RESEARCH PAPER

Involvement of products of chemical processing of polymer waste in the composition of building materials

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Abstract

The efficiency of the application of chemical processing products of polyethylene terephthalate as modifiers to petroleum bitumen has been confirmed. The possibility of processing sediments formed during a multi-stage technological process by burning them and involving ash in concrete has been established. The comparison of bitumen samples of different grades [Bisphenol A (BPA) 70/100 and 130/200] without modifier, with calcium and magnesium salts of terephthalic acid, with commercial samples based on ethylene copolymer with vinyl acetate, polyethylene with low melting point, low molecular weight polyethylene, etc. With the addition of calcium terephthalate, the softening temperature of BPA 70/100 increased from 50 to 57 °C, BPA 130/200 from 46 to 52 °C, the needle penetration depth increased from 77 to 84, and BPA 130/200 from 137 to 145. The grade of bitumen BPA 130/200 changed with the addition of magnesium terephthalate to BPA 90/130, the grade of BPA 70/100 remained unchanged with the addition of all modifiers except the polyethylene-based modifier. The possibility of improving the physical properties of bitumen by-products of processing polyethylene terephthalate is shown. The possibility of using combustion products of difficult-to-process hydrolysis sludge as an improving additive in concrete is shown. The elementary composition of contaminants on the surface of PET samples showed that the sludge combustion product can be used similarly to fly ash from thermal power plants to improve the properties of concrete, since the silicon modulus of the sludge is quite high — 35%, and the fly ash of thermal power plants — from 30 to 67%.

Keywords: Bitumen modifier, Bitumen, Chemical waste processing, Polyethylene terephthate, Polymer waste, Secondary polymers

1. Introduction

1.1. Relevance of research 'recycling of polymer waste'

Application of polymer waste as a secondary raw material allows the return of valuable polymer raw materials to production and reduces the amount of waste in the environment.[1] The application of natural resources and minerals as primary raw materials is the basis of the industrial economy of most developed countries.[2] Development of economy inevitably leads to an increase in the consumption of manufactured products and the growth of solid household and industrial waste,[3,4] liquid and gaseous industrial,[5,19] other types of waste and leads to an environmental catastrophe.[6,7] Unlike industrial waste, the processing and disposal of polymer waste is a more complex technological task. Methods and standards have been developed for industrial mining waste management,[8] which are regulated by the assessment of maximum permissible concentrations of substances, which facilitates their processing.

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1.2. Overview of existing recycling solutions

There are three main areas of polymer processing: mechanical, thermal, and chemical. The mechanical method accounts for 70–75% of the total volume of recycled waste due to its simplicity and low cost.[1] The thermal method takes 20–21% of the total processing, thermolysis products are used to produce thermal energy and pyrolysis.[9] The chemical method (5%) makes it possible to further obtain polyesters for the production of adhesives, coatings, and raw materials for the re-synthesis of polymers.[10] The chemical method is not as popular as the previous two, however, it gives more competitive target products at the output: various types of additives that allow regulating the physical and mechanical properties of concrete and mortar, bitumen plasticizers, improving additives for asphalt concrete mixtures, etc..[11] To date, the addition of polymer modifiers to bitumen is considered one of the innovative ways to increase their strength, improve deformation properties, and reduce the fluidity of raw materials for the construction of road surfaces.[18]

1.3. Recycling problems and ways of their solutions

The existing methods of recycling polymer waste have several disadvantages: low efficiency of processing contaminated recyclables, the difficulty of implementing a closed-cycle economy due to the lack of a unified integrated approach, and the consistency of technologies used by polymer manufacturers and processors.

1.4. Statement of the problem

This paper considers the problem of disposal of previously accumulated polymer waste, which is contaminated with earth, sand, and their physicochemical properties have been changed due to environmental influences and mechanical aging. Coastal areas are particularly in need of developing technologies for the disposal of old contaminated recycled plastic. Fig. 1 shows photos from the Black Sea coast in the city of Yalta, from where the samples were selected, further described in Section 2.1 'Objects of research'.

