
**SYNTHESIS AND PROCESSING OF POLYMERS
AND POLYMERIC COMPOSITES**

**СИНТЕЗ И ПЕРЕРАБОТКА ПОЛИМЕРОВ
И КОМПОЗИТОВ НА ИХ ОСНОВЕ**

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RESEARCH ARTICLE

Obtaining phthalate substituted post-consumer polyethylene terephthalate and its isothermal crystallization

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Abstract

Objects. Due to the polymer waste accumulation, the search for new directions for their utilization is urgent. Chemical recycling methods are of considerable interest, which allow one to obtain the original monomers or change the compositions of the copolymers. From the point of view of building a circular economy, a promising material is polyethylene terephthalate (PET), on the basis of which amorphous copolyesters can be obtained. The study aimed to analyze the simultaneous glycolysis and interchain exchange reactions of PET in the presence of the oligoethylene phthalate modifier with hydroxyl end groups and the study of isothermal crystallization of poly(ethylene phthalate-co-terephthalates) with different phthalate contents obtained in this way.

Methods. Oligoethylene phthalate is synthesized by polycondensation. Poly(ethylene phthalate-co-terephthalates) were obtained by the interaction of post-consumer PET with oligoethylene phthalate. The composition of the oligomer and copolymers was confirmed using Fourier-transform infrared spectroscopy, thermal characteristics and crystallization half-times were determined by differential scanning calorimetry.

Results. In this work, the use of the post-consumer PET chemical recycling process, aimed at obtaining copolyesters under the influence of small modifier amounts was proposed. The process consisted in carrying out the combined interchain exchange and degradation with a complex oligoester different from PET. Poly(ethylene phthalate-co-terephthalate) copolymers were obtained

via reaction of post-consumer poly(ethylene terephthalate) flakes and synthesized oligoethylene phthalate resin in the melt phase in the absence of catalyst. The effect of phthalate concentration in polymer on the isothermal crystallization of phthalate substituted poly(ethylene terephthalate) was estimated.

Conclusions. The hypothesis about the possibility of using an oligoester modifier to obtain the PET-based copolymer at the high rate and without reducing the molecular weight to values characteristic of a monomer or oligomer has been confirmed. The process can be used to obtain random copolyesters based on post-consumer PET. The phthalate unit concentration increase is followed by decrease in the glass transition temperature, temperature and heat of fusion, and increase in crystallization half-times. Phthalate has a better ability to retard PET crystallization than 2-methyl-1,3-propanediol or furandicarboxylic acid, but is inferior to some of the other modifiers known.

Keywords: polyethylene terephthalate, PET, copolyester, chemical recycling of PET, glycolysis, degradation, interchain exchange, isothermal crystallization

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НАУЧНАЯ СТАТЬЯ

Получение фталатзамещенного вторичного полиэтилентерефталата и изучение его изотермической кристаллизации

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Аннотация

Цели. Накопление полимерных отходов в последнее время обуславливает поиск новых подходов к их утилизации. Значительный интерес представляют химические способы вторичной переработки, которые позволяют получить исходные мономеры или изменить составы сополимеров. С точки зрения построения экономики замкнутого цикла перспективным материалом является полиэтилентерефталат (ПЭТ), из которого в процессе химического рециклинга получают аморфные сополиэфир. Работа посвящена исследованию одновременного протекания реакций гликолиза и межцепного обмена ПЭТ в присутствии модификатора олигоэтиленфталата с гидроксильными концевыми группами и изучению изотермической кристаллизации поли(этилен фталат-со-терефталатов) с разным содержанием фталата, полученных таким способом.

Методы. Олигоэтиленфталат синтезирован поликонденсацией. Поли(этилен фталат-со-терефталаты) получены взаимодействием вторичного ПЭТ с олигоэтиленфталатом. Состав олигомера и сополимеров был подтвержден с использованием ИК-Фурье спектроскопии, термические характеристики и полупериоды кристаллизации определяли методом дифференциальной сканирующей калориметрии.

Результаты. Разработан процесс получения сополиэфиров, основанный на химическом рециклинге вторичного ПЭТ под действием малых количеств модификатора. Отличительной особенностью процесса является одновременное протекание реакций межцепного обмена и деструкции сложным олигоэфиром, отличным по природе от ПЭТ. Реакцией в расплаве вторичного ПЭТ и синтезированного олигоэтиленфталата в отсутствие катализатора были получены поли(этилен фталат-со-терефталаты). Изучено влияние концентрации фталата в полимере на температуру стеклования, температуру и теплоту плавления, изотермическую кристаллизацию фталатзамещенного ПЭТ.

