Chapter 28

ECOLOGICAL AND ECONOMICAL ASPECTS OF COMPOSITION MATERIALS CREATION

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Pesticides are considered to be the most dangerous of all chemical compounds which are received with air, water and food by human organism. In 1988 the USA. National Academy of Sciences published a report which noticed that more than one million Americans risk to fall ill with cancer caused by twenty-eight cancerogen pesticides in food. The abuse of pesticides can provoke a burst of cancer diseases and mutations in developing countries. According to the World Health Services Organisation data 500,000 people are poisoned by pesticides and die yearly.

Chlororganic pesticides are widely used in agriculture (against vermin), forestry, veterinary and medicine. These compounds are characterized by two very important properties. Firstly, it is firmness to environment factor influence such as temperature, solar radiation, moisture. Secondly, they are expressed cumulative properties. All this caused a situation that firm chlororganic pesticides are found almost in all living organisms and their concentration in tissue and organs is high than in the environment.

DDT (dichlor diphenil trichloroethane, dichlordiphenil trichlormethyl-methane) is a chlororganic pesticide. It is a white crystal substance without taste and smell. In 1956 its world production was 80,000 tons. From 1942 till 1974 4,5 million tons of DDT were spent for agricultural vermin destruction. However, the World Health Services Organization forbade using that preparation because of its toxic influence on human organism through "food chain", consisting of plants, animals used by human being.

Despite the taken measures, the problem of influence of a given compound on environment and human organism is still vital. Firstly, DDT is firm to decomposition (is stands heating up to 115-120°C for 15 hours and doesn't decay at cooking) and can circulate in biosphere more than fifty years. Also, it is absorbed easily in deposits and soils which can be a depot for DDT and its derivatives. Such depots are the sources of chronic influence. Secondly, this given preparation is still used in developing countries. Thirdly, the DDT

spreading has a global character. It can be transported in migrating animals organisms, and with air and ocean streams too. That is why the DDT influence on environment is much wider than its using region (for instance, on Antarctic Continent, very far from using zones more than 2000 tons of DDT were accumulated in glaciers).

The problems of ecological safety became aggravated. It is caused by unstable social and economical conditions in Russia. In particular, the problems connected with processing and destroying of unused pesticides are solved very slowly and uneffectively. By the first of January 1998 11,75 tons of pesticides and more than 2,000 tons of DDT had been accumulated in the warehouses of the Republic of Adygea. The lack of utilization ranges forced the farms of the Republic to spents means on safe keeping of these preparations. The need in warehouses for long duration keeping of pesticides was satisfied only at 52% but the quantity of unused chemical preparations increases every year.

Thus it turned out that the situation is neither ecologically not economically profitable. It is a result of the unsufficiently considered agricultural activity.

One of the ways of solving this problem is the elaboration of the processing methods for unused and worthless pesticides.

We have worked out the DDT utilization method. It is the creating of composition materials. The method of synthesis of dichlorbenzophenon (dichlordiphenilketone) from DDT (that is alkaline dehydrochlorination of dichlordiphenil trichlorethane in ethanol with following oxidation by chromic anhydride in icy vinegar acid. On the basis of the obtained dichlorbenzophenon and diphenylolpropane as well as dichlorbenzophenon and phenolphtalane by the method of high-temperature polycondensation in surroundings of dimethylsulphoxide in nitrogen atmosphere the oligoketones of dian and phenoiphtalane rows with degrees of condensation 1.5.10.20. The synthesis of oligoketones was made at mole surplus of diphenylolpropane or phenolphtalane to dichlordiphenilketone according to the following scheme:

The structure of obtained oligoketones was confirmed by IR-spectroscopy and definition of hydroxide group quantity. The availability of absorption stripes in IR-spectrums corresponding to simple ether joints in domain 1135cant⁻¹, to isopropyliden group at dian surplus 2960 - 2980cant⁻¹ (in the case of dian oligorners), to lactone group 1710 - 1760cant⁻¹ (in the case of phenolphtalane oligomers), hydroxide group 3300 - 3600cant⁻¹ and keto-group 1600 167Scant⁻¹ testifies of oligoketones formation.

Some properties of oligoketones are cited in Table 1.

Synthesized oligoketones were researched as HOPE modifiers. To evaluating the effectiveness of putting the oligomers into the HDPE melt, their 0,1% (at mass) concentration was studied.