This article explores the possibility of processing contaminated polymers (in particular, plastic bottles) from roadsides and from reservoirs without their preliminary cleaning, and not just pure primary polymer. The sediment resulting from these contaminants, after the first stage of hydrolysis, it is proposed to start up for incineration, followed by the involvement of ash in the production of building materials. The cycle closure is achieved by the absence of waste in the technology. In addition, it is planned to study the possibility of reinforcing fiber concrete, polystyrene concrete with a polyethylene terephthalate (PET) hydrolysis precipitate, which in shape and structure corresponds to the properties of PET fiber (PETF). In this paper, the chemical composition of the initial contaminated raw materials and sludge ash after obtaining the modifier is investigated, and the possibility of using the resulting ash with a high silicon module as an additive to concrete is shown. The addition of the resulting ash is possible in proportions regulated by GOST 31108-2016 for fly ash.

Fig. 1. Black Sea coast, Yalta, Crimean Peninsula, Russian Federation. a–d polymer samples taken from the polluted coast.

1.5. Objectives of this research

The purpose of this research is waste-free processing of contaminated, secondary PET to obtain the most expensive and in-demand target products at each stage of the technology. Development of compositions and technologies for obtaining bitumen modifier and geopolymers based on recycled filtration waste.

2. Materials and methods. (Methodology)

All the laboratory and research activities were carried out based on the Scientific Center 'Problems of processing of mineral and technogenic resources' at St. Petersburg Mining University, Russia.

2.1. Research objects and materials

The object of this research is the obtained samples of calcium and magnesium terephthalate. Materials used to study the physico-chemical, mechanical and operational properties of the objects of study: thermoplastic PET, belonging to the class of polyesters, in the form of discarded contaminated PET bottles (see Fig. 1), crushed into 10 mm flakes, bitumen road grades BPA 70/100 and BPA 130/200, a commercial sample of modifier No. 1 (VOLUME- No. 1) based on ethylene copolymer with vinyl acetate (brands are not disclosed, to avoid advertising), a commercial sample of modifier No. 2 (VOLUME - No. 2) based on low melting point polyethylene, commercial sample of modifier No. 3 (VOLUME - No. 3) based on low-molecular-weight low-pressure polyethylene, commercial sample of modifier No. 4 (VOLUME - No. 4) based on reactive ethylene.

2.2. Research methods

2.2.1. Formation method of electron microscopic image

The analysis is carried out on a Vega 3SBH scanning electron microscope and a radiography microanalyzer of the x-Act Energy dispersive type with an accelerating voltage and probe current of 20 keV, $5\cdot10^{-10}$ A и 30 keV, $3\cdot10^{-9}$ A, respectively. The sensitivity of the method is 0.1 wt%. Atoms H, He, and Li are not analyzed elements. PET sample is coated on the Q150R thermal spraying unit with a thin conductive C-coating (20 nm) in order to remove the static charge and improve image contrast. To form an electron microscopic image, signals of secondary and reflected electrons (SE, BSE) are used to obtain morphological and compositional contrast of the image.

2.2.2. Derivatographic method

The research on the thermal stability of the samples is carried out using thermogravimetric (TG) analysis.[12,13] In this study, mixtures of secondary and contaminated plastics are used, therefore thermal properties are important, they allow us to identify the polymer, to assume the physico-chemical and operational properties of the polymer, which allows choosing the optimal method of introducing fiber into concrete.[14]

2.2.3. Method of determining the temperature by softening point ring and ball and the needle penetration depths

Carried out according to GOST 11506-73 and GOST 11501-78.

2.2.4. Method of preparation of bitumen with a modifier

Modification of road bitumen cannot be carried out without preliminary preparation due to differences in the properties of bitumen and polymer (high values of destruction temperature, molecular weight, degree of branching of the polymer). Improper introduction of terephthalates into bitumen worsens the operational properties of asphalt concrete mixture.[14] The proposed method of introducing the additive makes it possible to improve the physico-chemical characteristics of road bitumen. Bitumen was pre-dehydrated, heated to a certain temperature and a modifier was added in portions (from 1 to 5%) while stirring.