Выводы. Подтверждена гипотеза о возможности использования олигоэфирного модификатора для получения сополимера на основе ПЭТ с высокой скоростью и без снижения молекулярной массы до значений, характерных для мономера или олигомера. Процесс может быть использован для получения статистических сополиэфиров на основе вторичного ПЭТ. С увеличением концентрации звеньев фталата происходит снижение температуры стеклования, температуры и теплоты плавления, увеличение полупериодов кристаллизации. Фталат обладает лучшей способностью замедлять кристаллизацию ПЭТ, чем 2-метил-1,3-пропандиол или фурандикарбоновая кислота, но уступает некоторым другим известным модификаторам.

Ключевые слова: полиэтилентерефталат, ПЭТ, ПЭТФ, сополиэфир, химический рециклинг ПЭТ, гликолиз, деструкция, межцепной обмен, изотермическая кристаллизация

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INTRODUCTION

Currently, an urgent problem is the processing of polymer waste, a significant part of which is a large-tonnage polyester—polyethylene terephthalate (PET) [1].

The most promising are PET processing methods based on chemical reactions, while the most studied are degradation reactions: glycolysis, hydrolysis, alcoholysis, acidolysis, and others [2–4].

Among them, PET glycolysis is of considerable interest. Heterogeneous glycolysis is the most widespread; however, examples of the process under homogeneous conditions both in solution [5–7] and in melt [8–10] are also known. The degradation of PET by oligoesters is of particular interest. The possibility of PET glycolysis with oligoethylene terephthalates of various molecular weights was described in [7]. A similar process was also studied

in [10], however, in this case, glycolysis is carried out with large amounts of a modifier (an oligoester, which is different in nature from PET), which leads to a low molecular weight product. At the same time, it is known that to obtain a higher molecular weight product, it is necessary to use low concentrations of the glycolysis agent [6]. Since the authors obtained a random copolymer in their work, in their study glycolysis was combined with another chemical processing method—interchain exchange.

Today, the interchain exchange reaction for the modification of PET macromolecules in industry is less significant than glycolysis [11–14]. Although the polymeric materials produced in this way may be contaminated with additives from the original PET composition or degradation products, the interchain exchange makes it possible to obtain copolyesters in a single step and at a relatively low energy cost. The interchain exchange reaction

rate depends on the concentration of end groups in the reaction space [12, 14], the content of which will be higher if the same amount of modifier is used, but with a low molecular weight.

Most often, the purpose of such a modification is to obtain amorphous polymers [1] with lower crystallinity and longer crystallization half-time. For example, to obtain an amorphous copolymer, a number of units, such as units of 1,4-cyclohexanedimethanol (CHDM) [15–19], CHDM and isophthalate [20], only isophthalate [21], 2,5-furandicarboxylic acid as an alternative to isophthalate [22], 1,3-propanediol [23], 2-methyl-1,3-propanediol [24], and tricyclodecane dimethanol [25] are introduced into the PET chain. Amorphous terephthalate-based polymers, as well as CHDM and isosorbide [26], CHDM, 1,4-butanediol, and oligo-L-lactide [27] are also known. One of the poorly studied comonomers is inexpensive phthalate [28, 29]. The products of glycolysis, methanolysis, and hydrolysis of such a copolymer are easily separated.

The aim of this work is to study the simultaneous reactions of glycolysis and interchain exchange of PET in the presence of small amounts of oligoethylene phthalate (OEP-1) modifier with hydroxyl end groups, as well as to study the isothermal crystallization of poly(ethylene phthalate-co-terephthalates) (PEPT) with different content of phthalate obtained in this way. The use of a low molecular weight modifier in the interchain exchange reaction should make it possible to achieve a high process rate [12, 14] and avoid thermal degradation, and at the studied phthalate concentrations, a decrease in molecular weight will have almost no effect on the hard-chain PEPT properties.

EXPERIMENTAL

As the main raw material, transparent PET flakes were used, which are industrial products of crushing PET containers [7]. To calculate the degree of polycondensation, we used the data of [8], which indicated the average molecular weight equal to 26000 g/mol. The PET molecular weight value corresponds to the degree of polycondensation equal to 135.

Phthalic anhydride, chemically pure (*Sigma-Aldrich*, USA) was used without preliminary purification. Ethylene glycol, chemically pure (*Sigma-Aldrich*, USA) was pre-purified by vacuum distillation prior to use.

OEP-1 was obtained by the phthalic anhydride and ethylene glycol polycondensation. Ethylene glycol in a molar ratio of 1.25:1 was added to molten phthalic anhydride at 140°C in a 250 mL flask equipped with

a Liebig condenser. The mixture was kept for 1.5 h. The temperature was maintained using a heating mantle. After that, the temperature was raised to 190°C for 3 h, then the mixture was kept under a vacuum of 40 mbar until the end of the evolution of the water formed in the reaction.

