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# ИСПОЛЬЗОВАНИЕ ПРОДУКТОВ ХИМИЧЕСКОЙ ПЕРЕРАБОТКИ ПОЛИЭТИЛЕНТЕРЕФТАЛАТА ДЛЯ МОДИФИКАЦИИ БИТУМОВ

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***Полиэтилентерефталат (ПЭТФ) – это один из наиболее широко используемых пластиков в мире, для которого успешно освоены методы механической и химической повторной переработки. В последние годы продукты химической переработки ПЭТФ привлекают внимание в качестве потенциальных добавок в строительные материалы, включая асфальтовые смеси и бетон. Целью данного исследования является разработка подхода к вторичной химической переработке отходов ПЭТФ для получения продуктов с добавленной стоимостью, которые могут быть использованы для модификации битумов. Для достижения этой цели отходы ПЭТФ подвергали реакции аминолиза с последующим получением олигоэфирамидов, которые были охарактеризованы методами ИК-спектроскопии и вискозиметрии. Полученные продукты использовали для модификации битума марки БНД 60/90. Результаты испытаний модифицированных битумов (по ГОСТ 33137-2014, ГОСТ 11506-73, ГОСТ 18180-72, ГОСТ 12801-98) показали, что добавление олигоэфирамидов, может повысить устойчивость к образованию колеи, расширить температурный интервал эксплуатации и адгезию битума к минеральному наполнителю. Установлено, что введение олигоэфирамидов не вызывает существенного возрастания динамической вязкости битума (при температуре 135 °C), что позволит избежать технологических сложностей в процессе перекачки битумов; обеспечивает стойкость битума к тепловому старению на уровне не ниже исходного. Показано, что с увеличением молекулярной массы олигоэфирамида и его дозировок сдвиговая устойчивость и адгезия битума к минеральному наполнителю увеличиваются, при этом наилучшее сцепление наблюдается с мрамором. Таким образом, рациональное использование олигоэфирамидов, полученных из отходов ПЭТФ, может являться перспективным направлением решения экологических проблем загрязнения и открывает возможность для получения продуктов с высокой добавленной стоимостью.***

**Ключевые слова:** полиэтилентерефталат, аминолиз, олигоэфирамид, модификация битума, адгезия

# USING POLYETHYLENE TEREPHTHALATE CHEMICAL PROCESSING PRODUCTS FOR BITUMEN MODIFICATION

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***Polyethylene terephthalate (PET) is one of the most widely used plastics in the world, whose mechanical and chemical recycling has been successfully mastered. Recently, PET chemical recicling products have attracted attention as potential additives to construction materials, including***

***asphalt mixtures and concrete. The purpose of this research is to develop a way to chemically recycle PET waste to produce value-added products that can be used for bitumen modification. To achieve this goal, PET waste was subjected to an aminolysis reaction. The obtained terephthalic acid diamide underwent polycondensation in the melt obtaining oligoesteramide at different time of reaction. The structure of obtained products was confirmed through IR spectroscopy and viscosymetry. Bitumen BND 60/90 was modified by obtained oligoesteramides. The properties of the modified bitumens (per GOST 33137-2014, GOST 11506-73, GOST 18180-72, GOST 12801-98) demonstrated that the addition of oligoesteramides can increase ruting resistance, can expand the temperature operational range, and bitumen adhesion to minerals. Authors have established that the introduction of oligoesteramides does not cause any significant increase in the dynamic viscosity of bitumen (at 135 °C), which will help avoid technological difficulties when pumping bitumen. Also modified bitumenes have the resistance to thermal aging not lower than the base bitumen. It has been demonstrated that with an increase in the molecular weight of oligoesteramide and its content, both the shear resistance and adhesion of bitumen to the minerals increase, while the best adhesion is observed with crushed marble aggregate. Thus, the rational use of oligoesteramides prodused from PET waste be a promising way to solve environmental pollution problems, and opens up the possibility of obtaining products with high added value.***

**Keywords:** polyethylene terephthalate, aminolysis, oligoesteramide, bitumen modification, adhesion

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## INTRODUCTION

Bitumen and bitumen-derived materials are the key components for the modern road construction industry. Improving the properties of bitumen is one of the priority areas to enhance the quality of road-construction asphalt-concrete mixtures. In most cases, the improvement of the bituminous binder properties means an increase temperature resistance, rutting resistance, and better adhesion properties, which is carried out by adding modifiers. Traditionally, various polymers [1-10] use as bitumen modifiers. The increase in polymer modifier's production volumes, as well as, in general, the growth in the production of polymer materials results in increased waste [11-13]. Therefore, the issue of polymer recycling into new products, and, in particular, the use of recycled polymers as bitumen modifiers is becoming relevant.