3. Theoretical part

3.1. The proposed scheme for obtaining a PET-based modifier

Chemical processing of PET was carried out by its depolymerization into monomers terephthalic acid and ethylene glycol (EG) by alkaline hydrolysis. The general technological scheme for the production of calcium terephthalate and magnesium terephthalate consists of the stages shown in the diagram (Fig. 2). Process conditions and optimal technological parameters of the reaction, such as time, temperature, pressure, and concentrations, will be described below in section 4.1 'Results and discussion'.

3.2. Description of laboratory experiment

Alkaline hydrolysis of PET was performed on a linear automated parallel synthesis platform 'H.E.L'. Contaminated, crushed waste was loaded into the reactor, and water and caustic soda were added. The reaction mass under conditions of intensive mixing

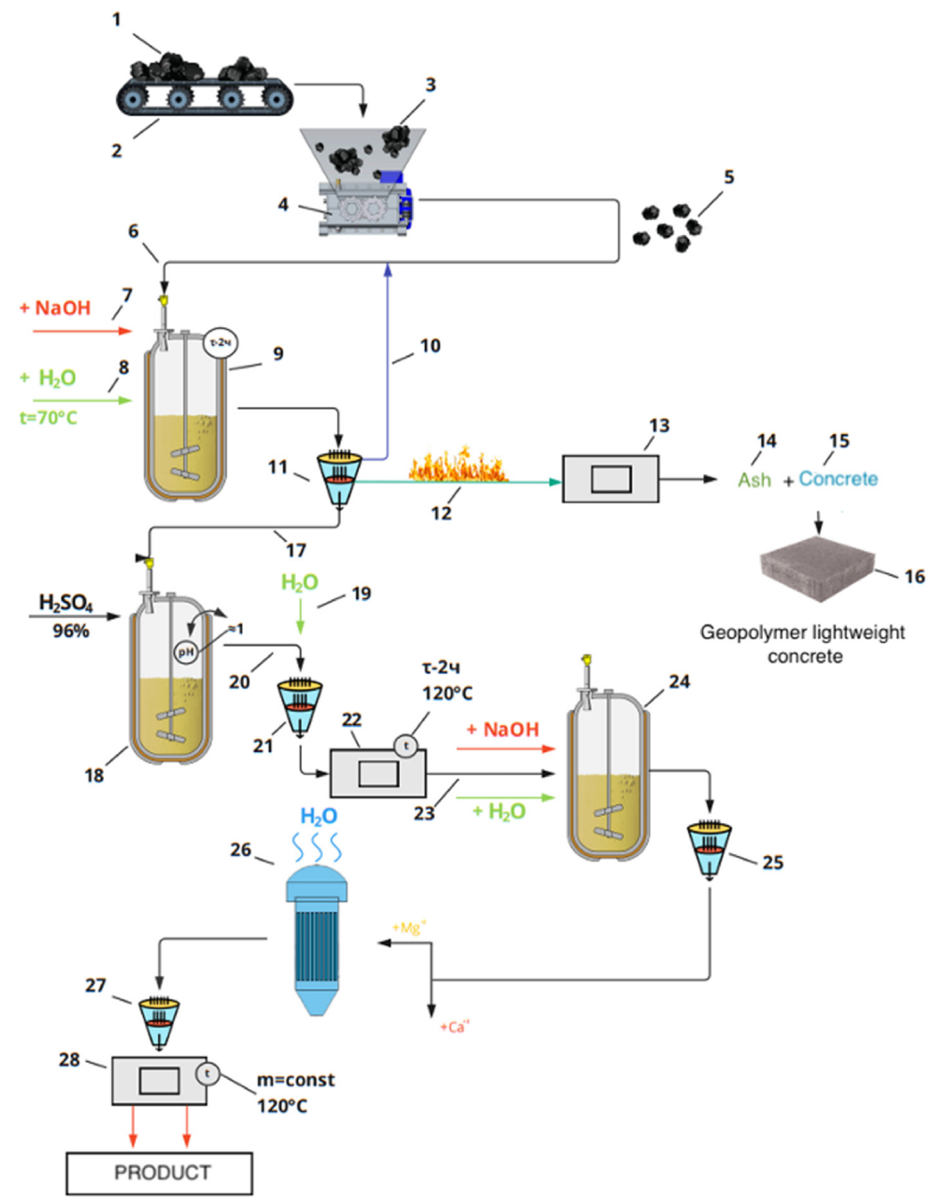


Fig. 2. Scheme of obtaining terephthalic acid and TPM. 1 gathering secondary raw materials, pre–sorting; 2 conveyor feed of raw materials for grinding; 3 sorted and prepared raw materials; 4 grinding of raw materials with a crusher; 5 small flakes up to 6 mm in size; 6 supply of water and raw materials to the reactor (PET flakes); 7 supply of crystalline caustic soda; 8 supply to the reactor with a mixture of reaction water, 9 reactor for the first stage of hydrolysis; 10 solid sludge; 11 vacuum filter; 12 solid sludge for burning and obtaining geopolymers; 13 cement clinker kiln; 14 combustion product of sediment of the first stage of hydrolysis with mechanical impurities from contamination; 15 cement-concrete base mixture; 16 finished improved product 'light concrete'; 17 filtrate; 18 hydrolysis second stage reactor; 19 sludge washing water; 20 reaction mixture; 21 filtration; 22 sludge drying after washing; 23 terephthalic acid; 24 reactor of the final stage of hydrolysis; 25 filtration of the sodium salt of terephthalic acid; 26 evaporation of the mixture to 1/3 of the volume; 27 re-filtration; 28 drying.

