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RESEARCH ARTICLE

# Morphology features of biodegradable plastic–elastomer blends

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## Abstract

**Objectives.** To obtain data on the compatibility of a polymer blend based on poly-3-hydroxybutyrate and butadiene-nitrile rubber for the development of a biodegradable polymer with improved mechanical properties.

**Methods.** Film samples of biodegradable plastic–elastomer blends, using mixtures of poly-3-hydroxybutyrate and butadiene-nitrile rubber as a case study, were investigated by means of optical and scanning electron microscopy with computer-aided image analysis, differential scanning calorimetry, mathematical analysis, and Fourier transform infrared spectroscopy.

**Results.** The mixtures studied herein were found to have a heterogeneous heterophase structure. The interaction between the carbonyl group of poly-3-hydroxybutyrate and the nitrile group of acrylonitrile block of acrylonitrile butadiene-nitrile rubber is shown due to kinetic compatibility. A change in the crystalline regions of poly-3-hydroxybutyrate when it is mixed with rubber was also noted. The results of the Gibbs energy calculation of mixing confirmed the interaction of carbonyl and nitrile groups. Microscopy results show the localization of poly-3-hydroxybutyrate particles around rubber particles. The reasons for this phenomenon are discussed here.

**Conclusions.** Studies have shown a relationship between morphology and component content of the samples. The change in composition affects the structure and properties of the surface and volume. The formation of associates leads to the formation of an interface which attracts the second component. The Flory–Huggins theory, Avrami equations, and microscopic data established a complex interaction mechanism: convergence and formation of chemical bonds, rearrangement of crystalline regions, transition of spherulitic particles into lamellar particles, diffusion of rubber macromolecules, association of poly-3-hydroxybutyrate particles around the rubber, and completion of chemical bonds.

**Keywords**

biopolymers, poly-3-hydroxybutyrate, butadiene-nitrile rubber, polymer compatibility, structure morphology, component compatibility

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НАУЧНАЯ СТАТЬЯ

# Особенности морфологии смесей биодеградируемый пластик–эластомер

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## Аннотация

**Цели.** Получение данных о совместимости полимерной смеси на основе поли-3-гидроксибутирата и бутадиен-нитрильного каучука для создания биодеградируемого полимера с улучшенными механическими свойствами.

**Методы.** Методами оптической и сканирующей электронной микроскопии с применением компьютерной обработки, дифференциальной сканирующей калориметрии, математического анализа, инфракрасной Фурье-спектроскопии изучены пленочные образцы смесей биодеградируемого пластика и эластомера на примере смесей полигидроксибутирата и бутадиен-нитрильного каучука.

**Результаты.** Установлено, что изучаемые смеси имеют гетерогенно-гетерофазную структуру. За счет кинетической совместимости проявляется взаимодействие между карбонильной группой полигидроксибутирата и нитрильной группой блока акрилонитрила бутадиен-нитрильного каучука. Замечено изменение кристаллических областей полигидроксибутирата при его смешении с каучуком. По результатам проведенного расчета энергии Гиббса смешения подтверждено взаимодействие карбонильных и нитрильных групп. Результатами микроскопии показана локализация частиц полигидроксибутирата вокруг частиц каучука. Обсуждаются причины такого явления.

**Выводы.** Исследования показали связь между морфологией и содержанием компонентов в образцах. Изменение состава влияет на структуру и свойства поверхности и объема. Формирование ассоциатов приводит к образованию границы раздела, притягивающей второй компонент. По теории Флори–Хаггинса, уравнениям Аврами и микроскопическим данным выявлен сложный механизм взаимодействия: сближение и образование химических связей, перестройка кристаллических областей, переход сферолитных частиц в ламеллярные, диффузия макромолекул каучука, ассоциация частиц полигидроксибутирата вокруг каучука и завершение химических связей.