The most mastered technologies are recycling of polyolefines and polyethylene terephthalate [14-15]. There are studies demonstrating the possibility of successful use of mechanically recycled polymers, for example polyolefines for bitumen modification [16-22].

Mechanically recycled PET have also been studied as potential additives to bitumen [23-26]. However, high temperatures are required for the homogeneous distribution of PET in bitumen (for wet process) (the PET melting point is 245-265 °C [27], which entail premature aging of the bitumen binder, and can also complicate the technological process (as the flash point of bitumen is in 220-240 °C range [1])). Futhermore, the use of mechanically recycled PET in bitumen mostly assumes dry process – PET flakes are added to the asphalt mixture directly during the mixing of the bituminous binder and the filler as its partial substitute [28-29].

Moreover, the characteristics of bitumen binder with the mechanically recycled thermoplastics always depend on the quality and nature of the used polymer waste, which is difficult to control [30]. Products (additives) from chemical recicling polymers lack such drawbacks. Such products are various low-molecular compounds having different functional groups in their structure. The most mastered method of PET chemical recicling is aminolytic destruction when PET wastes interact with various amines and amino alcohols producing low-molecular and oligomeric compounds [31-33]. Such monomers and oligomers can be further used as raw materials for the production of new products. The functional groups in their structure suggest the possibility of using chemical recycling PET products as an adhesive additive to bitumen (the higher polarity of products than polarity of bitumen can increase the adhesion to the polarity filler) [34-36].

This research is aimed at obtain and study of using chemically recycled PET waste as bitumen modifiers.

## METHODOLOGY

Preparation of the intermediate (N,N'-bis(2-hydroxyethyl) terephthaldiamide) was carried out according to the procedure described in [37]. Further on, the obtained terephthalic acid diamide underwent polycondensation in the melt obtaining oligoesteramide (OEA) at different time of reaction – 30, 60, and 90 min. The polycondensation reaction conditions were as follows: temperature 180-200 °C (slow heating), orthophosphoric acid catalyst (laboratory grade grade as per GOST 6552-80, produced by Component-Reactive, LLC), purging with an inert gas (nitrogen) to remove reaction by-products.

The solubility of products were assessed in dimethyl sulfoxide (DMSO) (laboratory grade grade as per TU 2635-114-44493179-08 manufactured by ECOS-1, JSC).

The relative viscosity of DMSO oligomer solutions (0.1 g/ml concentration) were assessed by using a Labtex series VPZh-2m glass viscometer as per GOST 10028.

The IR-spectra of the DMSO-solved OEAs were studiied by the Shimadzu FTIR-8400S device through multiple frustrated total internal reflection.

The sample of bitumen BND 60/90 (GOST 22245-90) was used for modification.

The OEAs (powdered products) were mixed in 1 and 3 wt.% to bitumen heated to a liquid state at 160-180 °C with a paddle stirrer at 270-300 rpm (to obtain modified bitumen). The mixing time for all experiments did not exceed 4 h.

The homogeneity of bitumen mixture was assessed by visual inspection of the on-glass film residue.

To assess the technological properties of unmodified bitumen and modified bitumen, the dynamic viscosity at 135 °C was tested as per GOST 33137-2014 with a REOLOGICA Instruments AB StressTech dynamic shear rheometer (parallel plane system; top plane surface diameter – 20 mm, shear rate – 1.5 $s^{-1}$, gap – 1.0±0.01 mm). The viscoelastic properties of the modified bitumen were assessed by measuring the complex shear modulus (G*) and the phase angle (δ) at various temperatures as per GOST 58400.10-2019 using mentioned reometer (angular frequency – 10±0.1 rad/s, gap – 1.0±0.01 mm).

The softening point of unmodified and modified bitumens were assessed by test as per GOST 11506-73. Then, the properties of aged bitumens were tested per GOST 18180-72 (the warming-up at 160 °C, the warming-up duration – 5 h).

The adhesion of bitumen BND 60/90 to minerals was assessed per GOST 12801-98. The various minerals (crushed limestone and marble aggregates) were used to assess adhesion, since in [38] it had been shown that with an increase in the total content of CaO, MgO, $Fe_2O_3$ and $Al_2O_3$ in mineral materials, their adhesion to bitumen increased, and with an increase in the total content of $SiO_2$, $Na_2O$, $K_2O$, $CO_2$, their adhesion to bitumen decreased.

## RESULTS AND DISCUSSION

The resulting polycondensation products were solid, room-temperature crystallizable compounds of yellow to light brown color.

Oligoesteramides (OEAs) were used to assess their usability as bitumen modifiers. The OEAs are terephthalic acid diamide polycondensation products obtained at different polycondensation time: 30, 60, 90 min, which will be further designated as OEA30, OEA60, OEA90.

