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IMPROVING THE CONDITIONS FOR THE SYNTHESIS OF PRIMARY POLYETHYLENE TEREPHTHALATE FROM BIS-(2-) HYDROXYETHYLENE TEREPHTHALATE

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Abstract: This article.....

Keywords: alcoholysis product, bishydroxyethylene terephthalate, synthesis, polyethylene terephthalate, physical-chemical and technological properties, IR spectrum, DSC, NMR spectrum, technological scheme,

Introduction

Currently, polyethylene terephthalate (PET) is widely used in the production of products, materials, and parts in many industries due to its advantages and technological properties, and ranks 2nd in production volume among polymer materials after polyethylene, with production reaching 70 million tons per year.

According to "PCI Consulting Group" data, out of 65 million tons of PET produced in 2012, 44 million tons were used for fiber (68%), 18 million tons (28%) for bottles, and 3 million tons for film production.

Currently, if we consider all natural and synthetic fibers as 100%, the share of polyester fibers based on PET is 74%. At the same time, a significant increase in their production and processing has been observed in some regions. For example, in China, Mexico, and Asian countries, the growth rate has reached 9%. We can also observe confirmation of these indicators in major enterprises in Asian countries. For instance, Reliance Industries (India) produces 2.5 million tons annually, Indorama (Indonesia, Thailand) produces 2.0 million tons, and Zhejiang Tongkun (China) produces 1.4 million tons of in-demand PET [1,2].

Based on the above information, the continued annual growth in polyester fiber production provides further justification for our work in the direction of PET production and processing.

In Uzbekistan, approximately 56 thousand tons of PET are used annually for packaging (bottles, flasks) plastic containers. About 10 enterprises in our republic are processing PET bottle waste. One of them is PET Recycling Group, which is engaged in the production of granules from secondary PET flakes and synthetic fibers from these granules [3].

In recent years, numerous studies have been conducted in various countries on the alcoholysis of secondary polyethylene terephthalate (SPET) to obtain bishydroxyethylene terephthalate (BHET) [4-8].

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In our previous studies, we examined the conditions for obtaining BHET by alcoholysis of SPET, and the influence of factors (reaction duration, catalyst amount and type, SPET:EG ratios) on the yield of BHET [9].

Taking into account the above information, since 2000, the Department of "High Molecular Compounds and Plastics Technology" named after T.R. Abdurashidov at the Tashkent Institute of Chemical Technology has been conducting research on alcoholysis of SPET with glycols and polyhydric alcohols, and using the resulting hydroxyl-containing oligomers to produce various grades of unsaturated polyesters [10-12], hydroxyl-containing polyesters for A-component of rigid and elastic polyurethane foams [13-14], alkyd oligomers [15], and various polymer compositions based on them [16-19].

We have also been researching the production of bishydroxyethylene terephthalate for several years [9].

This article is dedicated to studying the properties of BHET obtained by alcoholysis of SPET, as well as BHET obtained from initial raw materials, namely dimethyl terephthalate and ethylene glycol (EG), and primary polyethylene terephthalate synthesized from them in laboratory conditions.

Experimental part

The following substances were used in the research: secondary polyethylene terephthalate (SPET) =1.38-1.40; ethylene glycol according to SS 19710-83, =1.1171 g/cm3; dimethyl terephthalate according to SS 11363-91 =1.21 g/cm3; zinc acetate according to SS 5823-78, =1.735 g/cm3.

1.Synthesis of BHET from secondary polyethylene terephthalate was obtained using the method described in [our own] literature.

2. Synthesis of BHET based on dimethyl terephthalate and ethylene glycol. In a four-necked flask equipped with an inert nitrogen gas tube, stirrer, 2 thermometers, a reflux condenser, and a device for collecting the released methanol, the process was carried out with a DMTF:EG=1:2.5 mol/mol ratio and 0.5% zinc acetate (relative to RPET). The process was conducted in two stages: in the 1st stage, the reaction mass was maintained at 180-190°C for 3-6 hours (until 85-90% of methyl alcohol was released); in the 2nd stage, the excess ethylene glycol was maintained at 260-280°C. After that, the heating of the reaction mass was stopped and cooled. The resulting product was collected in a container.