The experimental plant (with the help of which all physics and mechanic properties of compositions on the basis of HDPE and aromatic oligomers) represents a pendulum setting UT-1/4 which is supplied with sensor of loading. Its signal was transmitted directly to memory oscillograph, model C 8-12.

It was found out that oligoketones on the basis of diphenylolpropane, independently from condensation degree, influence on HDPE as plasticizers. We can make a conclusion on these facts that there is some increasing of relative deformation during the destroying of models. But at the transition to phenolphthalein oligoketones there is an essential distinction, that is the polymer models become harder (the modulus of elasticity increases). Both the shock viscosity and the limit of forced stretchiness stay at high level which exceeds a little these parameters

for primary polyethilene. Apparently, it is connected with harder structure of the phenolphtalane in compare with dian. Practically all researched oligoketones increase cristallinity degree of the polyethilene. Probably, getting into amorphous part of the polymer can become the "embryo" of crystallization. During multiple (five times) extruding the character of oligoketones on physical and mechanic properties of high-density polyethilene doesn't change.

Oligoketones	Output, %	T _{softing} , K	M.M.	OH-group content, %	
				Calculated	Found
OK-1D*	98	402-408	634,78	5,36	5,30
OK-5D	98	420-425	2260,72	1,50	1,55
OK-10D	99	433-438 4293,17	4293,17	0,79	0,75
OK-20D	99	440-448	8358,43	0,41	0,40
OK-1F**	98	469-473	814,85	4,17	4,20
OK-5F	98	483-488	2800,94	1,21	1,20
OK-10F	99	510-517	5283,75	0,64	0,65
OK-20F	99	528-533	10248,77	0,33	0,60

Table 1. The properties of aromatic oligoketones

D*-oligoketones on the basis of diphenylolpropane with condensation degrees 1-20. F** - oligoketones on the basis of phenolphthalein with condensation degrees 1-20

Besides physic and mechanic properties of obtained compositions we have researched such characteristics as thermostability, melt index and molecular-mass distribution, chemical firmness, dielectric properties.

Complex study of aromatic oligoketones' influence on properties of high density polyethilene allows to recommend them as quite perspective modificators of HDPE."

Also, we have to notice, that the represented oligoketones in the capacity of additive to highdensity polyethilene can be used as fbrpolymers for the synthesis of high-molecular compounds of aromatic polyetherketones class which are very perspective materials of construction purpose with higher physics and chemical characteristics.

Thus, the results of our researches show, that with direct utilization of DDT we san obtain new perspective composition materials. This method permits to solve a very vital ecological and economical problem of processing of unused chlororganic pesticides.

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Chapter 29

POLYARYLATE OXIMATES (PAO), THEIR PHYSICOCHEMICAL PROPERTIES AND STABILIZING INFLUENCE ON POLYALKYLENE TEREPHTHALATE (PAT)

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ABSTRACT

Physical and chemical properties of polyarylate oximates (PAO) synthesized by catalyticacceptor polyesterification from terephthalic acid anhydrides and diacetylphenyloxid are submitted. The modification of polybutylene terephthalates (PBT) by PAO in amount of 0,5-1 % wt. increases its thermal stability and heat resistant.

Key words: polyarylates; polyesters; antioxidants; thermal stability; polybutylene terephthalate.

INTRODUCTION

Polyarylates represent a rather perspective class of polymers, which can be successfully applied in many fields of polymeric technology required a use of materials with high heat-resistance, good dielectric and mechanical properties [1]. In industrial scale polyarylates are produced on the basis of various diphenols [2].

EXPERIMENTAL PART

Polymer Preparation

Polyarylate oximates (PAO) were prepared from various diketoximes (DKO) and dichloride anhydrides of *iso*-phthalic (CAIP) and terephthalic acids (CAT). Synthesis of polyarylate oximates was carried out by low temperature catalytic-acceptor polycondensation, with triethylamine as catalyst (Fig.1)

