

STUDYING THE CONDITIONS FOR THE FORMATION OF POLYETHYLENE TEREPHTHALATE, WHICH FORMS A PRIMARY FIBER ON THE BASIS OF SECONDARY POLYETHYLENE TEREPHTHALATE RAW MATERIALS

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Abstract. Polyethylene terephthalate is widely used in the manufacture of products, materials and parts in many areas of the economy and is the second largest polymer in terms of production. In Uzbekistan, about 56 thousand tons of polyethylene terephthalate (PET) plastic bottles are produced annually.

Keywords: secondary polyethylene terephthalate, alcoholysis, ethylene glycol, bis (hydroxy ethylene terephthalate), synthesis, polyethylene terephthalate, fiber-forming, physicochemical and technological properties.

Introduction. Polyethylene terephthalate is widely used in the manufacture of products, materials and parts in many areas of the economy and is the second largest polymer in terms of production.

In Uzbekistan, about 56 thousand tons of polyethylene terephthalate (PET) plastic bottles are produced annually [1]. At the same time, the amount of polyethylene terephthalate household waste is 1,65 kg per capita. About 10 factories in the country recycle used PET bottles, mainly on flexi. This waste is an ideal source of raw materials for the production of synthetic fibers [2]. It should be noted that after the use of PET containing containers, its properties change for the worse, i.e., there is a decrease in its average molecular weight and, thus, changes in its structure, physical and mechanical properties. In addition, after using PET, the container becomes contaminated and for its reuse it is necessary to clean it from this dirt, as well as impurities of other polymers (labels, corks, etc.). This leads to the fact that obtaining flexi requires a large amount of water, detergents, as well as a technological stage of purification from impurities of other polymers.

For the Republic of Uzbekistan, the most acceptable option for recycling PET-containing waste is its decomposition to primary monomers to obtain fiber-forming PET [3,4]. There are several ways to obtain monomers - hydrolysis with the formation of paraterephthalic acid and ethylene glycol and alcoholysis with the formation of bis 2-hydroxyethylene terephthalate (BHET) [5]. Of the following methods, alcoholysis of secondary PET with ethylene glycol is more

accessible and technologically advanced, which allows you to immediately obtain BHET - a compound that is the product of the reaction of dimethyl terephthalate (DMT) with ethylene glycol (EG), which is formed in the production of PET. In this case, it is possible to exclude the stage of the reaction of DMT with EG [6].

For a long time at the Department of "Chemical technology of high molecular weight compounds and plastics" named T.R. Abdurashidov of the Tashkent Institute of Chemical Technology in the polymer school of F.A. Magrupov, research is being carried out, in particular, on the chemical disposal of PET-containing household waste [7,8]. To date, the team of this school has discovered the mechanisms of alcoholysis of secondary PET (SPET) with polyhydric alcohols, unsaturated polyesters have been proposed on their basis for the production of fiberglass pipes and polymer-composite pipes that transport water, scientific developments are underway on the synthesis of saturated polyether polyols for the production of polyurethane materials and fiber-forming polyethylene terephthalic with properties inherent in primary PET [9-11].

In light of the above, the goal of this work was to optimize the conditions for alcoholysis of SPET with ethylene glycol and the synthesis of fiber-forming primary PET from alcoholysis products.

Research methods

We used recycled polyethylene terephthalate with a density of 1380-1400 kg/m³ in the form of shapeless particles with a size of 2-4 mm, ethylene glycol according to GOST 19710 with $\rho=1,1171$ g/cm³, dimethyl terephthalate according to GOST 11363-91 with $\rho = 1,21$ g/cm³, zinc acetate according to GOST 5823-78 with $\rho=1,735$ g/cm³.

BHET from SPET was obtained according to the method described in [12].