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3. Obtaining polyethylene terephthalate from synthesized BHET. In a two-necked flask equipped with an inert nitrogen gas tube, adapter, 2 thermometers, reflux condenser, and vacuum pump, the synthesized BHET was placed and in the 1st stage kept at 240°C, under 0.97 kgf/cm2 excess pressure for 40 minutes; in the 2nd stage at 280°C, under 0.97 kgf/cm2 excess pressure until the final ethylene glycol was released (1-1.5 hours), after which the reaction was stopped.

The physical and chemical properties of the resulting PET were determined using the following methods: hydroxyl group according to SS 25261-82, average molecular weight SS 10028 [6], melting temperature SS TC 25-11-398-69. IR spectra of RPET, BHET, and PET were determined using "FT Ir spectrum Perkin Elmer" brand.

Analysis of the obtained results

Table 1 shows the main properties of BHET obtained from dimethyl terephthalate and ethylene glycol, as well as from alcoholysis of RPET with ethylene glycol.

Table 1. Main properties of bis-hydroxy-ethylene terephthalates obtained from various raw materials

BHET synthesized	Melting point, °C	Average molecular weight *	Hydroxy-group, %
Based on DMTF and EG	109-110	243/254	13,5/13,38
IPET and subjected to alcoholysis with EG	109-111	256/254	13,3/13,38

* In the numerator, what is found in practice/in the denominator, what is found in theory

The data shown in Table 1 indicates that the melting point of BHETs obtained by both methods corresponds to the melting point of BHET obtained by other researchers [7]. The average molecular mass of the synthesized BHETs determined by the cryoscopic method is slightly smaller than the theoretical molecular mass (theoretical calculation - 254), equal to 243. The determined hydroxyl group content of this BHET is 14.1%, which is 0.72% higher than the theoretical calculation. These values indicate that there is a small amount of free EG present in the BHET synthesized from the raw materials.

At the same time, it should be noted that the BHET obtained by alcoholysis of IPET matches all theoretical calculated indicators, taking into account experimental errors. Thus, a rather pure BHET (without impurities) was successfully synthesized through alcoholysis.

As can be seen from Table 2, it was determined that the properties of BHET synthesized from traditional and secondary PET correspond to each other.
The BHET obtained from IPET was analyzed by the DSC method (Figure 1.)

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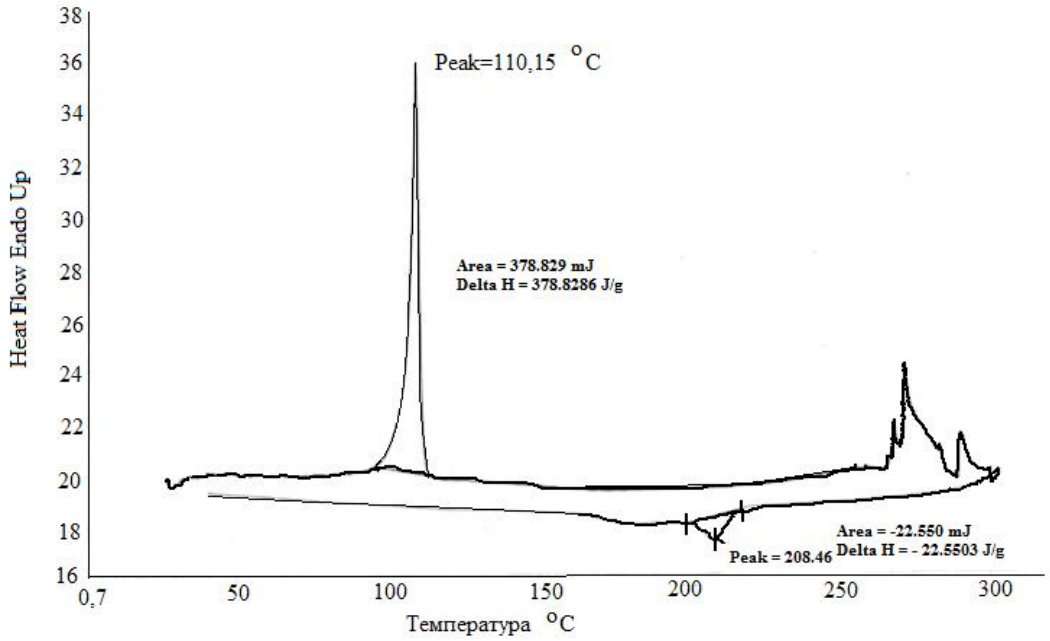

1-Figure. DSC result of bishydroxyethylene terephthalate obtained from secondary PET

DSC results show that the melting temperature of BHET obtained from IPET corresponds to the melting temperature of BHET (108-110°C) reported in the literature [7,8]. The very narrow DSC peak indicates, as mentioned above, the purity of the synthesized BHET (very low impurity content).