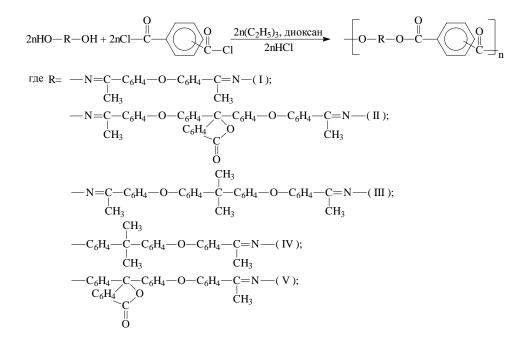


Fig. 1. Schematic representation of polyarylate oximates synthesis

The full-scale test were performed to find the influence of reaction time, solvent nature, temperature, and reagents feed ration on intrinsic viscosity of polyarylate oximates to find out the optimal reaction conditions for their preparation in accordance with the Scheme 1. It was found that synthesis of polyarylate oximates can be carried out more efficiently in dioxane at 303 K during 40 min. with triethylamine as catalyst and molar ratio DKO/ (CAT) / (DCAIP) = 2/1/1. The reduced specific viscosity η_{red} for a resulting polyarylate oximates from phenyl ketoximes in acetone as a solvent proceeded as a heterogeneous process and the resulting polymer precipitated from a solution.

Chemical structure of prepared polyarylate oximates was supported by elemental analyasis data and by IR-spectroscopy (Table 1). A group of bands in the region of $(1735 - 1750 \text{ cm}^{-1})$ and the absence of absorption at $3300 - 3600 \text{ cm}^{-1}$ belonging to hydroxyl groups is an argument in favor of polyarylate oximate structure formation.

Polymer	η_{red} tetrachloroethylene/phenol	Elemental analysis data *		
rorymen		С,%	Н,%	N,%
PAO-I	0.75	64,73	3,96	13,45
rao-i	0,75	65,98	4,18	14,07
PAO -II	0,81	73,59	4,15	3,57
	0,81	74,05	4,28	4,18
PAO -III	0,71	75,01	5,00	4,39
TAO -III		75,17	5,29	4,83
PAO -IV	0,68	76,85	5,03	2,81
		77,02	5,18	2,90
PAO -V	0,80	73,43	4,14	2,31
	0,00	73,68	4,21	2,46

 Table 1. Reduced viscosity and elemental analysis

 data for prepared polyarylate oximates

* Numbers: numerator – found; denominator – calculated.

METHODS AND INVESTIGATION

A number of physical methods were used for to characterize properties of the prepared polymers. The degree of crystallinity was determined by X-ray diffractometer DRON-6.0 using nickel-filtered radiation CuK_{α} (1.5405 Å). A sample was exposed within the θ angle range from 7 to 45 degrees with preset exposure spacing of 1°/min. and measurement accuracy of 0.030 degree. Chemical stability of polymers was studied on disk-like film samples with diameter 5×10^{-3} m by measuring a change of their weight during exposition according to GOST No 12020-72. Thermomechanical properties were studied by applying UIP-70 device with a constant stress of compression 0.08 MPa. The softening temperature of a polymer was found as a point at which the tangents of two branches of thermo-mechanical curve intersect. The differential scanning calorimetry (DSC) was performed in argon atmosphere by the Mettler TA – 4000 instrument supplied by DSC-30 cell with a heating rate of 20 C°/min. Dielectric strength of the obtained polymers were determined by using the high voltage generator BMW-30-01 as a part of AM-A-02F1 analyzer. Thermo-gravimetric analysis was carried for 25 mg samples in the air by using "MOM "(Hungary) instrument with a heating rate of 5 C°/ min.

RESULTS AND DISCUSSION

The results of X-ray analysis of synthesized polyarylate oximates show the high crystallinity in samples that, perhaps, may explain bad solubility of these polymers in chlorinated solvents (chloroform, dichloroethane, dicholoromethane). The examined samples of PAO showed a good chemical resistance against the influence of aggressive medium such as H_2SO_4 (of 10 % and 30 % concentration), concentrated HCl, and NaOH (of 10 % and 50 % concentration), which was measured according to GOST 12020-72 by measuring a change in

weight of samples (the amount of the extracted substances). The diluted solutions of acids had no actual effect on PAO that was in agreement with the observed absence of significant change in the weight of samples during 24 hours, and small loss in the weight (not exceeding 2 %) after 28 days. At the same time PAO displayed less stability in alkaline solutions. In the concentrated sulfuric acid samples of all synthesized polymers were dissolved after 1 day.

The character of thermomechanical curve of PAO showed that the tested samples revealed a rather rigid structure with high glass and viscous flow temperatures. The values of glass and viscous flow temperatures from thermomechanical data (Fig. 2) were found to be around 393 K and 468 K, respectively.