PEPT of various compositions was obtained by the interchain exchange reaction in the melt in the absence of a catalyst. Pre-prepared PET flakes and synthesized OEP-1 were co-melted in a flask. The melt was stirred at a temperature of 280°C in an inert nitrogen gas stream until a constant viscosity was reached. The time to reach the melt constant viscosity was no more than 1.5 h for all samples.

Differential scanning calorimetry (DSC) curves were obtained on a DSC 204 F1 Phoenix calorimeter (*NETZSCH Gerätebau GmbH*, Germany) in an inert medium (argon) at the 10 deg/min scanning rate, as well as in an isothermal mode.

The composition of the products was confirmed by Fourier transform infrared spectroscopy (FTIR) on a Spectrum 65 FT-IR spectrometer (*Perkin Elmer*, USA) with a resolution of 1 cm⁻¹ in the range from 500 to 4000 cm⁻¹ at a temperature of 25°C. The oligomer average molecular weight value was determined from the infrared spectrum according to the procedure [6, 30].

RESULTS AND DISCUSSION

The resulting oligoethylene terephthalate was examined by DSC. The absence of crystallization, melting, and the heat capacity increase peaks on the DSC curve indicates the formation of the completely amorphous oligomer. The oligomer average molecular weight value determined from the FTIR spectrum (Fig. 1) was 800 g/mol. The oligomer degree of polycondensation is approximately 4.

Thermal properties and isothermal crystallization were studied by DSC. The thermal characteristics of the obtained copolymers are given in the table.

In certain scientific literature, one can find disagreements in the thermal characteristics values of such copolymers [28, 29]. This may be due to both the difference in molecular weight and the composition of the original PET. Despite this, the obtained thermal characteristics correlate well with the known data both on the glass transition temperatures [28] and on the decrease in heat levels [28] and melting temperatures [29]. With the sample phthalate to terephthalate ratio increase from 0 (sample PET-1) to 15 (sample PEPT-15:85), the decrease in the melting point from 250 to 221°C and the heat of fusion from 71 to 45 J/g

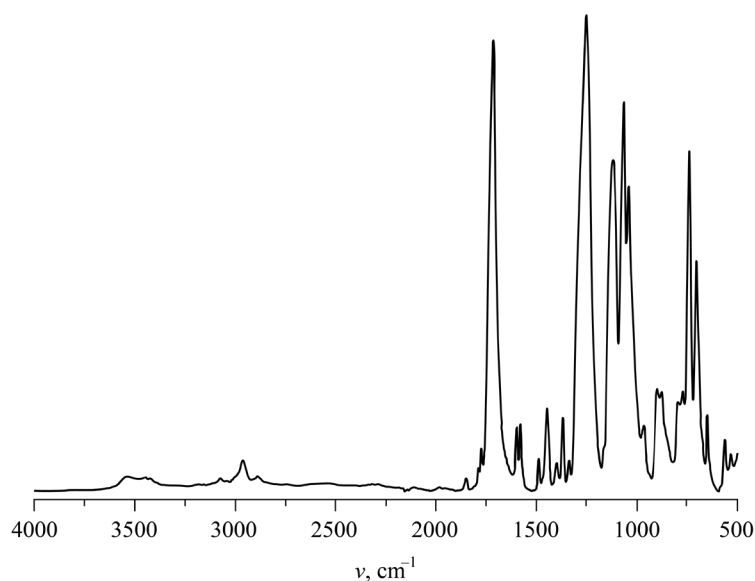


Fig. 1. Fourier-transform infrared (FTIR) spectrum of the OEP-1 sample (oligoethylene phthalate).

Table. Composition, polycondensation degree and thermal characteristics of copolymers

Polymer	Terephthalate to phthalate ratio	Degree of polycondensation	Glass transition temperature, °C	Melting point, °C	Heat of fusion, J/g
PET-1	100:0	135	78.4	250.4	71.2
PEPT-5:95	95:5	53	75.3	242.4	61.1
PEPT-10:90	90:10	33	72.2	233.3	53.6
PEPT-15:85	85:15	24	69.5	220.8	45.4

confirms the crystal structure violation at the chain regularity changing. There is also a decrease in the glass transition temperature from 78 to 70°C with the same increase in the phthalate concentration in the polymer.

The obtained PEPT composition was confirmed by the FTIR spectroscopy. The position and intensity of the peaks in the spectra of the samples (Fig. 2) corresponds to the theoretical data [6, 29].

The crystallization half-times were calculated from the isothermal DSC curves of the copolymers. The half-times of crystallization of copolymers at different temperatures are shown in Fig. 3.