The structure of the synthesized BHET was also studied using IR spectroscopy (Figure 2).

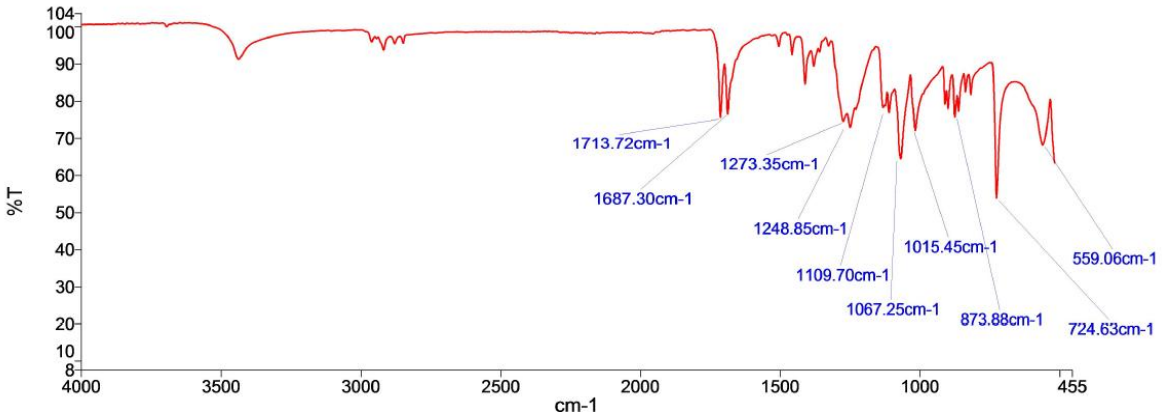

Figure 2. IR spectrum of bis-hydroxy-ethylene terephthalate obtained from secondary PET

Table 2

IR spectrum transmission of BHET

Wavelength, cm-1	Transmission
BHET based on PET	
3440	OH
2850-2930	-CH_2
1713	C=O, carbonyl

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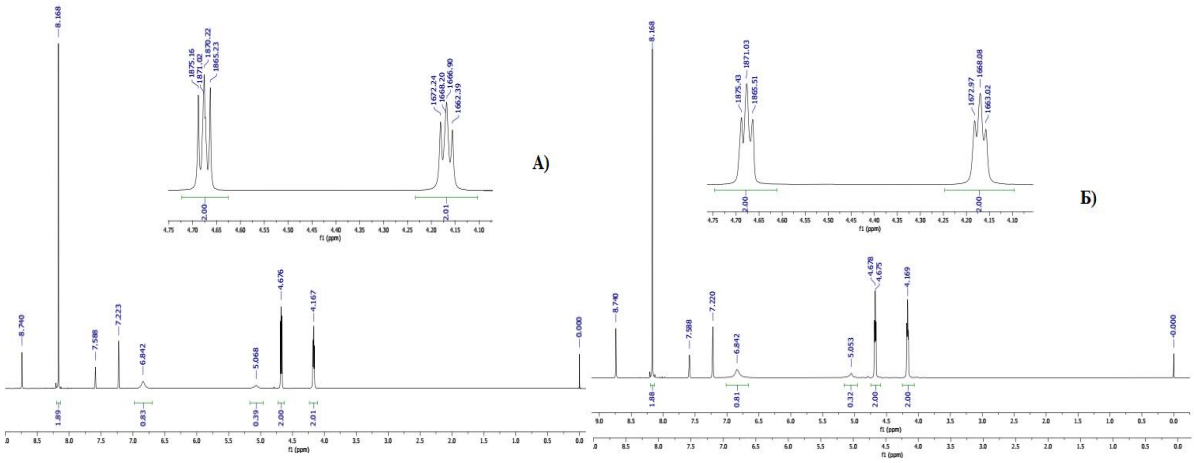
1506, 1410	Ar-CO-
1273	C-O-
1109	1,4-Ar-
1067	C-O-C-
1015	OH primary
873, 724	1,4-Ar-
724	Ar-C-H

In the IR spectrum table [20], we can see the presence of transmission lines characteristic of bis-hydroxy-ethylene terephthalate. The IR spectrum wavelength of the BHET we synthesized also matched the analysis results reported in [4] literature.