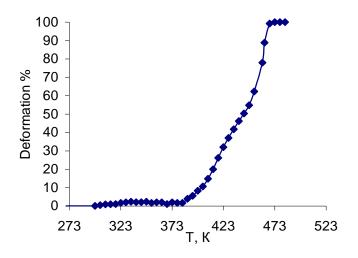


Fig. 2. Thermomechanical curve for polyarylate oximate PAO-1

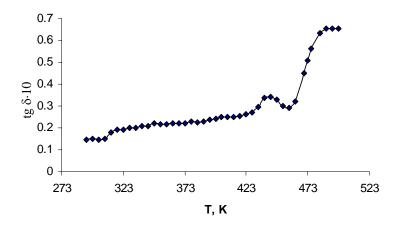


Fig. 3. A plot of loss-angle tangent (tg\delta) versus temperature for PAO-1

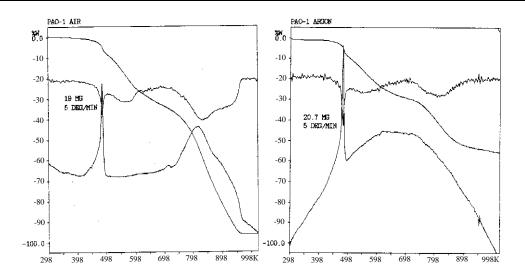


Fig. 4. The data of thermal analysis (TG, DTA, TMG) of PAO-I in argon and in the air

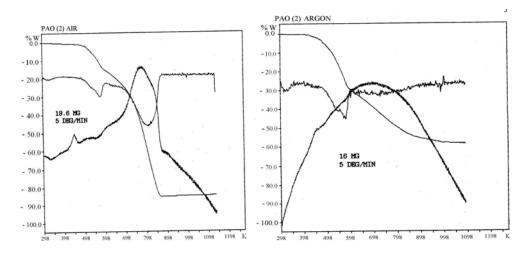


Fig. 5. The data of thermo analysis (TG, DTA, TMG) of PAO- V in argon and in the air

DSC analysis was used for glass temperature and melting point of PAO determination and presented values were in a good agreement with those obtained by thermomechanical and dielectrical methods. It is essential to note, that the regions of structural transitions predicted by increment modeling coincide within the limits of 5 % with DSC and thermomechanical analysis data.

The performed tests of dielectric strength using the high voltage generator BMW-30-01 at room temperature showed that PAO did not revealed electric conductivity even at high electric potential of about 3000 volt, i.e. PAO is a good dielectric.

Dielectric measurements of PAO-I demonstrated that this polymer reveals one dipolesegmental relaxation transition within the temperature range of 430 - 470K (Fig. 3), the nature of which is under research. Besides, PAO-I has rather high temperature of through conductivity around 470 K. The thermal analysis showed that polyarylate oximates were of good thermal stability. It is evident from TGA curve for PAO-I that charcoal residue at 998 K in the air was about 5 % compared to 48 % in argon (Fig. 4). The charcoal residue for PAO -V containing card-like group was about 15 % in the air, whereas in argon this values came to 43 % (Fig. 5).

CONCLUSIONS

The results of the physical and chemical studies point out that polyarylate oximates obtained in this work revealed stability in aggressive media, good dielectric characteristics, and high thermal stability. These results enable us to recommend the use of synthesized polymers in manufacturing of industrial products for various purposes.

The performed earlier studies on modification of PBT by polyformal oximates with the purpose of improvement of its operational properties brought us to positive results[3]. Polyarylate oximates and polyformal oximates belong to a class of polyesters and, therefore, one should expect a positive effect from their use as modifying additives for polyakylene terephthalates. The preliminary experiments afforded positive results.

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Chapter 30

THERMOSTABLE POLYBUTYLENE TEREPHTHALATE (PBT) MODIFIED WITH POLYFORMAL OXIMATES (PFO)

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ABSTRACT

The physical and chemical properties of polybutylene terephthalte (PBT) modified with polyformal oximates (PFO) on the basis of di-acetophenyloxid dioxime were investigated by thermogravimetric analysis (TGA), melt flow index (MFI), and differential scanning calorimetry (DSC). It was shown that the addition of 0,5-1% PFO by weight to PBT increased the initial temperature of thermal-oxidative degratation by 25-65°C, and it was possible to change the melt flow index (MFI) to the values convenient for processing.