Synthesis of BHET based on dimethyl terephthalate and ethylene glycol

In a four-necked flask equipped with a capillary for supplying an inert gas, a stirrer, a thermometer, a reflux condenser, a Dean-Stark trap, DMT and EG are loaded in a molar ratio of 1: 2,5, zinc acetate 0.5% with respect to SPET. The process is carried out at (180-190)°C for 3-6 hours until the (85-90)% of methyl alcohol is distilled off, then the temperature is raised to (260-280)°C until the evolution of methanol ceases and cooled. The finished product is poured hot into a dry, clean dish.

Synthesis of PET from the resulting BHET

BHET is loaded into a 2-necked flask equipped with a capillary for supplying an inert gas, a thermometer, a reflux condenser and a tube connected to a vacuum pump. Then the temperature is raised in steps: first, to 240°C at a residual pressure of -0.97 kg/cm², hold for 40 minutes, then to 280°C at a residual pressure of -0,97 kg/cm² until all ethylene glycol is distilled off (1.0-1 , 5 hours). At the end of the reaction, the finished PET is poured onto a thin stainless steel sheet and cooled, after which it is crushed [13].

The physicochemical properties of the synthesized BHET, PET were studied by the following methods: the content of hydroxyl groups - GOST 25261-82 [14], the average molecular weight - GOST 10028, the melting point - TC 25-11-398-69 [15]. IR spectra of VPET, BHET and PET were recorded on an FT- Ir spectrum Perkin Elmer instrument.

Discussion of experimental results

The chemical method for processing SPET is based on the use of destruction reactions in the presence of chemical agents. Usually these are reverse reactions in relation to the reactions of formation of high molecular weight compounds. Low molecular weight products released during

the formation of polymers, starting monomers and / or compounds capable of entering into exchange reactions with the polymer can be used as a chemical destructive agent. In this case, depending on the nature, functionality and amount of the destructive agent, the decomposition products can be the initial monomers, adducts, oligomeric products.

The starting monomers from SPET can be obtained either by hydrolysis in an alkaline or acidic medium, or by glycolysis (ethylene glycol) [16].

One of the most common monohydric alcohols is methanol. It can be used to obtain the starting PET monomers. The methanolysis of SPET mainly produces ethylene glycol and dimethyl terephthalate. In Japan, high-tech methods have been developed for processing used PET bottles into feedstock (terephthalic acid and dimethyl terephthalate), based on the use, in particular, of supercritical methanol [17]. In [18], methanolysis is carried out in an autoclave at a temperature of 220 °C and elevated pressure in the presence of a strong inorganic acid as a catalyst. It should be noted that recently, scientists from developed countries such as the USA, Japan, China, Korea have been researching and developing technologies for producing bishydroxyethylene terephthalate using methanolysis, which is used as a feedstock for PET synthesis [19].

Another and the most common method for producing BHET is alcoholysis of SPET with ethylene glycol. The efficiency of alcoholysis and the yield of BHET depends on the nature of the catalyst used, on the molar ratio of the starting compounds, and also on the duration of the process. Thus, modified cobalt oxide ($\text{SO}_4^{2-}/\text{Co}_3\text{O}_4$) is an effective catalyst for alcoholysis. The yield of bis-2-hydroxyethylene terephthalate is 76%. [20]. Bis (2-hydroxyethyl terephthalate) was synthesized in 72% yield [21]. To achieve 100% conversion of HPET, selective catalysts were used, including ($\text{SO}_4^{2-}/\text{ZnO}$, $\text{SO}_4^{2-}/\text{TiO}_2$ and $\text{SO}_4^{2-}/\text{ZnO-TiO}_2$ [22]. In the case of using solid acid catalysts ($\text{SO}_4^{2-}/\text{ZnO}$, $\text{SO}_4^{2-}/\text{TiO}_2$), bis (2-hydroxyethyl terephthalate) was obtained in high yield.

In alcoholysis of SPET, zinc acetate, sodium carbonate, sodium bicarbonate, sodium sulfate and potassium urea were used as catalysts. A comparative analysis of the effect of catalysts on the course of the process has been made [23].

