liquid phase version of the reaction is the absence of mixing from the polyester mass and a very low rate of ethylene glycol removal from the mass. Diffusion of ethylene glycol from the depth of the polyether mass to the surface of the granules occurs with the reaction of glycolysis of macromolecules, with a change in their type in spherulites.

Oligomerization processes develop on the surface of the granules, which occur with the formation of finely dispersed polyester. Different mechanical loads lead to different deformations of polyethylene terephthalate granules.

At the beginning of the solid-phase polycondensation process, the condensation of the hydroxyethyl ends of macromolecules in the polyester mass is quite high and can be roughly estimated by the value of the carboxyl number of the amorphous product. On average, 3-4 hydroxyethyl groups correspond to each carboxyl group, that is, when the carboxyl number is 40-45 mg-eq/kg, the concentration of hydroxyethyl groups is 160-180 mg-eq/kg does. At such a concentration and at a much higher temperature, the hydroxyethyl ends of the chains of macromolecules, which are in constant oscillating motions (especially pendulum oscillations of the end fragments), are in the zones of mutual comfort. This leads to a reactive interaction with the growth of the macromolecule chain and the release of the ethylene glycol molecule.

The polycondensation reaction develops during the initial crystallization of polyethylene terephthalate granules in the "boiling bed" and crystallization in the moving mass. In the second stage of crystallization, the temperature rises to 208-212 ℃, and the polycondensation process proceeds at a much faster rate. Figure 1 shows the dependence of granule weight loss on heating time at 212 ℃. It can be seen that the maximum rate of ethylene glycol release is observed during the first hour of heating.

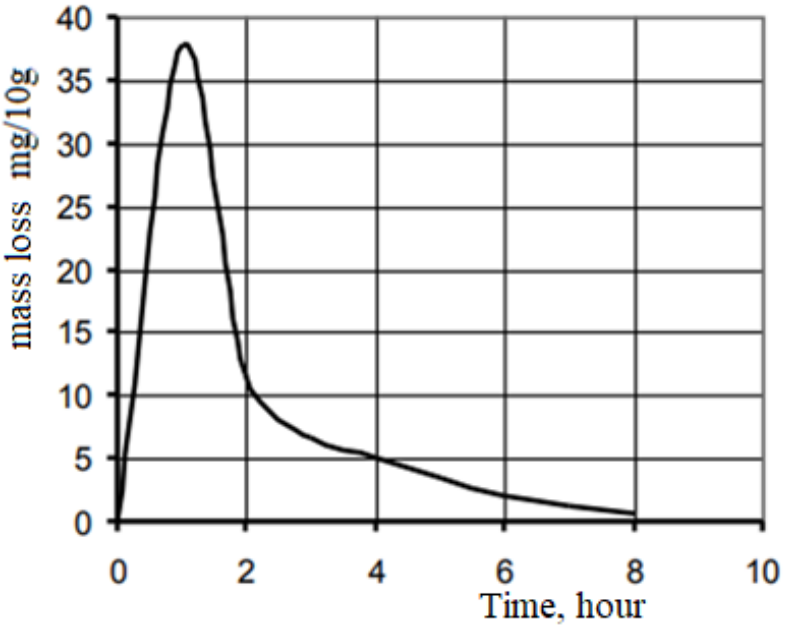

Figure 1. Dependence of granule weight loss on heating time at 212 ℃

In static conditions, the release of not only ethylene glycol, but also mono- and polyether diglycols can occur in the polymer mass. Due to the large molecular mass of the latter, ethylene glycol is not able to diffuse into solid polyester. The interaction of hydroxyethyl groups occurs simultaneously throughout the entire mass of polyester, as a result of which the concentration of ethylene glycol in the volume of polyester increases. Unlike short-chain polyester molecules,

ethylene glycol is able to diffuse polymer particles on the surface and leave it with a hot stream of nitrogen from the reaction zone at a high temperature.