Key words: Polyformal oximates; polyethers; antioxidants; thermal stability; polybutylene terephthalate.

INTRODUCTION

It is known that, the polybutylene terephthalate (PBT) is one of the perspective and universal thermoplastic polymers belonging to polyesters. It is produced industrially in wide scale and has different applications as a constructional material. The growing world wide

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production of PBT and of various products from it requires improving of its physicalchemical properties [1].

Earlier, we have synthesized polyformal oximates (PFO) on the basis of diacetophenyloxid dioxime and methylene cloride. The aromatic polyether has possessed a new conjunction of chemical fragments in a polymeric chain due to a structure of the initial monomers [2].

The results of the physical and chemical studies revealed that polyformal oximates was stable against the influence of the aggressive medium, exhibited high thermal stability and good dielectric characteristics. Because of the chemical structure of PFO, it was of interest to study its possible use as a modifier in PBT blends with the purpose of increasing its thermal stability during the processing, improving of the operational qualities, and enlarging the exploitation resource of articles [3].

EXPERIMENTAL PART

Preparation of PBT Blends

Samples were obtained by extruding the mixtures of granulated PBT (grade B-305) and appropriate amount of 1 % solution of polyformal oximates in chloroform, which was initially dried up under vacuum at temperature 100°C within two hours. The mixture was then extruded at temperature 100°C at rotational speed of 50-70 rpm. The obtained polybutylene terephthalate samples contained 0,05-1% PFO-1.

Instrumental Methods

Physical properties of the prepared samples were investigated by various analytical methods. Thermal behavior was analyzed under air by TGA instrument "MOM" (Hungary), the heating rate of samples was kept at 5 °C/min, the weight of a sample was 25 mg. The melt flow index (MFI), which determines the processing method for thermoplastics, was measured by the standard method of GOST 11645-73. For the estimation of MFI values the instrument IIRT-M2 was used. Dielectric properties of the obtained samples were investigated by the method of dielectric losses. Electric measurements were carried out with the help of the bridge by applying alternating current of 10^3 Hz and digital readout R-5058 in a temperature interval from 20 to 250°C. The error in measurements of a loss-angle tangent did not exceed 5%.

RESULTS AND DISCUSSION

Results of our study showed that physical and chemical properties (thermo-stability, electric strength) of polybutylene terephthalate samples containing PFO as modifying additive in a wide temperature range considerably exceeded the properties of the known samples.

Figures 1 and 2 represent the thermal analysis data for PFO and PBT modified by PFO, as well as for non- stabilized PBT and PBT industrially stabilized.

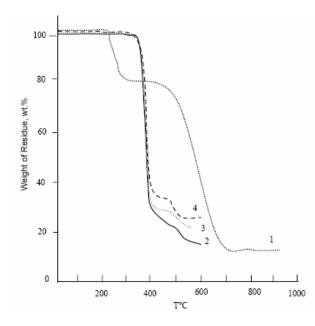


Fig. 1. TG analysis of various PBT/(PFO) blends with PFO content: (a) 0.5w % (curves 2, 3,) and (b) 1w % (curve 4). Curve 1 is reffered to PFO

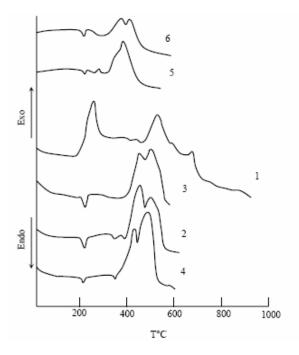


Fig. 2. Differential thermalal analysis (DTA) for non-stabilized (curve 5) and stabilized industrial PBT (curve 6) and for various PBT/PFO blends with PFO content: (a) 0.5w % (curves 2, 3); (b) 1w % (curve 4). Curve 1 is referred to PFO

It is evident from TGA curves (Fig.1) that the increase of the weight fraction of PFO-1 brings about the enlargement in mass fraction of the charcoal residue. Curve 3 is referred to a sample, which was exposed to preliminary thermal aging for 30 minutes at $T = 250^{\circ}$ C. The increase in the PFO-1 content displays a dual effect: simultaneously with the accelleration of thermal degradation the structural reorganization in samples takes place that is good agreement with the character of TG curves.