When studying the effect of the time of alcoholysis with ethylene glycol, it was found that an increase in the duration of alcoholysis up to 10 hours leads to an increase in the yield of final products. It was found that alcoholysis products contain $\geq 75\%$ of bis (2-hydroxyethyl) terephthalate monomer in crystalline form [24].

Investigations [25] were carried out on the alcoholysis of SPET EG, on the study of the composition of the reaction mixture - the products of alcoholysis. Fractionation of alcoholysis products PET (PAPET) isolated three fractions: the first is unreacted SPET, the second is the dimer and oligomers, by-products, reaction by-products (EG and others), and the third is BHET. The optimal conditions for alcoholysis were determined: temperature 96°C, mass ratio SPET:EG = 1:3, concentration of zinc acetate catalyst 0.2 wt.%, Reaction time 1 hour, which made it possible to obtain BHET with a yield of 81.8% [26]. The resulting BHET and dimers have a melting point of 113 and 172°C, respectively (DSC). IR spectroscopy showed that primary PET has absorption bands - C = O at 1709 cm^{-1} , ester groups (-C-O-) at 1271 and 1097 cm^{-1} , an aromatic ring at 1504, 872, 729 cm^{-1} . The IR spectra of BHET and the dimer differ from the spectra of primary PET by the presence of an absorption band of hydroxyl groups at 3446 cm^{-1} , and the intensity of the peak in the case of fraction III (BHET) is greater than that of fraction II (dimers). The results of IR spectroscopy were supplemented by PMR and ^{13}C NMR spectroscopy. According to the results of the studies carried out, it was shown that fraction III is BHET, fraction II is dimers.

Subsequently, primary PET was synthesized from fractions III and II, as well as primary PET from TPA and EG at a molar ratio of 1:1,3. The structure of the synthesized PET was studied by PMR spectroscopy, DSC, TGA analysis. It was shown that the structure of the synthesized PET contains signals of protons $-\text{CH}_2\text{-CO}-$ at 4,90 ppm, $-\text{CH}_2\text{-O-CH}_2-$ in diethylene glycol residues (d 4,27 and d 4,75 ppm), aromatic protons (8,21 ppm).

The composition of the synthesized PET was studied, in particular, the content of diethylene glycol units was determined: in primary PET 3,2% by weight, in PET from BHET – 1,66% by weight, in PET from dimer – 3,91% by weight. The different content of diethylene glycol units in the composition of the synthesized PET determines the different value of the melting point: 249; 254 and 247°C, respectively [27].

The intrinsic viscosity of all synthesized PETs is almost the same, their values are in the range of 0,56-0,58 dl/g.

Noteworthy in the results obtained is the confirmation of the presence in the composition of the synthesized PET of ether bonds formed as a result of the reaction of two fragments of macromolecules with terminal ethylene glycol residues.

At the same time, the authors have not studied in more detail the structure of PET, in particular, the content of amorphous and crystalline phases in its composition.

Based on the above, we carried out alcoholysis of SPET with ethylene glycol with the release of BHET [28]. The main physical and chemical properties of BHET have been determined. The degree of purity of BHET was established by parallel determination of the same indicators of BHET obtained by its counter synthesis from DMT and EG (Table 1).

Table 1
Basic physical and chemical properties of bis-2-hydroxyethylene terephthalate synthesized by various methods

BHET is derived from:	Melting point, °C	Molecular mass*	Hydroxyl group content, %
DMT and EG	109-110	250/254	13,5/13,38
SPET и EG	109-111	256/254	13,3/13,38

* numerator - experimentally determined, denominator - theoretically calculated

The data in Table 1. show that, within the experimental error, the physicochemical properties of BHET synthesized from DMT, EG and SPET and EG coincide. This allows us to conclude about a sufficiently high degree of purity of BHET obtained from SPET and recommend it for the synthesis of PET. This conclusion is also confirmed by the satisfactory coincidence of the experimentally determined basic physicochemical properties with the theoretically calculated ones.