was heated to a temperature of 107 °C. Reaction time was 2 h. With a shorter duration of the process, the output of the target product is low. The resulting sodium terephthalate, diluted with hot water (1: 7) before its dissolution, was filtered. As a result of the hydrolysis of PET, sodium salts of oligo ethylene glycol terephthalate, sodium terephthalate and EG were formed. Target products for the production of bitumen adhesive additives are the first two, EG is a by-product. To obtain magnesium terephthalate (Fig. 3), sodium hydroxide was added to terephthalic acid and diluted with water. The sodium salt of terephthalic acid was filtered, and the filtrate was treated with magnesium acetate. Then evaporated to 1/3 of the volume and filtered again. To obtain calcium terephthalate, calcium chloride ($CaCl_2$).

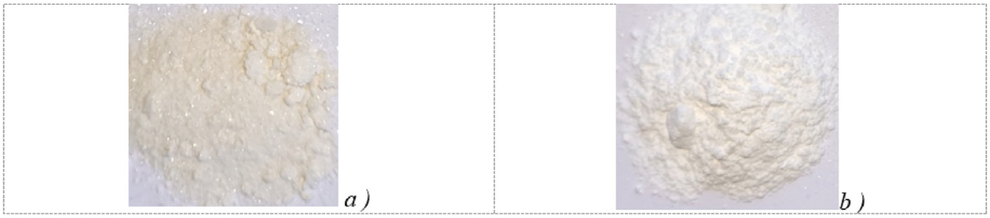


Fig. 3. Terephthalate samples: a magnesium terephthalate, b calcium terephthalate.

Was added to terephthalic acid. The obtained terephthalate samples are shown in the photos of Fig. 3a and b.

4. Results of experiment

4.1. Justification of the choice of polymer processing method results of derivatography

For the chemical method of polymer recycling, the derivatographic TG analysis is less informative than for mechanical processing, however, it allows to set the maximum processing temperature, evaluate the thermal stability of polymer melt.[10,12] In Fig. 4, the DSC curve (blue) shows the change in the heat flux emanating from the polymer and characterizes the change in the energy state of the sample (modification of its structure). TG curve, indicated in red on the graph, shows the loss of mass of the composite with an increase in temperature.

TG studies of the PET sample demonstrate the beginning of polymer destruction at a temperature of 244 °C. As follows from the presented data, the weight loss value is insignificant and amounts to 0.3374 mg (10.17% of the initial mass of the sample). The temperature of PET processing with the thermal method is 280–300 °C. Elevated temperature during processing can lead to partial destruction of polymer, polymer raw materials are modified, its molecular weight decreases, and the release of toxic substances is also possible.[12] Therefore, the chosen chemical method is more efficient than thermal recycling. In addition, the disadvantages of thermal PET recycling include high power consumption, and the need to locate production at a considerable distance from cities, which increases transportation costs.[13]