Crystallization half-times with increasing comonomer concentration show the growth typical of PET-based copolymers.

In terms of crystallinity reduction, phthalic anhydride as a modifier is inferior to CHDM [15] and isosorbide [31], but is superior to 2-methyl-1,3-propanediol [24] and furandicarboxylic acid [32].

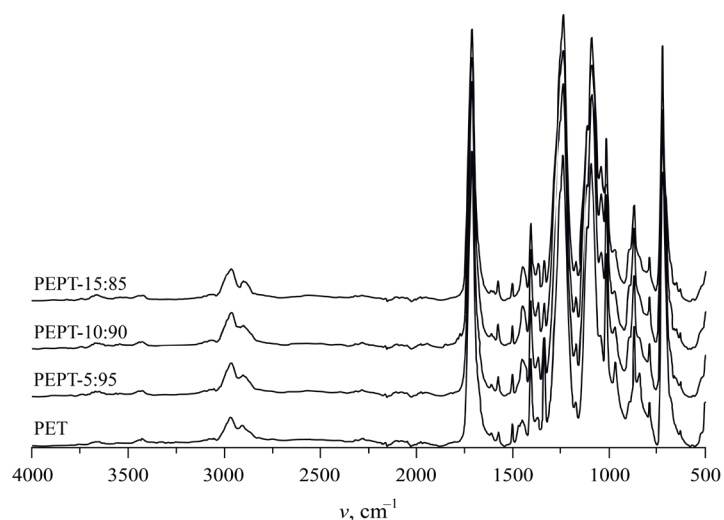


Fig. 2. FTIR spectra of PET, PEPT-5:95, PEPT-10:90, and PEPT-15:85 samples.

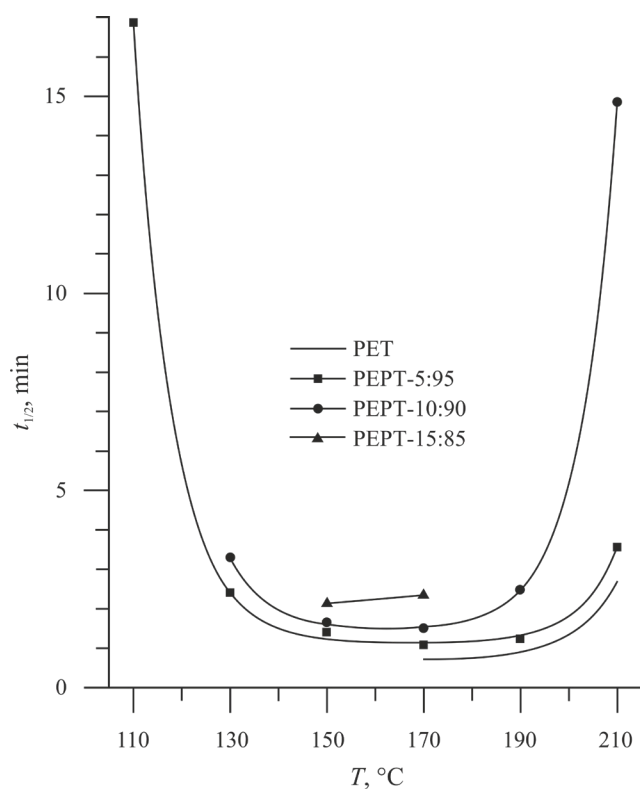


Fig. 3. Isothermal crystallization half-times $t_{1/2}$, min, of PET samples [32], PEPT-5:95, PEPT-10:90, and PEPT-15:85 at different temperatures T , °C.

CONCLUSIONS

The simultaneous glycolysis and interchain exchange reactions can be used as a method for obtaining amorphous copolyesters based on recycled PET. The hypothesis about the possibility of using

a small amount of an oligoester modifier to obtain a PET-based copolymer at the high rate and without reducing the molecular weight to the values characteristic of the monomer or oligomer was confirmed.

The introduction of phthalate into PET macromolecules makes it possible to reduce the glass transition temperature, and temperature and fusion heat of the copolymer. It is also possible to observe a slowdown in crystallization with an increase in the concentration of phthalate, as evidenced by the crystallization half-time. Comparison of the PEPT crystallization isotherms with crystallization isotherms of other copolyesters leads to the conclusion that phthalate has a good ability to slow down crystallization.

Authors' contributions

K.A. Kirshanov – planning and conducting research, collecting and analyzing experimental materials, writing the manuscript;

A.Yu. Gervald – management and scientific consulting;

R.V. Toms – scientific consulting and assistance in processing the results obtained;

A.N. Lobanov – management and preparation of material for publication.

The authors declare no conflicts of interest.

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