The purity of the obtained BHET was also established by the DSC method (Fig. 1). The endothermic peak at 110,15°C is the melting point of BHET; at 262 (beginning) - 280 (end) °C, a peak is observed that characterizes the melting of crystals with higher molecular weight than BHET, residues of the alcoholysis products of SPET [29].

An exothermic peak at 210°C is characteristic of the BHET crystallization process. The discrepancy between the melting and crystallization temperatures and the discrepancy between the areas of the corresponding peaks indicates incongruent melting.

This is apparently due to the presence of higher molecular weight homologues in BHET. Thus, the obtained BHET is a fairly pure product containing minor impurities of its higher molecular weight homologues.

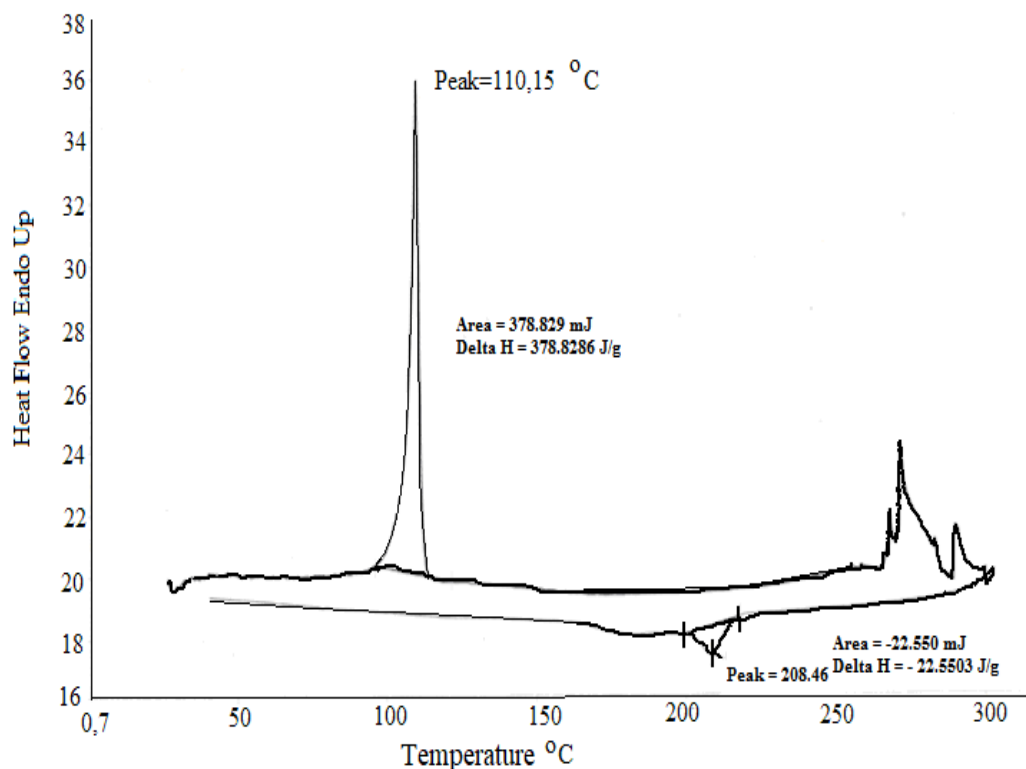


Figure 1. DSC curves of BHET obtained from SPET

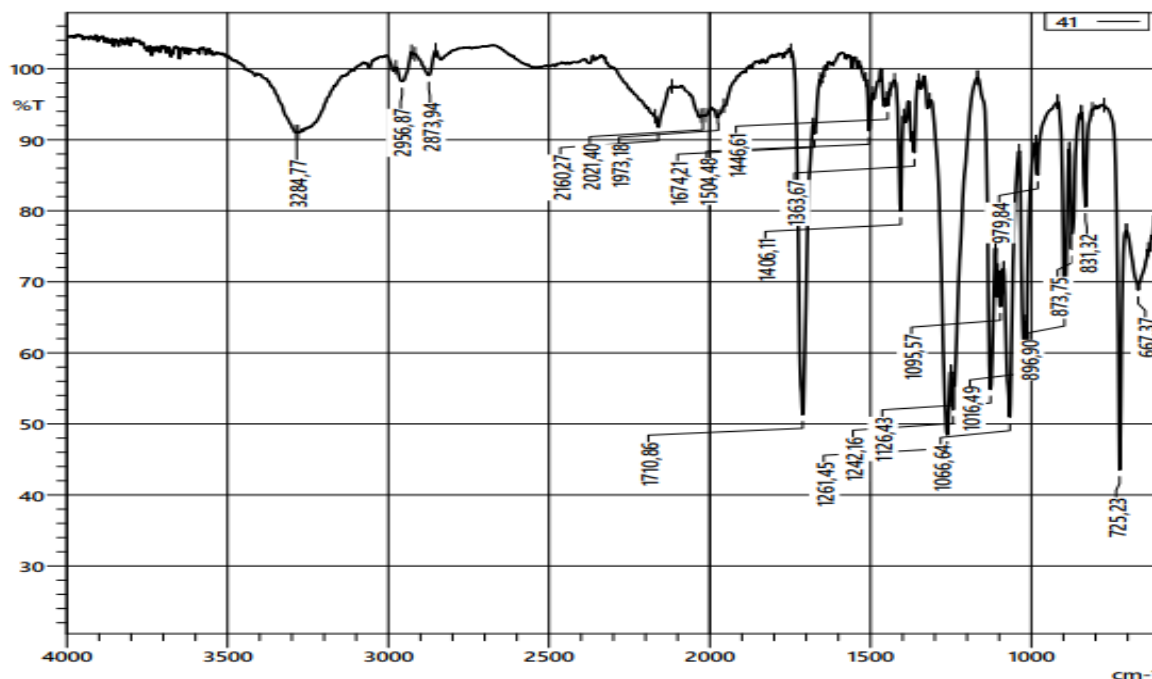


Figure 2. IR - spectrum of BHET obtained from SPET in the presence of zinc acetate catalyst

The purity of the obtained BHET was also confirmed by IR spectroscopy. The IR spectra obtained from SPET BHET have absorption bands of associated primary hydroxyl groups at 3284, 1016 cm^{-1} , methylene groups at 2957, 2874, 1446, 725 cm^{-1} , carbonyl groups in the ester group at

1711 cm^{-1} , 2,4 - substituted aromatic groups at 1580, 1504 and 896, 831 cm^{-1} , “acetate band” of ester groups at 1261 - 1126 cm^{-1} [30].

The above results allow us to conclude that BHET is sufficiently pure and suitable for use as a monomer.

Further research was carried out in the direction of the synthesis of primary PET from BHET. The synthesis was carried out according to the method described in [31]. At the same time, the physicochemical properties of primary PET used in the production of containers for bottling beverages were determined, secondary PET that was in use, PET was synthesized from DMT and EG. The research results are summarized in table. 2.

Table 2

Physicochemical properties of PET

№	Polyethylene terephthalate	Melting point, °C	Viscosity average molecular weight
1.	Primary used in the production of PET preforms	250-260	33000-35000
2.	Recycled PET in the form of flakes	220-230	19000-20000
3.	PET synthesized from BHET based on DMT and EG	240-249	24000-26000
4.	PET synthesized from BHET obtained by alcoholysis of SPET	234-240	22000-24000

Table data 2. show that the primary PET used in the production of "eggplant" has the highest average molecular weight, processing and operation leads to a decrease in the average molecular weight as a result of destructive processes (secondary PET), the synthesis in laboratory conditions of PET from industrial raw materials and BHET, obtained from SPET allows to obtain a primary polymer with comparative physicochemical properties.