The DSC and IR spectral analysis results complemented each other, indicating a high degree of product purity.

From the IR spectra, we can observe the correspondence between the formed terephthalic acid diester. The DSC melting temperature being relatively high indicates the presence of high molecular weight segments in the product. Subsequently, we studied the structural composition of synthesized bis-(2-hydroxyethylene) terephthalate using nuclear magnetic resonance (NMR).

The NMR spectrum of bis-(2-hydroxyethylene) terephthalate is presented in Figure 3.17.



3.17- NMR Spectrum of Bis-(2-hydroxyethylene)terephthalates a) Based on IPET; b) Based on DMTF.

NMR spectrum shows signals 1, 2, 3, and 4 corresponding to aromatic ring protons (dH=8.11 ppm, s, 4H), hydroxyl groups (dH= 4.96 ppm, t, 2H), methylene groups (-CH2–) linked to -OH groups (dH= 3.53 ppm, m, 4H), methylene groups (-CH2–) linked to -COO groups (dH= 4.33 ppm, t, 4H). From this, it can be concluded that the NMR spectra of secondary polyethylene terephthalate and BHET synthesized based on DMT are quite similar.

In our next study, we achieved the synthesis of polyethylene terephthalate based on alcoholysis products of BHET and studied their physicochemical properties. The reaction equations for these

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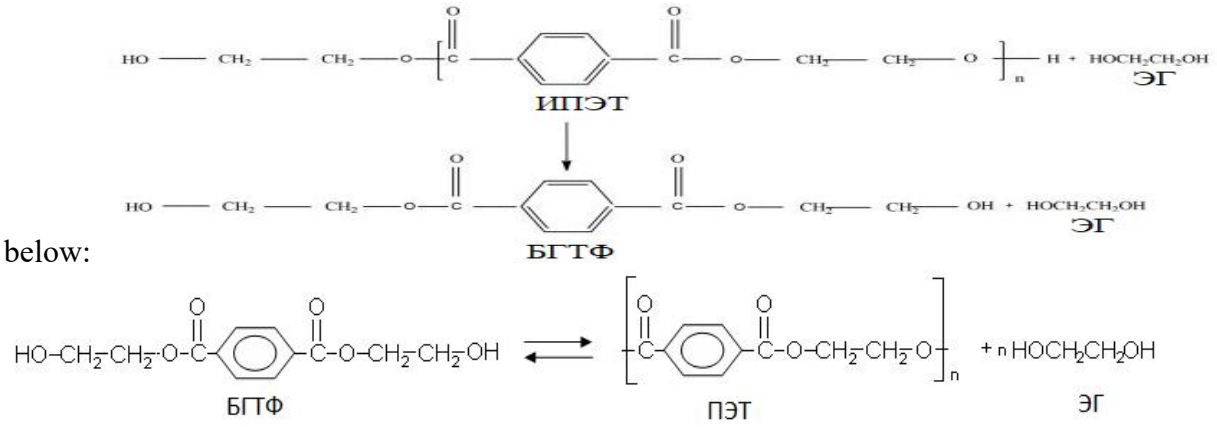
processes are presented below:



Based on the BHET obtained, primary PET synthesis was carried out using the method and technique described on page 101 of the literature [6]. The obtained results were compared with the properties of BHET and polyethylene terephthalate obtained in industry. The obtained results are presented in Table 3.

Table 3

Properties of Obtained BHET and PET Synthesized on Their Basis

№	Name	Melting point, °C	Average molecular weight
1.	Primary PET (packaging bottle production)	250-260	33000/35000
2.	Bottle flakes (flexes) - secondary PET	220-230	19000/20000
3.	PET obtained from BHET synthesized in laboratory conditions from dimethyl terephthalate and ethylene glycol	240-249	24000/26000
4.	PET obtained from IPET by alcoholysis in laboratory conditions from BHET	234-240	22000/24000

The data presented in Table 3 shows that the PET obtained on the basis of DMTF is close to the properties of primary polyethylene terephthalate in terms of melting temperature and average molecular weight. In the PET obtained on the basis of IPET, these indicators are somewhat lower. Such a difference indicates the presence of partially low molecular weight substances in the PET obtained on the basis of IPET.