The process of removing ethylene glycol from the polymer depends on its concentration in the polyester and gas phase. The rate constant of this process can be expressed by formula (1):

$$k=k_i([C]_{gf} / [C]_{tf})(1)$$

where k_i - is a coefficient that depends on the speed of the chemical reaction of polycondensation, the nature and speed of diffusion of ethylene glycol in the polymer mass, and the speed of the return process of leaving the surface of polymer particles by ethylene glycol;

C_{gf} – concentration of ethylene glycol in the gas phase;

C_{tf} – concentration of ethylene glycol in the solid phase.

Since the concentration of ethylene glycol in the gas phase C_{gf} in a large flow of nitrogen is very small and tends to the minimum, the diffusion rate can be considered approximately proportional to the concentration of ethylene glycol in the solid phase, that is, in the polyester mass. The time it takes for ethylene glycol to leave the polyester mass depends on the particle size. Ethylene glycol is released faster from smaller particles, which means that the polycondensation process ends earlier in them.

The ordered selection of lamellar structures helps the movement of ethylene glycol molecules along macromolecular chains, which are connected with polyester molecules by relatively weak hydrogen bonds and intermolecular interaction forces. In lamellar structures, the movement of ethylene glycol molecules perpendicular to the chains of macromolecules can also be due to the reorientation of hydrogen bonds from one chain to another nearby parallel chain.

Movement of ethylene glycol molecules in such a mechanism becomes difficult in the amorphous spheres of crystalline polyether. Chains of macromolecules are arranged in such areas in an irregular, partially confused state. the movement of ethylene glycol molecules through such areas is accompanied by glycolysis of polyester, and they contribute to the reduction of the degree of disorder of amorphous areas. Macromolecules disintegrate, become more ordered, and condense again with mutually favorable reaction centers. Glycolysis also occurs in crystallites, but less often. From what has been said, it can be assumed that the speed of movement of ethylene glycol molecules and their exit from the reaction zone in the crystalline areas of the polyester is higher than in the amorphous ones.

Ethylene glycol molecules change direction while moving to the edge. If they come across carboxyl groups, they interact with them. The resulting water molecule hydrolyzes the polyester with the formation of new molecules. Secondary ethylene glycol is released on the surface of phase separation as the molecule aspires. Since polycondensation takes place in the entire volume of the polyester, the only outlet of ethylene glycol is the phase separation surface. Due to the departure of molecules from the surface of the granules, a concentration gradient is formed, which is considered as a factor directing the molecules of ethylene glycol to the surface of the granules.

Ethylene glycol formed and adhering to the surface of the granule must evaporate and leave the reaction zone together with the nitrogen flow. In practice, it happens differently. The rate of evaporation of ethylene glycol from the surface of granules is smaller than the rate of diffusion from the volume of granules to their surface. As a result, ethylene glycol forms a layer and begins to accumulate on the surface. At a temperature of 208-212 ℃, polyethylene terephthalate enters

the glycolysis reaction and causes the breakdown of macromolecules into oligomers. Granular surface is covered with a thin film of oligomer liquid. A fast stream of hot nitrogen vaporizes and removes ethylene glycol, shifting the balance of the glycolysis reaction toward polyester formation. The product can be covered in it. Raising the temperature helps this process. Gradually, the reaction ends (6-8s) and granules become a product.

When an amorphous granule with a degree of polycondensation of 80-100 and does not contain free ethylene glycol and water enters the stage of solid phase polycondensation, the process of expelling ethylene glycol and granulate sanding does not cause adhesion. The resulting pieces are easily crushed and form a homogeneous granule.

The situation changes for the worse when amorphous granules with a degree of polycondensation less than 80 are received. A rather thick layer of oligomer solution is formed on the surface of the granules, and the granule mass does not have time to give ethylene glycol. The lumpy mass does not pass nitrogen well. A channel is formed in the mass of granules, and most of the nitrogen is released through these channels without fulfilling its function. When the granulate mass moves, it approaches the brackets of the thermocouples and deforms them. A semi-liquid solution of oligomers adheres to the surface of the brackets and forms semicircular protrusions.

The appearance of the granules is an elliptical cylinder with a white color mass of 17 ± 2mg (the weight of the granules can be changed up to 20 ± 2mg) with a non-glossy surface.

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