The DSC curves of PET synthesized from DMT have one endothermic peak at 249.4°C and one exothermic peak at 202°C (Fig. 3a).

The endopic characterizes the PET melting process, the exopic characterizes crystallization upon cooling the melt. It should be noted that the peaks of melting and crystallization do not coincide and their areas are not equal.

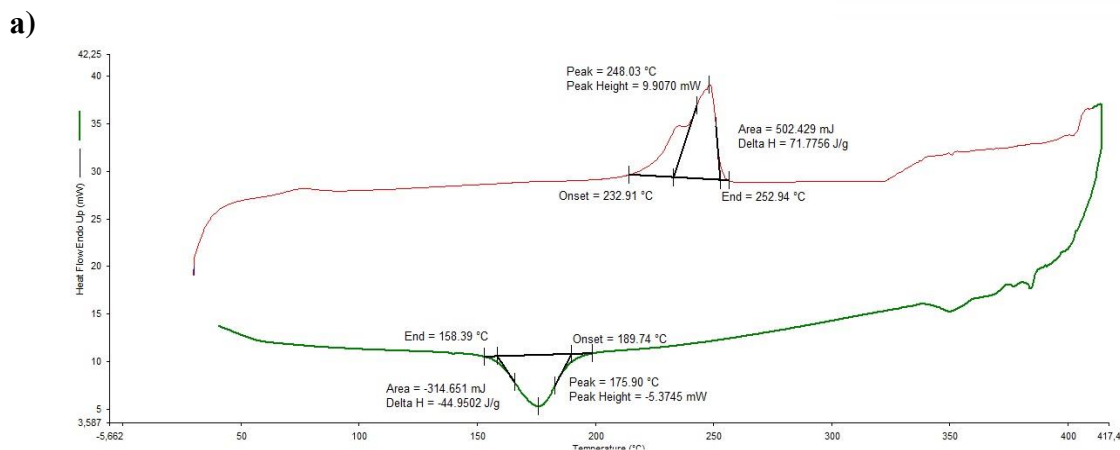
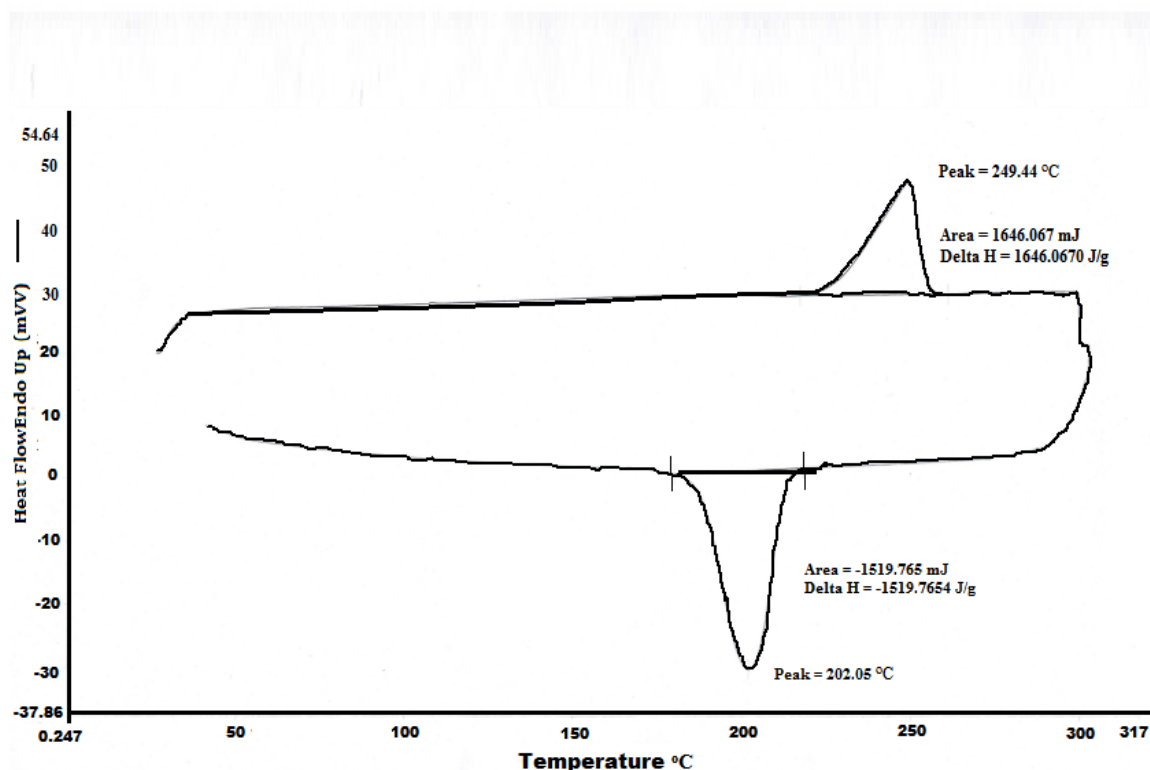
This allows us to conclude about incongruent melting, i.e., upon melting and subsequent cooling, the PET structure changes; most likely, a more ordered structure is formed. In contrast, PET synthesized from BHET at 230°C has a solid-solid phase transition and then melts at 249°C. In the process of cooling the melt at 200°C, the polymer crystallizes.