**Ключевые слова**

биополимеры, полигидроксибутират, бутадиен-нитрильный каучук, совместимость полимеров, морфология структуры, совместимость компонентов

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## INTRODUCTION

The use of traditional materials in industry remains an important aspect of production. This is due to their proven reliability, availability, and well-studied properties, ensuring consistent product quality and optimizing costs [1]. As fossil resources naturally deplete, nonmetallic and composite structural materials have gained increasing importance [2]. At the present time, there has been rapid growth in the production of polymer composites, as well as their integration into various technical sectors, and successful displacement of traditional materials such as metals and ceramics, *inter alia* [3, 4]. The high growth rate of the polymer composite materials market is determined by their wide range of properties. They surpass those of traditional materials, as well as a versatile approach to product creation—from modeling its structure, properties, and form to selecting manufacturing technologies [5].

A disadvantage of these materials is the need for disposal and recycling, since unprocessed plastic waste harms the environment [6, 7]. The majority of plastic produced is single-use plastic. Approximately 40% of plastic produced is used in the packaging sector. The most commonly used thermoplastics for packaging are polyethylene, polyethylene terephthalate, polypropylene, and polystyrene. In 2016, they accounted for over 60% of the total plastic demand in Europe. In Russia, the main share of plastics is also used for packaging, constituting over 40% of the polymer market. This, combined with a culture of single-use, leads to two main consequences: environmental pollution and loss of a valuable resource. In the European Union, 70% of collected plastic waste is sent to landfills or incinerated. In the USA, 53% of all municipal solid wastes ends up in landfills, with plastic waste making up 13% of municipal solid waste. The situation in Russia also remains challenging. Over 3.5 mln tons of plastic waste is generated annually, of which only about 10–12% is recycled [8, 9].

The potential for recycling plastic waste remains largely untapped worldwide. The global plastic recycling rate remains extremely low—only 6% of the total plastic demand. In Russia, recycling also faces challenges such as a lack of infrastructure, low public awareness, and a high proportion of mixed waste which complicates sorting and further disposal [10, 11].

Polymer materials are known to decompose in soil over a period of 100 to 500 years, depending on their composition. When plastics enter the soil, they break down into small particles and begin releasing toxic substances into the environment, such as chlorine, styrene, formaldehyde, phenol, chloroprene, urethane, and others [12, 13]. The use of biodegradable materials could be a solution to this problem.

Biopolymers differ from conventional plastics in their ability to degrade under natural conditions through physical processes and the action of microorganisms such as bacteria and fungi. These polymers are based on biodestruction, i.e., the totality of chemical and physical processes which break down the material under the influence of microorganisms [14].

Biopolymers today account for approximately 1% of the 335 mln tons of polymers produced annually. However, as demand grows and more advanced biopolymers emerge, the market is steadily expanding [15].

When developing and creating new polymeric biodegradable materials, the issue of component compatibility arises. In order to achieve the required set of properties, the problem of stiffness in biodegradable plastics needs to be addressed. The addition of an elastomer is a solution, but then another problem arises. This consists of thermodynamic incompatibility between poly-3-hydroxybutyrate (P3HB) and butadiene-nitrile rubber, due to differences in their solubility parameters, polarity, and macromolecular structure. Thermodynamic incompatibility leads to the formation of a multiphase morphology which can have both negative and positive effects on the blend's properties. In this context, the morphology of such systems becomes of particular interest.

This work is devoted to the study of morphology in a biodegradable plastic–elastomer blend.

## MATERIALS AND METHODS

P3HB, produced by microbial synthesis, was selected from the wide variety of bioresorbable polyhydroxyalkanoates. Due to its biosynthetic origin, P3HB is considered to be one of the safest polyesters of fatty acids. It is a fully biodegradable plastic, resistant to ultraviolet radiation with a number of advantages over others: high biocompatibility, stable properties, and controlled biodegradation across a wide temperature range. Although this polymer is stable in an aqueous environment, it is biodegradable in seawater, soil, composting environments, and waste processing facilities. In compost with 85% humidity and temperatures of 20–60°C, it decomposes into water and carbon dioxide within 7–10 weeks [16–18].