The DSC results of each obtained sample were analyzed (Figure 4).

a)

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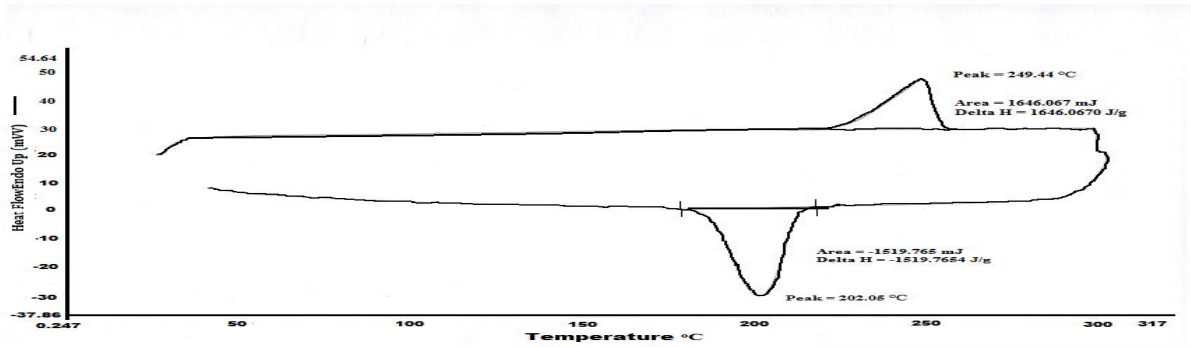

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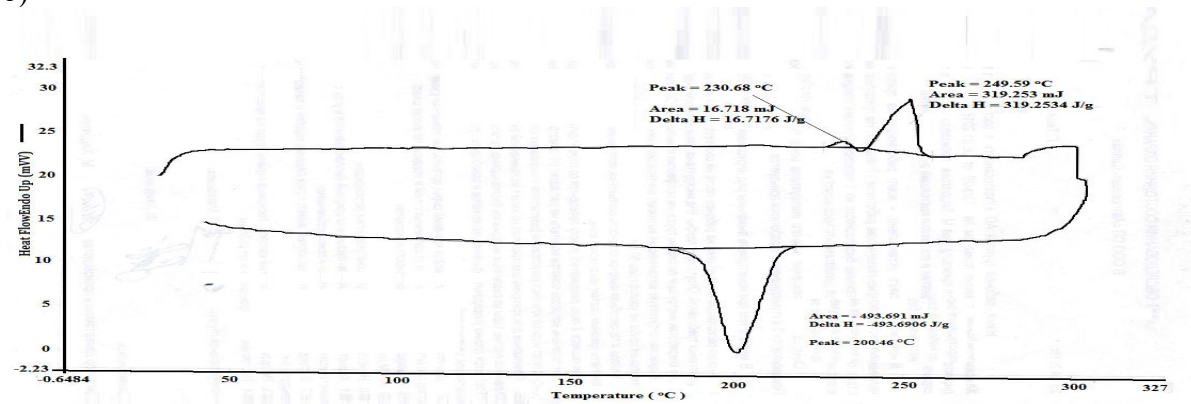
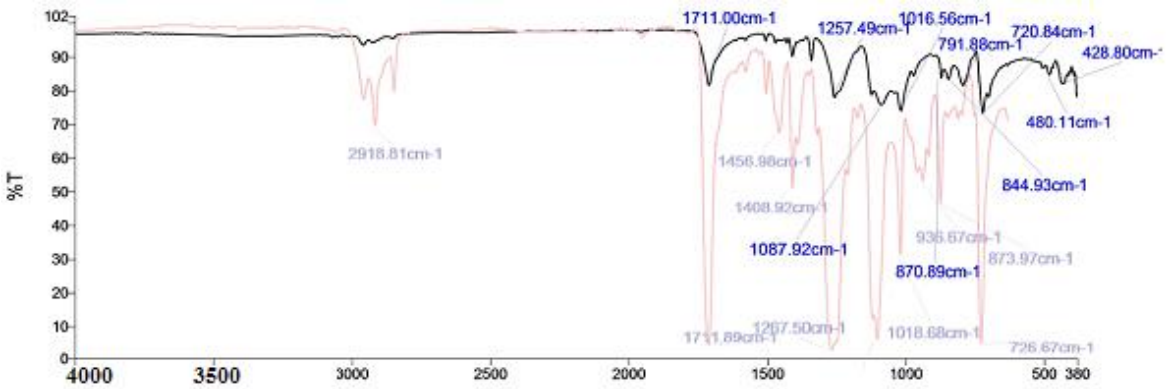


Figure 3. PET IR Spectra. a) PET based on DMTF; b) PET based on BHET

According to Figure 3 (a), the DSC of PET obtained from DMTF shows crystallization at 200-202°C with an endothermic absorption at 249°C, indicating the sample's melting and revealing a stable crystalline structure; (b) shows the PET synthesized from BHET crystallizing at 200°C and having a melting temperature with a small interval at 230°C and a wide interval at 249°C. We can observe the sample's melting at 230°C and 249°C. This indicates the presence of macromolecules of various sizes and partial crystals in its composition. Modern and laboratory-determined indicators showed mutual correspondence and complementarity.