The DMSO solubility test demonstrated that OEA30 and OEA60 were completely soluble, while OEA90 only swelled in the selected solvent to form a gel, which might indicate the formation of branched/crosslinked structures at polycondensation. Consequently, OEA90 was further excluded from the experiment.

The formation of branched/crosslinked structures is presumably due to the presence of a mobile (reactive) hydrogen atom within the amide groups of terephthalic acid diamide (Fig.1) [39-40]. The IR spectroscopy proves that with an increase in the polycondensation time, the degree of branching/crosslinking increases.

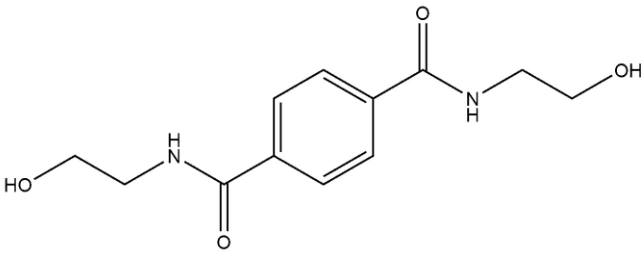


Fig. 1. Structural formula of terephthalic acid diamide
Рис. 1. Структурная формула диамида терефталевой кислоты

The IR spectra of the DMSO-solved OEAs are shown in Fig. 2.

A number of features can be noted in the presented spectra [41]:

- almost complete disappearance of the peak in the 1740-1720 cm$^{-1}$ range corresponding to the valence oscillations of the C=O bond in the PET-molecule ester group, which indicates the success of the chemical destruction;
- the presence of peaks corresponding to amide-I (mainly due to valence bonds of the carbonyl group) of 1660 cm$^{-1}$ and amide-II (due to planar deformation oscillations of the N-H bond in amides) 1540 cm$^{-1}$;
- the presence of peaks in the 3300-3500 cm$^{-1}$ range, corresponding to N-H valence oscillations in the amino group, whose intensity decreases with the increase in polycondensation time; it makes possible to assume the formation of branched/cross-linked structures.

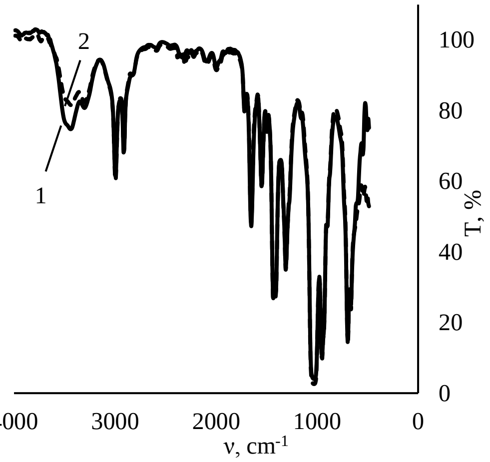


Fig. 2. IR spectra of DMSO-solved OEAs (1 – solution of OEA30; 2 – solution of OEA60)
Рис. 2. ИК спектры растворов олигоэфирамидов в ДМСО (1 – раствор ОЭА30; 2 – раствор ОЭА60)

The relative viscosity for OEA30 and OEA60 solutions was 2.71 and 2.92, respectively, which means a higher molecular weight of OEA60.

The properties of unmodified and modified bitumen containing OEA30 and OEA60 in various content are presented in Table 1.

When OEA30 and OEA60 were mixed with bitumen, there was no significant increase of dynamic viscosity, which is good from a technological point of view (as for example, GOST R 58400.2-2019 limits the upper level for dynamic viscosity at a test temperature of 3 Pa·s). Moreover, the higher the molecular mass and OEA's content, the greater dynamic viscosity is.

The softening point of bitumen (the main performance parameter for bitumen) increased with the introduction of OEAs, which suggests a higher operating temperature of modified bitumens. With an increase of the amount of OEAs in the mixture, an increase in the softening point was also registered. However, no significant difference between the use of OEA30 and OEA60 was noted.

***Table 1***

**Properties of unmodified and modified bitumen containing OEA30 and OEA60 in various content**

***Таблица 1.*** **Сопоставление свойств битумов, содержащих продукт в различных дозировках, с характеристиками немодифицированного битума**

| Parameter | Bitumen BND 60/90 | Bitumen BND 60/90 containing OEA30 | | Bitumen BND 60/90 containing OEA60 | |
|---|---|---|---|---|---|
| | | 1 wt.% | 3 wt.% | 1 wt.% | 3 wt.% |
| Dynamic viscosity (condition 1) at 135 °C, Pa·s | 0.7 | 0.92 | 1.00 | 0.95 | 1.12 |
| Softening point, °C | 45.7 | 51.6 | 53.9 | 51.2 | 53.8 |
| Change in the softening point once heated, °C | 0 | 2.1 | 1 | 0.4 | 0.5 |
| Weight change once heated, % | less than 1 | | | | |
| Homogeneity | homogeneous | | | | |