However, films made from P3HB are extremely rigid and brittle which are disadvantages for their use [19]. In order to increase elasticity and, consequently, improve physic-mechanical characteristics, P3HB is blended with butadiene-nitrile synthetic rubber BNKS-28AMN. The main properties of these materials are presented in Tables 1 and 2.

**Table 1.** Main characteristics of P3HB

| Characteristics | Indicator |
|---|---|
| Particle size, μm | 40 |
| Melt flow rate, g/10 min ($T$ = 185°C, 2.16 kg load) | 3.2–3.6 |
| Molecular weight (viscosity average) | 250000 |
| Density, g/cm$^3$ | 1.25 |
| Tensile breaking stress, MPa, no less | 35–40 |
| Relative elongation at break, % at least | 5–7 |
| Glass transition temperature, °C | 5–10 |
| Melting temperature, °C | 173–180 |
| Temperature of thermal destruction onset, °C | 150 |
| Crystallinity level, % | 65–80 |

**Table 2.** Main characteristics of BNKS-28AMN

| Indicator | BNKS-28AMN 2nd group | Testing method |
|---|---|---|
| Mooney viscosity (1+4) 100°C, a.u. | 61–70 | Technical Specification TU 38.30313-2006 |
| Variation of Mooney toughness within the MB batch (1+4) 100°C, a.u. | ≤ 8 | Technical Specification TU 38.30313-2006 |
| Mass fraction of bound acrylic acid nitrile, % | 27–30 | Technical Specification TU 38.30313-2006 |
| Weight loss during drying, % | ≤ 0.8 | Technical Specification TU 38.30313-2006 |
| Mass fraction of ash, % | ≤ 1.0 | Technical Specification TU 38.30313-2006 |
| Methyl ethyl ketone solubility, % | ≥ 95 | Technical Specification TU 38.30313-2006 |
| Antioxidant type | Nonstaining | – |
| Mass fraction of agidol-2 antioxidant, % | 0.5–1.2 | Technical Specification TU 38.30313-2006 |

The polymer blends under study, consisting of P3HB (*Biopolymer*, Germany) and BNKS-28AMN (*Krasnoyarsk Synthetic Rubber Plant*, Russia), were prepared using a solution-based technology. The films were formed by mechanically mixing 2% solutions of the components at 80°C.

The interaction of the components was studied using Fourier transform infrared (FTIR) spectroscopy on a SIMEX FT-801 IR spectrometer (*SIMEX*, Russia) equipped with a diamond attenuated total reflectance accessory.

The feasibility of mixing and component compatibility was assessed based on the results of calculating the enthalpy, entropy, and Gibbs free energy of mixing according to the Flory–Huggins theory.

Morphology analysis was performed using microphotographs obtained with a Levenhuk MedPro 600 FLUO microscope (*Levenhuk*, Russia) equipped with an Altami UCMOC2000KPB digital eyepiece camera (*Altami*, Russia). Additional morphology visualization was carried out using scanning electron microscopy (SEM) by means of a

MIRA 3 LMH microscope (*TESCAN*, Czech Republic) with a Schottky cathode. The surface of the film samples for SEM was sputter-coated with a gold layer approximately 10 nm thick using an S150A Sputter Coater (*Edwards*, United Kingdom). The calculation and determination of crystalline region characteristics were performed using Avrami equations in OriginPro 8 software, based on data from differential scanning calorimetry (DSC) at a scanning rate of 8° per minute, obtained using a DSM-2M microcalorimeter (Russia).

## RESULTS AND DISCUSSION

In order to form a unified system from thermodynamically incompatible polymers, the physical, physicochemical, or purely chemical interaction need to be established. The results of the component compatibility calculation are presented in Tables 3 and 4.

Using the Flory–Huggins theory, it was shown that $\Delta G_{mix} < 0$, when attempting to combine P3HB structures and blocks of acrylic acid nitrile.

However, when calculating for the complete molecules of the components, the calculation results ($\Delta G_{mix} > 0$) confirm the thermodynamic incompatibility of these polymers.