Their IR spectra also recorded the following results.

a)



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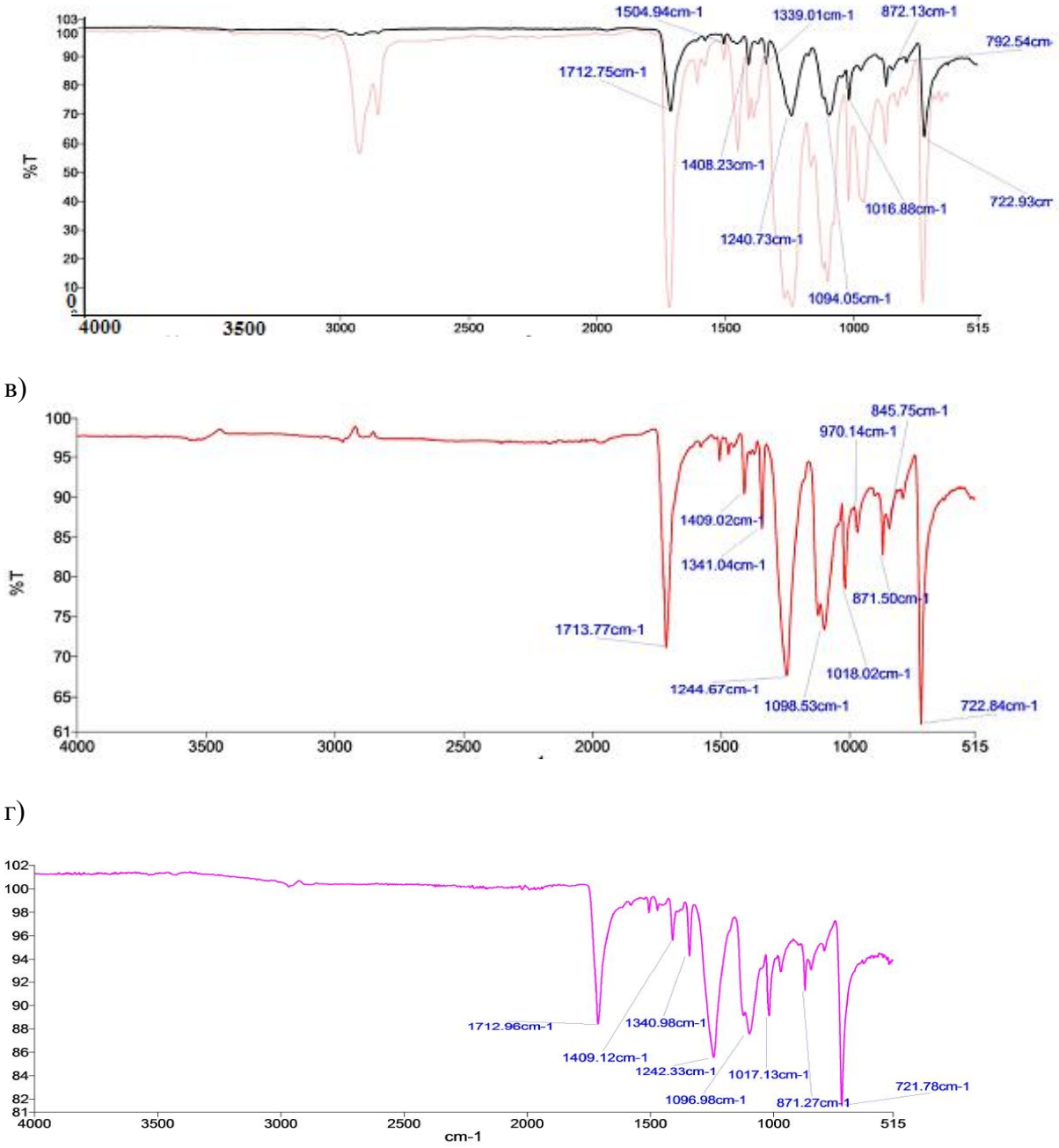

Figure 4. PET IR Spectra. a) Primary PET; b) Secondary PET; c) PET based on BHET; d) PET based on DMTF

The absorption lines in the IR spectrum in Figure 4, especially the reflections of 1711-1713, 1408-1410, 1340-1344, 1016-1018, 871-873, 720-723 transition lines, precisely indicate that primary PET was synthesized [20].