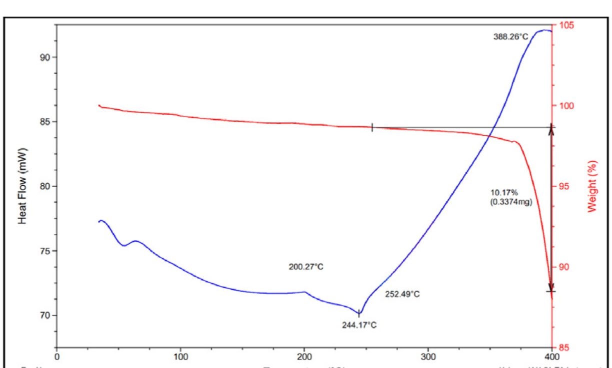


Fig. 4. DSC and thermogravimetric curves of the PET sample.

4.2. Determination of optimal technological parameters for hydrolysis

To determine the optimal conduct of the process, the temperature was studied in the range from 50 to 150 °C. It is possible to visually trace the result in the reactors of different samples shown in Fig. 5. From left to right, samples with the most obvious changes in the reacted substances are demonstrated—50 °C, 80 °C, 100 °C, and 135 °C.

Fig. 6 shows the dependence of sediment mass change on temperature.

It follows from the graph that 100 °C is the optimal temperature for conducting the process when the yield of target hydrolysis products is high. Reducing the temperature to 100 °C, set during the experiment, from the recommended 107–135 °C in the sources listed above, makes it possible to carry out the process with less heat input. Similarly, an experiment was conducted to identify the optimal time for conducting the hydrolysis process. It follows from the graph in Fig. 6 that in the range from 30 to 75 min of the process, the intensity of hydrolysis increases with increasing time, at points from 65 to 100 min, the hydrolysis process is also effective, after 100 min the process slows down

Fig. 5. Sample reactors after the experiment.

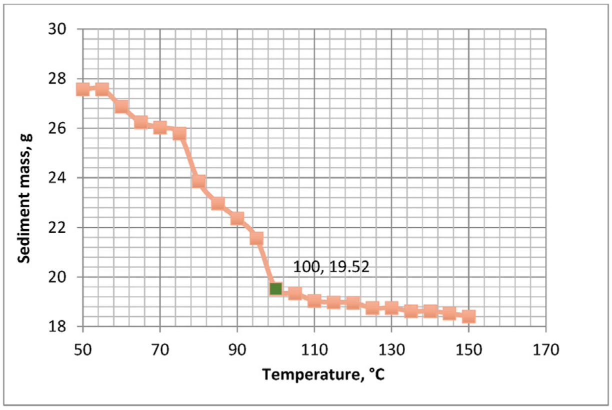


Fig. 6. Intensity of hydrolysis at different temperatures.

significantly. The yield of the product and the mass of resulting sediment changes slightly, increasing the hydrolysis time, as it is shown in Fig. 7.

The optimal time of the process was 105 min. Reducing the process time reduces electricity consumption and leads to a reduction in financial costs for the technological process.

5. Discussion

5.1. Comparison of physicochemical properties of initial and modified bitumen

Adhesive additives were introduced into bitumen grades BPA 70/100 and BPA 130/200 in an amount from 1 to 5 wt%. Evaluation of the physical and mechanical characteristics of bitumen obtained with modifiers was carried out according to the following indicators: softening temperature, and needle penetration depth. The dependence of these indicators on the concentration of modifier in bitumen was determined. The recommended concentration