DTA data (Fig. 2 curves 5, 6) discloses that oxidation of non-stabilized and stabilized industrial PBT begins at 260 and 325°C, respectively. For PBT samples containing 0,5 wt % of PFO-1 (after 30 minute exposure at 250°C, curve 3) and 1 % wt. (curve 4), the oxidation process occurs at 350°C. The DTA curves of the samples shows active thermo-oxidative degradation with two peaks, the area and position of which depends on the PFO-1 contents (Fig.2).

DTA curves 5 and 6 (Fig. 2) show the peak corresponding to thermo-oxidation degradation with a bend at 462 - 466°C. The addition of the PFO-1 in the composite changes the character of DTA and DTG degradation and crosslinking becomes the basic process (curves 2-4). If PBT composite contains 0,5 % PFO-1 (without thermo-ageing) then no difference in character of DTA curves for PBT (non stabilized) and samples PBT + PFO-1 in region of 460-470°C is observed. For a composite of PBT with 0,5 % PFO-1, which was held at 250°C for 30 minutes, the essential stabilizing effects of PFO-1 were found to be (the first peak corresponding to oxidation processes was reduced, while the second peak corresponding to crosslinking was increased).

In this respect, the best results were received for PBT samples with PFO-1 content of about 1 %. It can be seen from curve 4 (Fig. 2) that oxidation does not occur up to 390°C and crosslinking of composite happens in the range of 460-470°C. The first peak responsible for thermal-oxidative degradation dissapears almost completetely.

The melt flow index (MFI) was measured at 230°C and 2,16 kg for a PBT composite containing PFO-1 as a modifier and had a tendency to decrease up to 2,4 times (Fig. 3), depending on its content. Most likely, this is caused by increase in molecular weight of a polymer due to the chemical interaction between PBT and PFO-1 molecules at this temperature.

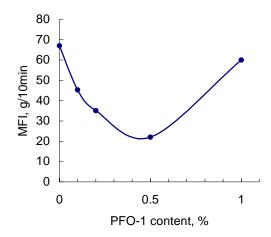


Fig. 3. A plot of melt flow index versus PFO-1 content in blends with PBT

A complex of positive effect resulting from the addition of PFO-1 in small quantities (~ up to 1. % wt) to PBT such as increasing of the coke residue and temperatures of the beginning thermal-oxidative degradation, and also the possibility to change MFI up to value convenient for PBT processing, PFO-1 can be used as a chemical modifier in a blends with PBT for the purpose of improving of its operational properties.

Addition of PFO-1 to a polybutylene terephthalate blends improves the dielectric properties, as well. This was proved by the results obtained from the dielectric analysis carried out on the blends of PBT +0.5% PFO-1 and PBT +1% PFO -1 (Fig. 4, Diagram 1).

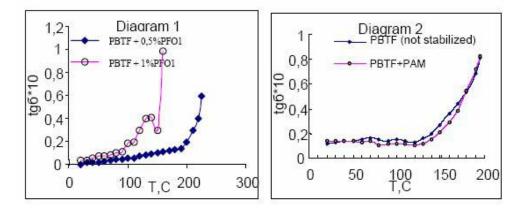


Fig. 4. A plot of loss-angle tangent versus temperature for PBT blends with PFO-1 (Diagram 1) and non-stabilized and stabilized with PAM PBT (Diagram 2)

The data on dielectric properties of industrial (non stabilized) PBT and PBT stabilized with PAM are submitted on the Diagram 2 (Fig. 4). It is evident from these data that molecular mobility in blends of PBT + 0,5% PFO -1 and PBT + 1% PFO-1 is a slightly higher than that for the industrial (not stabilized) PBT and compositions PBT/PAM in the range temperatures below glass temperature. Besides, a temperature of through conductivity is somewhat higher. Thus, for industrial non-stabilized PBT it is equal to 125° C, for PBT/polyazomethin – is 135° C, whereas for PBT +0,5% PFO-1 this temperature raises up to 190° C, and for PBT +1% PFO -1 mixture is 160° C.

CONCLUSIONS

A complex of positive effects of polyformal oximates (PFO) on PBT properties such as the increase of coke residue, of melting temperature, of the initiall temperatures of crosslinking and degradation was discovered. The change of a melt flow index up to the values convenient for PBT processing can be also reached. PFO in amount up to 1 % by weight can be used as a modifying additive to PBT for incraeasing its operational characteristics, e.g., the practical operational temperature and for broadening the temperature interval of processing.

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