The characteristic viscosity of the synthesized samples was determined based on the Mark-Houwink equations cited in the literature [21]. The determined indicators are presented in Table 4.

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Table 4

Technological Properties of Synthesized PET and Industrial Samples

Name	Indicators		
	Characteristic viscosity , [η]	AVERAGE MOLECULAR WEIGHT	Density, г/см^3
Primary PET (intended for packaging bottle production)	0,91	35000	1.394
Bottle fragments (flexa) - secondary PET	0,63	19952	1.248
PET obtained from BHET synthesized in laboratory conditions from dimethyl terephthalate and ethylene glycol	0,73	23865	1.284
PET obtained from BHET produced by alcoholysis of IPET under laboratory conditions	0,68	21896	1.278

From Table 4, we can see the relationship between characteristic viscosity and average molecular weight. For example, when comparing PET based on DMTF and IPET, the characteristic viscosity was found to be correspondingly 0.73 and 0.68, and we can observe that the average molecular weight was around 20-23 thousand.

Thus, it was determined that alcoholysis of secondary polyethylene terephthalate with ethylene glycol produces bis-hydroxyethylene terephthalate and its dimers. It was found that the catalyst used and the duration of alcoholysis directly affect the formation and yield of BHET, and the primary PET obtained from them was compared with PET obtained by traditional methods. The technological properties of the synthesized PET were determined, and the results were confirmed by their accuracy and reproducibility.

A technology for producing fiber-forming polyethylene terephthalate based on secondary polyethylene terephthalate and ethylene glycol has been developed. The distinctive feature of this technology is that excess EG produced during alcoholysis and fiber-forming polyethylene terephthalate production is purified and returned to the reaction medium. This accounts for 50-60% of the initial EG. Fiber-forming polyethylene terephthalate was produced at a temperature of 280°C and a pressure of 0.1 kg/. The fiber-forming polyethylene terephthalate was obtained almost entirely from local raw materials.

The technological scheme for producing bis-(2-hydroxyethylene) terephthalate (BHET) from secondary polyethylene terephthalate is shown in Figure 4.1.
To obtain bis-(2-hydroxyethylene) terephthalate (BHET) (Figure 4.1), the required amounts of secondary PET, EG, and catalyst are measured from dosers 1, 2, 3 and poured into the reactor.

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The reaction is carried out in reactor (5-8) at a temperature of 190-195°C for 4-8 hours while being stirred with mixer (4-9). The EG condensed during the reaction is returned to the reaction medium through cooler 6. Then, the resulting alcoholysis product is transferred to the second reactor 8 via pump 7, 10 for separation, where the alcoholysis product is kept with four times as much distilled water at 90-95°C for 25-30 minutes and separated into fractions through filter 11, 13 (1st fraction). The aqueous part that passes through the filter is filtered again at 13 at a temperature of 0-2°C in cooler 12 (2nd fraction). The remaining part is separated into water and EG through distillator 14.