Thus, for P3HB and BNKS, the interaction can be expressed as the formation of bonds between carbonyl and nitrile groups. According to FTIR spectroscopy data, blending these components leads to intermolecular interactions involving the nitrile groups of the rubber and the carbonyl groups of P3HB with the formation of crosslinks. This is evidenced by the intensity of the peak at 2250 cm$^{-1}$, absent in the spectra of the individual omponents.

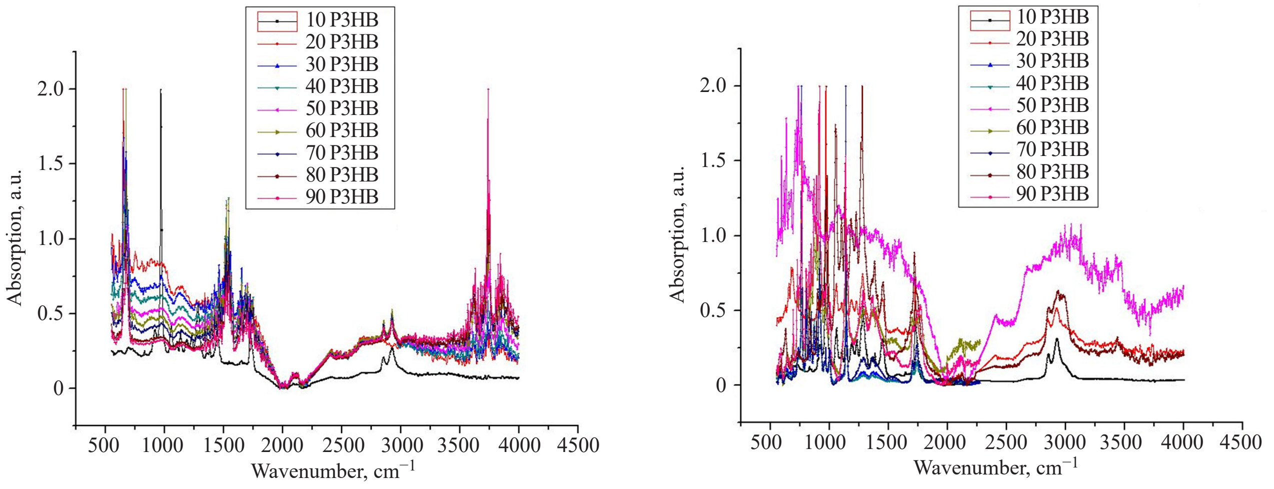
The results of FTIR spectroscopy (Fig. 1a) show that the surface spectra contain a band at ~967 cm$^{-1}$,

**Table 3.** Gibbs energy $\Delta G_{mix}$ of mixing P3HB with acrylonitrile

| Mixture composition, % | | $\Delta G_{mix}$, J/mol |
|---|---|---|
| P3HB | Acrylonitrile | |
| 10 | 90 | −0.006 |
| 20 | 80 | −0.011 |
| 30 | 70 | −0.014 |
| 40 | 60 | −0.017 |
| 50 | 50 | −0.018 |
| 60 | 40 | −0.019 |
| 70 | 30 | −0.018 |
| 80 | 20 | −0.015 |
| 90 | 10 | −0.011 |

**Table 4.** Gibbs energy $\Delta G_{mix}$ of mixing P3HB with BNKS-28AMN

| Mixture composition, % | | $\Delta G_{mix}$, J/mol |
|---|---|---|
| P3HB | BNKS | |
| 10 | 90 | 8585.964 |
| 20 | 80 | 15263.937 |
| 30 | 70 | 20033.918 |
| 40 | 60 | 22895.907 |
| 50 | 50 | 23849.903 |
| 60 | 40 | 22895.907 |
| 70 | 30 | 20033.918 |
| 80 | 20 | 15263.937 |
| 90 | 10 | 8585.964 |



(a)

(b)

**Fig. 1.** Infrared spectra of the surfaces (a) and volumes (b) of the samples

corresponding to trans-structures in BNKS, as well as a band at ~2250 cm$^{-1}$, indicating interaction.