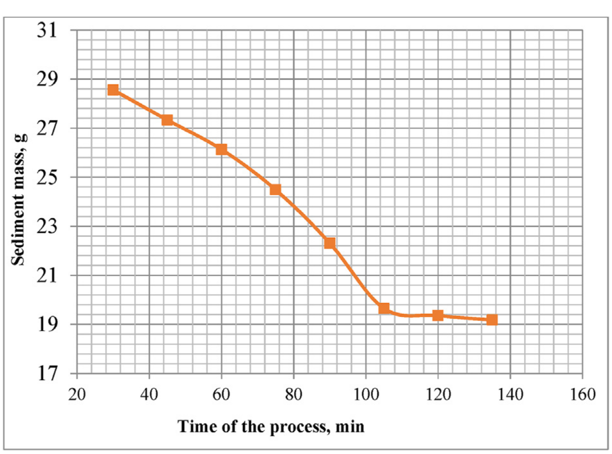
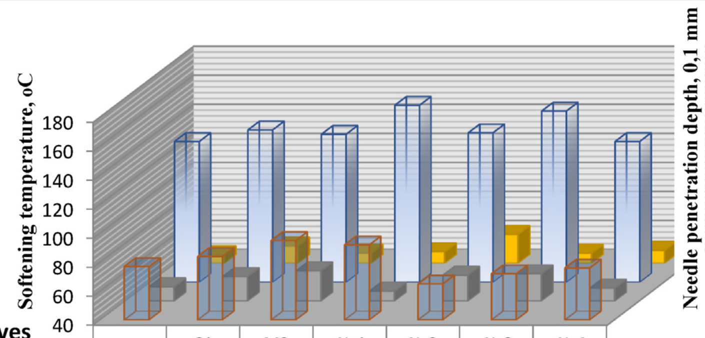


Fig. 7. Intensity of hydrolysis reaction at different times of the process.

of the developed additives was compared with the physicochemical indicators of commodity images: modifier No. 1 (VOLUME - No. 1), No. 2 (VOLUME - No. 2), No. 3 (VOLUME - No. 3), No. 4 (VOLUME - No. 4) (main components of each of the samples is described in the objects of research). Due to significant differences in the properties of road bitumen and polymer additives, it is necessary to prepare bitumen beforehand before introducing calcium terephthalate and magnesium terephthalate. Dehydrated bitumen was heated with constant stirring. Upon reaching the set temperature, a portion modifier was added and the temperature was maintained at 170 °C. After 3–5 min, the mixing speed was increased by 80% from the initial one until a homogeneous mass was obtained.

Indicators of softening temperature and needle penetration depth are improved with the addition of modifiers to road bitumen in an amount of up to 3% of the weight of the bitumen suspension. A further increase in the concentration of additives does not lead to an improvement in the properties of bitumen. The test results for both the obtained calcium (M1) and magnesium (M2) terephthalates and commercial samples are shown in Fig. 8. Based on the diagram in Fig. 10, the modifiers obtained as a result of laboratory tests improve the properties of the analyzed road bitumen BPA 70/100 and BPA 130/200. When calcium terephthalate (M1) was added at a concentration of 3% by weight of bitumen, the softening temperature of BPA 70/100 increased from 50 to 57 °C, and BPA 130/200 from 46 to 52 °C. Needle penetration depth in the BPA 70/100 increased from 77 to 84, and in the BPA 130/200 from 137 to 145. The grade of bitumen BPA 130/200 changed with the addition of magnesium terephthalate (M2) to BPA 90/130, grade of BPA 70/100 remained unchanged with the addition of all modifiers except Volume No. 2 based on polyethylene. When M2 was added at a concentration of 3% by weight of bitumen, the softening temperature of BPA 70/100 increased from 50 to 61 °C, and BPA 130/200 from 46 to 47 °C; needle penetration depth at BPA 70/100 increased from 77 to 95, and at BPA 130/200 from 137 to 142, which meets the requirements of GOST 9128-97 'Mixtures of asphalt road, airfield and asphalt concrete' and can be used for the construction of highways in climatic zone with low temperatures.

Grade of bitumen BPA 130/200 changed with the addition of M2 to BPA 90/130, grade of BPA 70/100 remained unchanged with the addition of all modifiers, except VOLUME No.2 based on polyethylene. Bitumen modifiers obtained during PET hydrolysis have shown high efficiency due to their good



Samles of additives	-	C1	M2	№1	№2	№3	№4
BPA 70/100 Needle penetration depth,0,1mm	77	84	95	92	65	72	76
BPA 70/100 Softening temperature, oC	50	57	61	47	58	59	49
BPA 130/200 Needle penetration depth 0,1 mm	137	145	142	162	143	158	137
BPA 130/200 Softening temperature, oC	46	52	47	48	60	47	49

Fig. 8. Properties of modified bitumen.