As in the first case, the melting of PET based on BHET is incongruent (Fig. 3b). The foregoing indicates that PET synthesized from BHET based on SPET has a more complex phase composition, which causes solid-solid transitions associated with structural reorganization that occur with heat absorption.

This, apparently, is due to the persistence of the arrangement of macromolecules, which then melt as a whole [32]. This assumption is confirmed by the absence of such an anomaly in the case of PET synthesized from DMT, as well as primary PET used in production.

A more detailed study of the structure of the synthesized PET was made possible by IR-spectroscopy (Fig. 4). The IR spectra of PET synthesized on the basis of BHET (Fig.4a) have

absorption bands at 1720 cm^{-1} of carbonyl groups in the ester groups, 1570, 1510, 1480 cm^{-1} of the aromatic ring, 1250, 1120 acetate bands in the ester groups, 730 cm^{-1} methylene groups. The absorption band at 870 cm^{-1} indicates that the aromatic ring has substituents at the 1,4 - positions [24]. The presence of absorption bands at 1420, 1100, 1020, 890 cm^{-1} - for gauche isomers.



b)

**Figure. 3. DSC PET synthesized in the laboratory from. a) DMT and EG;
 b) BHET derived from SPET**

This allows us to conclude about the predominantly amorphous structure of the synthesized PET from BHET. The IR spectra of the synthesized PET based on DMT and EG contain absorption bands of all characteristic of polyethylene terephthalate (Fig. 4b). Distinctive is the presence of absorption bands at 1480, 1350, 1110, 980, 850 cm^{-1} characteristic of trans-isomers of $\text{OCH}_2\text{CH}_2\text{O}$ groups in the elementary units of macromolecules. At the same time, absorption bands at 1350, 1020 and 980 cm^{-1} are characteristic of partially crystalline PET [33]. This allows us to conclude about the partially crystalline structure of the synthesized PET from DMT and EG. It was the

amorphous structure that predetermined the presence of a roof phase transition on DSC at 230°C (Fig. 3b).

Our results are in good agreement with the data of [34] in terms of obtaining BHET. The main difference from this work, according to our data, is that the melting point of synthesized PET is 249° C, and melting is preceded by a solid - solid phase transition. Moreover, the value of the melting point lies in the range of those for the primary, used in the production of PET. We also managed to synthesize PET from BHET with a higher molecular weight under laboratory conditions (Table 3).