The presence of the 2250 cm$^{-1}$ band characterizes, on the one hand, the nitrile group of the BNKS-28 rubber, and on the other hand, the occurrence of the reaction shown in Fig. 2.

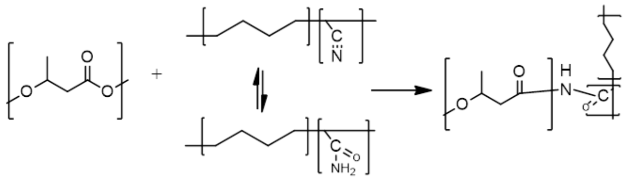


**Fig. 2.** Scheme of chemical interaction between P3HB and BNKS through amide bond formation

In addition to the chemical process, the formation of a compound based on physical interaction is possible (Fig. 3).

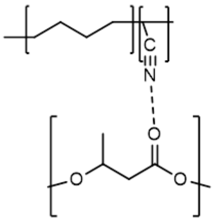


**Fig. 3.** Scheme of compound formation via physical bonds

In the bulk material (Fig. 1b), the 2250 cm$^{-1}$ band practically disappears, the intensity of the ~967 cm$^{-1}$ band decreases, and a series of bands appear in the region of ~1500–1580 cm$^{-1}$, indicating double bonds.

It can thus be concluded that BNKS is the matrix across the entire range of component content, and the interaction between components is stronger on the surface of the samples.

Such interaction should influence the processes of crystallization and melting. Based on this, the parameters of the isothermal crystallization process were calculated using the Avrami equations (Table 5).

The data obtained shows that a transition from spherulitic to lamellar structures occurs, and such structures distribute themselves around the bound rubber chains.

The localization of P3HB particles around rubber chains on the surface can be visualized due to the charging of P3HB particles under an electron beam, and this is confirmed by the results of scanning electron microscopy (Fig. 4).

Microscopy results show that a portion of the P3HB disperses onto the surface. Depending on the composition and strength of component interaction, the presence of P3HB on the surface varies. Analysis of SEM micrographs reveals the localization and aggregation of P3HB particles around the particles of the rubber matrix. Based on the results obtained, and considering the concept of a possible component mechanism, the following conclusions can be drawn:

- When rubber is introduced into P3HB, two modifications of crystallites form during the mutual diffusion of the components which interact differently with the rubber.
- A portion of the crystallites clearly remains in the bulk of the material, while another portion diffuses to the surface. Upon interacting with the rubber, the latter undergoes a transformation from spherulitic to lamellar structures and localizes around the rubber macromolecules bound to them.

Similar processes occur both in the bulk and on the surface. As shown in micrographs taken in transmitted and reflected light (Figs. 5 and 6), the formation of associates takes place in the samples of the studied compositions, occurring primarily on the sample surfaces.

**Table 5.** Parameters of the crystallization process

| Sample | Temperature, °C | Parameters of isothermal crystallization | | | | |
|---|---|---|---|---|---|---|
| | | $n_1$ | $k_1$ | $n_2$ | $k_2$ | $\tau_{0.5}$, s |
| 100% P3HB | 105 | 3.66 | $1.10^{-10}$ | 2.25 | $1.10^{-6}$ | 400 |
| 90% P3HB | | 2.33 | $3.16.10^{-7}$ | 2.0 | $1.10^{-5}$ | 285 |
| 80% P3HB | | 2.56 | $5.10^{-7}$ | 1.25 | $1.10^{-3}$ | 190/380 |
| 70% P3HB | | 3.3 | $4.10^{-9}$ | 1.4 | $1.58.10^{-4}$ | 165/240 |

*Note:* $n_1$ is the Avrami exponent for the primary crystallization stage; $k_1$ is the rate constant for isothermal crystallization for the primary stage; $n_2$ is the Avrami exponent for the secondary crystallization stage; $k_2$ is the rate constant for isothermal crystallization for the secondary stage; $\tau_{0.5}$ is a half-crystallization period.