solubility in bitumen, they are diphilic in nature. The polar part is oriented to the mineral surface of the final asphalt concrete mixture, providing high adhesion, and the organic part is oriented to bitumen.[10,11] According to the mechanism of action, modifiers can be attributed to adhesive additives. The ability to polarize allows, without changing the concentration of resins and asphaltenes, to transfer of the dispersed phase of bitumen into a gel structure from a sol or sol-gel structure and increases the softening temperatures. Hardness is also increased due to the compaction of the structure of the organic bitumen matrix, which takes on the entire load and increases the structuring of dispersion, which makes it possible to resist brittle fractures, and plastic deformations and improves the quality of dispersed-hardened composite material (asphalt concrete) as a whole.

Nonpolar ethylene links polymerize the 'solvent' in the form of an organic part of bitumen, and the acetate part of the vinyl acetate molecule is polar and exhibits adhesive properties.

5.2. Utilization of hydrolysis sediment after filtration

For the technology to be eco-friendly and meet the principles of a waste-free enterprise, the possibility of disposal of contaminated filtration sediment after hydrolysis is considered. Recycling of PET waste by chemical method allows to remove any contaminant, even attached to the polymer chain. Several ways of disposal of sediments of 'waste' are considered, both by incineration and by adding it without prior preparation to concretes. This precipitate is a fine fraction of PETF. The paper[15] presents the results of the influence of secondary PETF of smooth and ribbed shape (fiber length from 1 to 5 cm) on the strength of concrete. Fig. 9 shows photos of concrete with various fibers to represent the structure of the reinforced material.

Reinforcement with polyamide fibers shows high dispersibility, shrinkage limitations, improved mechanical characteristics (toughness), resistance to alternating temperature changes, and strength.[16] The most reliable fixation of fiber in the concrete matrix can be achieved by introducing into its composition a fiber with a fraction of thickness from 1 to 5 mm and a length from 5 mm to 12 mm. These dimensions correspond to the dimensions of sediment from the first stage of hydrolysis obtained as a result of this work. Michael Short and his team in 2022 proposed to irradiate PETF with gamma rays to increase the strength of concrete.[17] To determine the elemental composition of the samples, the method of electron probe microanalysis was used, based on a comparison of the characteristic radiography spectra of the analyzed sample and standards of known composition. Result of analysis is to obtain an electron microscopic image of film surface (Fig. 10) and data on its elemental composition (Table 1).

Fig. 10 shows the structure of the surface of the PET sample, Spectrum 1 and Spectrum 2 regions are highlighted, the elementary composition of which is shown in Table 1. As a result of research, it was

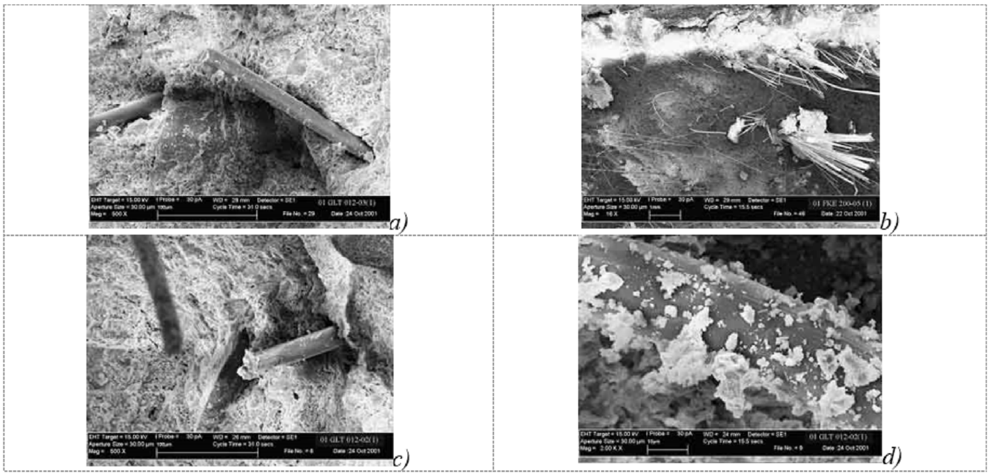


Fig. 9. Photographs of concrete reinforced with various polymer fibers obtained on a scanning electron microscope: a) concrete reinforced with polyamide fiber, b) concrete reinforced with fiberglass, c) concrete reinforced with polypropylene; d) concrete reinforced with PET.[9]