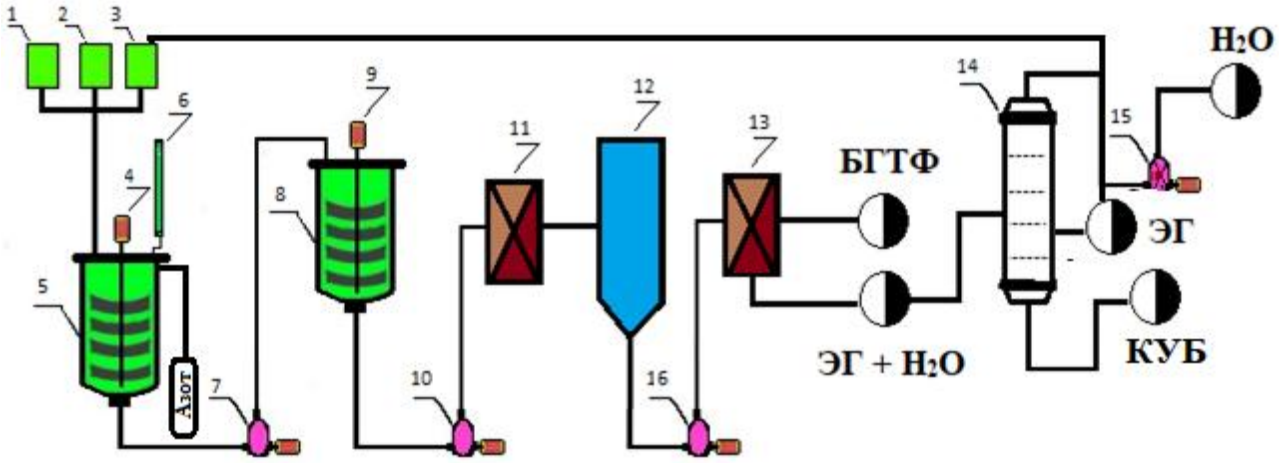

Figure 4.1. Technological scheme for the production of bis-(2-hydroxyethylene) terephthalate (BHET) from secondary polyethylene terephthalate.

1, 2, 3–dosing containers; 4, 9–electric motor; 5, 8–reactor; 6- cooler; 7, 10–pump; 11, 13-filter; 12- cold water; 14- distiller; 15–vacuum pump.

The technology for producing fiber-forming Polyethylene terephthalate is shown in Figure 4.2.

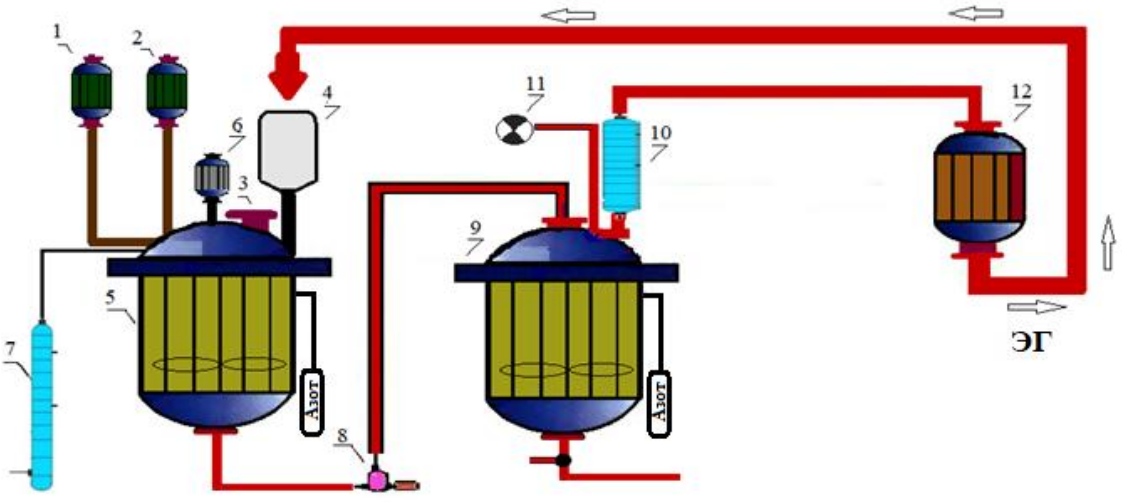

Figure 4.2. Technology for producing polyethylene terephthalate that forms fiber.

1, 2, 4–dosing containers; 3-hatch 5, 9–reactor; 6–electric motor; 7, 10-cooler; 8–pump; 11–vacuum pump; 12–containers.

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The primary polyethylene terephthalate is obtained as follows:

First method – in this (Figure 4.2) the obtained BHET was placed in reactor 9. The synthesis process was carried out in an inert environment, under vacuum 11 (-1kgf/cm2) for 1-2 hours at a temperature of 270-280 0C.

Second method – in this method, from dosers 1,2,4, secondary PET and EG along with catalyst are measured in required amounts and poured into the reactor. In reactor (5) at a temperature of 195±2 0C for 4 hours, the reaction was carried out in an inert environment 7-10 while being continuously mixed with mixer (6). After the synthesis process was completed, it was transferred to reactor 9 through pump 8, and here the synthesis process was carried out under vacuum 11 for 1-2 hours at a temperature of 270-280 0C. This process is a polycondensation process where excess EG is released and collected in container 12 to be returned to the reaction medium.

In our future research, we will focus on studying the conditions for obtaining fiber based on the synthesized primary PET and improving it.

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