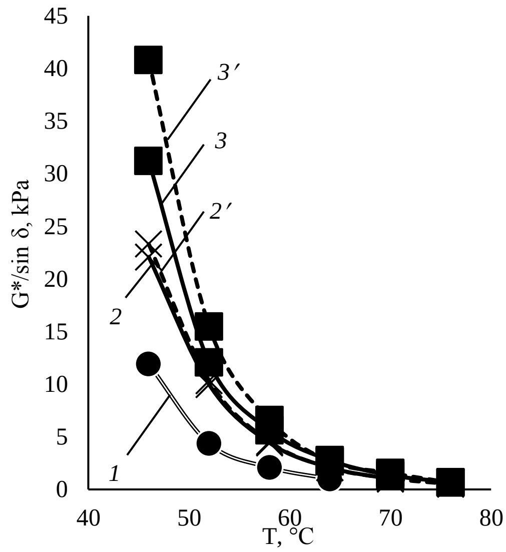


Fig. 3. The rutting parameter (G*/sin δ) of unmodified (1) and bitumen modified by OEA30 (2 – containing 1 wt.%, 2' – containing 3 wt.%) and OEA60 (3 – containing 1 wt.%, 3' – containing 3 wt.%)
Рис. 3. Результаты оценки сдвиговой устойчивости немодифицированного битума (1) и битумов, модифицированных ОЭА 30 (2 – содержание 1 мас.%, 2' – содержание 3 мас.%) и ОЭА60 (3 – содержание 1 мас.%, 3' – содержание 3 мас.%)

As per GOST 32054 – 2013, the change in the softening point should not exceed 5 °C. All modified bitumenes meet this requirement.

*Table 2*

**The results of adhesion of unmodified and modified bitumen containing OEAs to various minerals**

***Таблица 2.*** **Результаты оценки адгезии битума, содержащего исследуемые продукты к минералам различной природы**

| Sample | Example of bitumen film on various minerals | | Adhesion test, point | |
|---|---|---|---|---|
| | crushed limestone aggregate | crushed marble aggregate | crushed limestone aggregate | crushed marble aggregate |
| Uncoated minerals |  |  | - | - |
| Bitumen BND 60/90 |  |  | less than 2 | less than 2 |
| Bitumen BND 60/90 containing 1 wt.% OEA30 |  |  | 2 | 2 |
| Bitumen BND 60/90 containing 3 wt.% OEA30 |  |  | 2 | 3 |
| Bitumen BND 60/90 containing 1 wt.% OEA60 |  |  | 2 | 3 |
| Bitumen BND 60/90 containing 3 wt.% OEA60 |  |  | 3 | 4 |

The rutting parameter (G*/sin δ) of unmodified and modified bitumen containing OEA30 and OEA60 in various content are presented in Fig. 3. It can be clearly seen that all of the modified bitumen have greater G*/sin δ values than the unmodified bitumen, thus indicating that the OEAs successfully enhanced the high-temperature properties of the bitumen. It means that the modified bitumen are less susceptible to the rutting phenomenon.

Clearly, with the increase in the molecular weight of OEA, the shear resistance of the modified bitumen increases. With an increase of OEA content in bitumen mixture, an increase in its shear deformation resistance is noted.

The results of adhesion of unmodified and modified bitumen containing OEAs to various minerals are presented in Table 2. It was confirmed that bitumen adhesion to a minerals depends on the chemical composition of the minerals: the greatest bitumen adhesion in all tests was observed for crushed marble aggregate, in which the total content of calcium and magnesium oxides predominates.

The use of OEAs increases the adhesion to the minerals: the greater the content of the OEA used and the higher its molecular weigh, the greater adhesion is.

## CONCLUSION

The authors have obtained chemically recycled polyethylene terephthalate products, which have further been polycondensed at different reaction times to form OEAs. These OEAs have been used to modify bitumen BND 60/90.

It has been established that the OEAs can be successfully used as an adhesive additive to bitumens: with an increase of the molecular weight of the OEA and its content, the adhesion of bitumen to the minerals increases (the best adhesion is observed to crushed marble aggregate). It has been demonstrated that the introduction of OEAs does not result in any significant increase of the dynamic viscosity of bitumens, while the modified bitumens are resistant to thermal ageing.

With the addittition of OEA to bitumen authors have registered both the bitumen softening point and the ruting parameter increase: the higher molecular weight of the OEA and its content, the higher the temperature class of modified bitumen as per GOST R 58400.3-2019.

Based on this reaserch, we have concluded that OEAs obtained from PET waste can be effectively used as a modifying additive to bitumens.

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*The authors declare the absence a conflict of interest warranting disclosure in this article.*

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