The computer processing of micrographs allowed particle sizes to be assessed in the bulk and on the surface of the samples (Figs. 7 and 8).

As can be seen from the dependencies presented here, there are more particles on the surface of the samples, and their size distribution is broader. A shift in the size maximum from 70% to 80% P3HB is also observed.

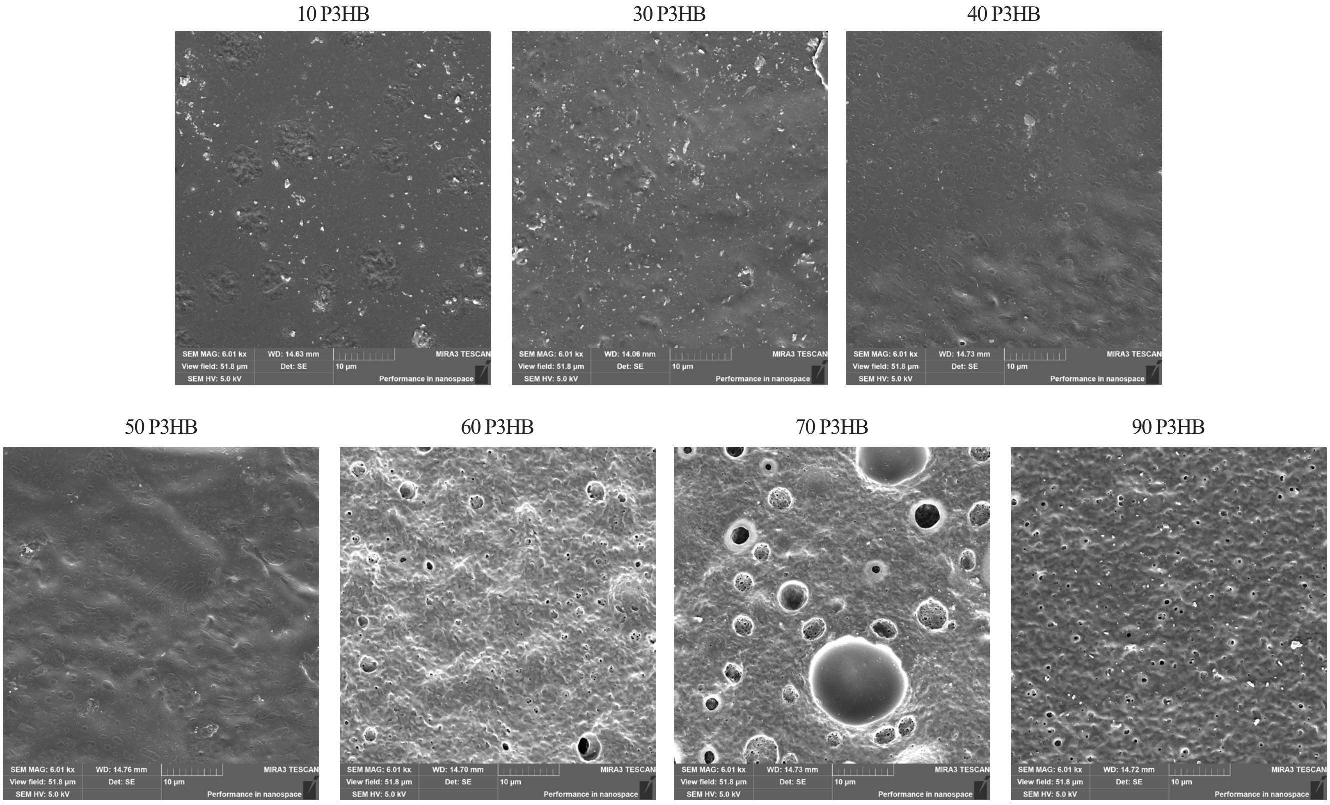


**Fig. 4.** SEM microphotographs

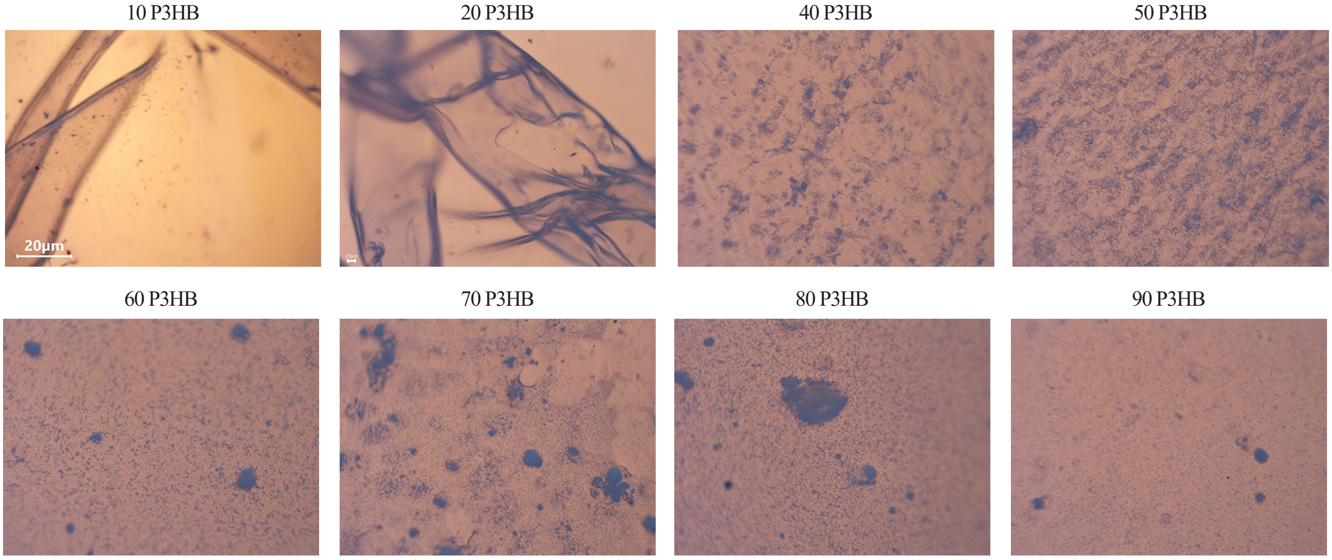
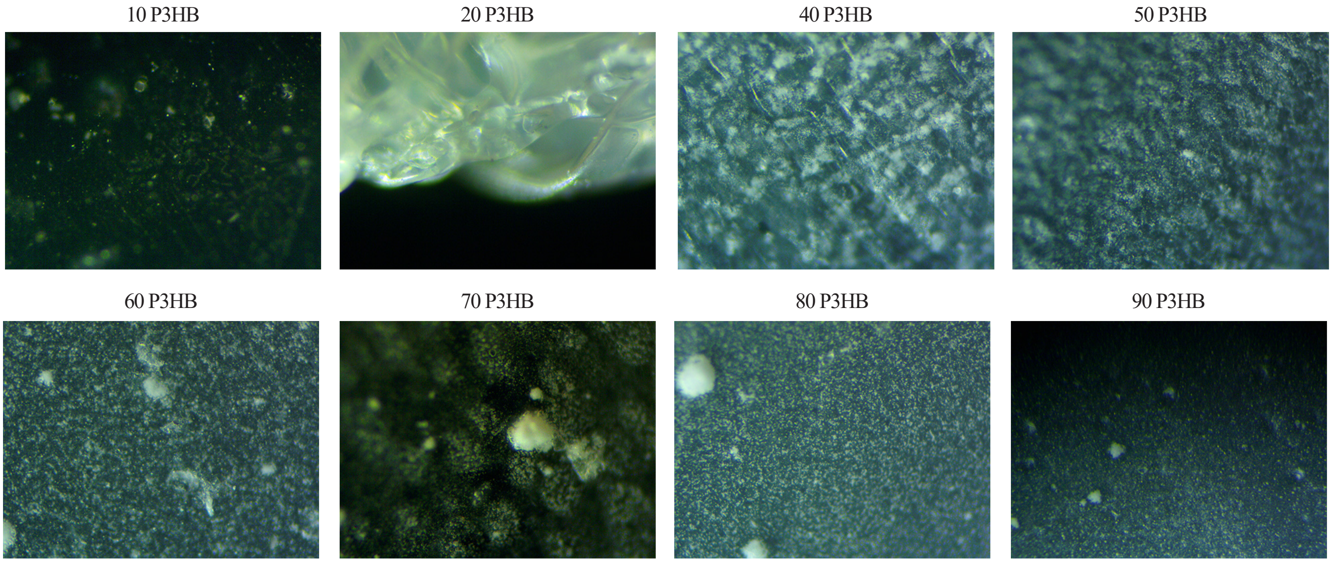