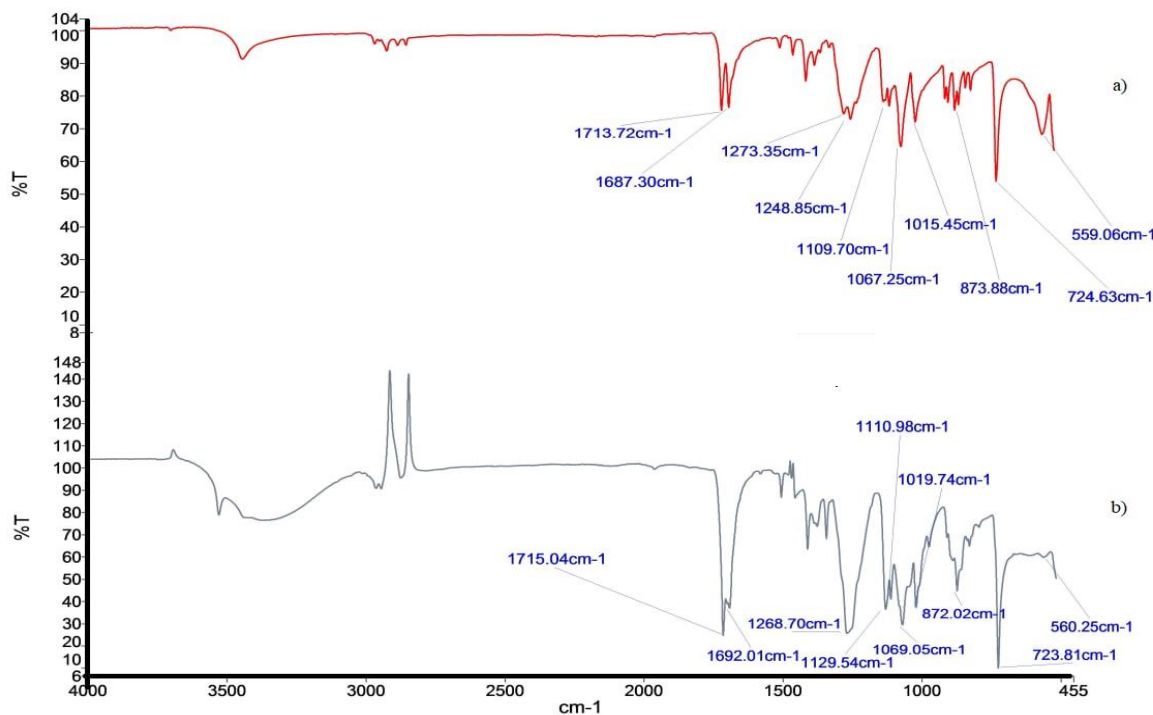


Figure 4. IR spectra of synthesized PET based on BHET (a) and DMT (b)

From the data table 4. it follows that primary PET has the highest molecular weight, is partially crystalline; after processing and operation, the molecular weight of PET decreases due to destructive processes, and the degree of crystallinity of the polymer also decreases. Laboratory-synthesized PET from DMT and EG is also partially crystalline; PET from BHET - amorphous.

Table 4

Properties of synthesized and industrial PET

NAME	Characteristic viscosity, [η], dl/g	Average viscosity molecular mass	Density, kg/m ³
Primary PET (used in the production of "eggplants")	0,91	35000	1394
Flexa - secondary PET	0,63	19952	1248
Synthesized in the lab. PET conditions from DMTP and EG	0,73	23865	1384
Synthesized in the lab. PET from BGTP	0,68	21896	1278

Conclusion. The results of these studies have shown the fundamental possibility of obtaining fiber-forming PET from household waste; made it possible to determine the optimal

conditions for alcoholysis of PET EG and the synthesis of fiber-forming PET from BHET - a product of alcoholysis of secondary polyethylene terephthalate.

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