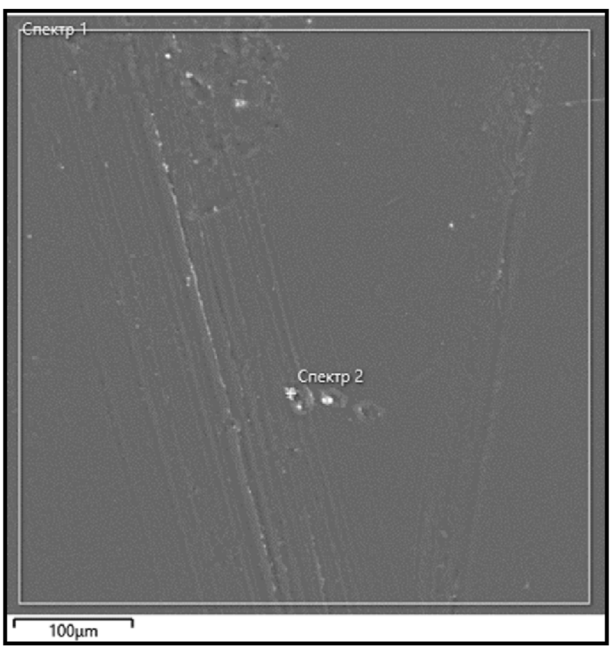


Fig. 10. Micrography of the surface of PET sample film.

found that the sample is an ordinary film made of an organic polymer in which there are no nitrogen and oxygen atoms. On Spectrum 1, chlorine impurities with a content of 0.03% are noted. Spectrum 2 shows surface contamination with sodium, magnesium, aluminum, potassium, titanium and iron metals, as well as increased oxygen and silicon content.

Table 1 shows that the chemical composition of Spectrum 1 is very different from the contaminated mechanical part, designated as the Spectrum 2 region. The elementary composition of contaminants on the surface of PET samples showed that the sediment combustion product can be used similarly to fly ash from a CHP plant to improve the properties of concrete. Composition of ash after combustion of filtration sediment is shown in Table 2.

Researchers have confirmed the possibility of using sediment combustion ash from the filtration process to improve the quality of light concrete, which makes recycling waste-free. High silicon ash modulus, 20–25% higher than that of most known combustion products that are added to concrete.

Table 1. Elemental composition of the surface of PET sample film.

Spectrum label	C	O	Na	Mg	Al	Si	Cl	K	Ti	Fe	Total
Spectrum 1	99,97						0,03				100,00
Spectrum 2	35,62	41,22	0,34	0,42	4,14	15,36	0,29	1,51	0,23	0,85	100,00

Table 2. Chemical composition of sediment combustion ash from filtration processes when using a modifier.

Spectrum Label	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	Na_2O	K_2O	MgO	ппп	Total
Spectrum 1	67,21	13,65	1,97	1,01	1,49	4,42	1,71	8,55	100,00
Spectrum 2	68,42	13,04	2,03	1,46	1,52	4,76	2,65	6,12	100,00
Spectrum 3	67,56	12,89	1,56	1,26	2,02	4,23	1,89	8,59	100,00
Average value	67,73	13,19	1,85	1,24	1,68	4,47	2,08	7,75	100,00

5.3. Conclusions

As a result of the conducted research, the following conclusions were made: 1. Products of chemical processing of PET are promising additives to petroleum bitumen. Application of calcium and magnesium salts of terephthalic acid in bituminous materials can improve durability and mechanical properties of asphalt concrete mixture in terms of tensile strength, and plastic deformation with optimal selection of concentrations of secondary PET waste; 2. All the described technologies for processing plastic waste are possible when organizing separate garbage collection in Russia. It is necessary to introduce separate storage devices for the collection of household waste, to transfer state institutions to separate garbage collection.

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Conflict of interest

The authors declare no conflict of interest.

Author contributions

The authors conceived the study, performed the experiments, analyzed the data, discussed the results, wrote the manuscript, provided critical feedback, and contributed to the final manuscript.

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