**Fig. 5.** Microphotographs of sample volume (transmitted light, magnification 10×)



10 P3HB
20 P3HB
40 P3HB
50 P3HB
60 P3HB
70 P3HB
80 P3HB
90 P3HB

**Fig. 6.** Microphotographs of sample surface (reflected light, magnification 10×)

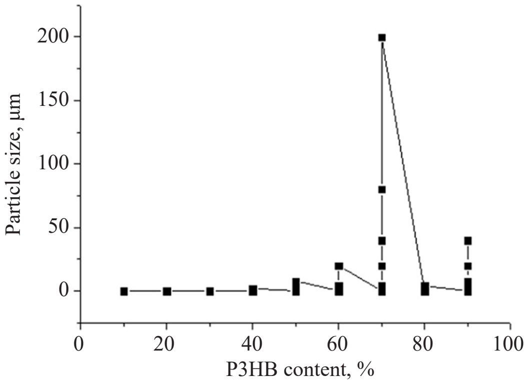


**Fig. 7.** Estimation of particle sizes in the sample volume

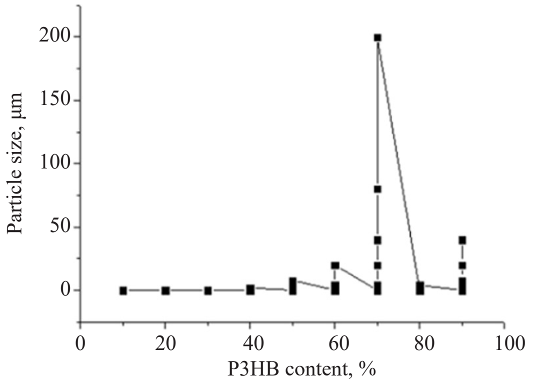


**Fig. 8.** Estimation of particle sizes on the surface of samples

## CONCLUSIONS

The studies conducted herein reveal a relationship between morphology and component content in the samples under investigation. The influence of composition changes on the structure and properties of both the surface and the bulk was also demonstrated. It was shown that the formation of associates leads to the creation of an interface that attracts the second component.

Based on calculations using the Flory–Huggins theory, the particle geometry criterion $n_1$ of the Avrami equations, and the results of optical and scanning electron microscopy, the interaction identified here manifests as quite a complex mechanism. This mechanism sequentially consists of:

- approach of acrylic acid nitrile blocks to P3HB spherulites during heating, initiation of chemical bond formation;
- reorganization of crystalline regions in P3HB upon the introduction of rubber;
- transition of spherulitic particles into lamellar ones;
- diffusion of rubber macromolecules bound to the unfolded spherulites to the surface;
- association of P3HB particles and their localization around rubber particles;
- completion of chemical bond formation.

### Authors' contributions

**N.D. Blinov**—obtaining samples for the study, writing the text of the article.

**L.S. Shibryaeva**—research idea, methodological support, general management.

**M.O. Kulpina**—carrying out the study of the crystallization process.

**A.N. Neretina**—carrying out electron microscopy and its interpretation.

**V.E. Slavkina**—literature review, editing the article.

*The authors declare no conflict of interest.*

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