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FULL PAPER

Impact of ultrasonic treatment on physicochemical properties of polyethylene compositions containing silver nanoparticles

Kirsh Irina Anatolyevna[a],* |Frolova Yulia Vladimirovna[b] |Beznaeva Olga Vladimirovna[a] | Bannikova Olga Anatolyevna[a] |Gubanova Marina Ivanovna[a] |Kondratova Tamara Alexandrovna[a]

[a]*Moscow State University of Food Production, 125080 Moscow, Russia*

[b]*Federal Research Center of Nutrition and Biotechnology, 109240, Moscow, Russia*

***Corresponding Author:**
Kirsh Irina Anatolyevna

Tel.: +7(916)1732158

Ensuring the microbiological safety of food products is a problem currently at issue. The use of antimicrobial packaging materials is a way of solving the problem. Accordingly, the aim of this work was to create packaging materials based on polyethylene (PE) and silver nanoparticles (Ag-NPs) using the ultrasonic treatment of the melt to obtain antimicrobial materials that provide an extended shelf life of packaged food products. Materials based on PE were obtained with Ag-NPs concentrations of 0.5%, 1.0%, 2.0% and 5.0% with and without ultrasonic treatment of melts. Standard methods for studying rheological, physicomechanical, antimicrobial properties and sanitary-chemical indicators of materials were used in the article. It was found that ultrasonic treatment increases the melt flow and contributes to the production of materials with the uniform distribution of additives. The Ag-NPs content from 1.0% and higher in the contents of the material provides antimicrobial properties. The prolongation of the shelf life of the food product, stored in a material based on PE and Ag-NPs 2.0%, was established. In the study of oxygen and water vapour permeability of polymer compositions based on PE and Ag-NPs, it was found that the introduction of a filler increased vapour permeability by about 8-10%, compared with the control samples. The development of polymer composite materials (PCM) with antimicrobial properties and degradability was carried out.

KEYWORDS

Polyethylene; thermoplastic starch; silver nanoparticles; antimicrobial properties; physico-chemical characteristics; ultrasonic treatment; biodegradation.

Introduction

Research in the field of application of nanotechnology, including silver nanoparticles (Ag-NPs), in various industries, is still prevalent today, which is connected with their excellent antimicrobial properties [1]. Efficiency in inhibiting the growth of many pathogenic and conditionally pathogenic microorganisms such as Escherichia coli, Staphylococcus aureus, Pseudomonas aeruginosa, Bacillus cereus, Bacillus subtilis, Candida albicans, Listeria innocua, Salmonella pycus choleraesuis, Stregaris mucus, has been considered in numerous works [2,3]. In the food industry, a developing area is the use of nanoparticles to create packaging with antimicrobial properties for food safety [4].

Food products are the nutrient substrate for the vital activity of microorganisms that can cause spoilage of products and produce substances that have an adverse effect on the consumers' body. The use of packaging allows ensuring the safety of food products during production, storage, transportation and sale. To ensure the protection of food products, packaging must have good strength characteristics, barrier and antimicrobial properties [5]. To impart antimicrobial properties to the packaging, substances of both natural [6,7] and synthetic origin [8,9] are used. The use of silver nanoparticles in packaging is considered as an object allowing the creation of innovative nanocomposite materials for food products packaging. In addition to good antimicrobial properties, the use of Ag-NPs in packaging does not change the organoleptic characteristics of the packaged product [10].

The inclusion of natural compounds, such as essential oils, into the polymer matrix can lead to a change in the organoleptic characteristics of the packaged product, which limits their use [11]. The antimicrobial activity of Ag-NPs is better than natural compounds, and due to the slow rate of release of silver ions into the packaged product, they can become an excellent alternative to natural compounds in the packaging. Effective protection of packaged products in materials with Ag-NPs from microbial spoilage has been demonstrated in a number of works and on various food products, including meat and meat products, dairy products, bakery and confectionery products, and others [12-15].

The properties of modified packaging materials depend on the uniform distribution of auxiliary components in the polymer matrix. When modifiers are introduced into the polymer matrix, there is a high probability of their agglomeration [16], which leads to uneven distribution of modifiers in the volume and to inhomogeneous material characteristics. To prevent agglomeration of the introduced components in the polymer matrix, the use of the ultrasonic (US) treatment of the polymer melt is currently being investigated. The use of the ultrasonic treatment of the polymer melt leads to the uniform distribution of modifiers in the volume of the polymer matrix and prevents agglomeration of the introduced components [17].

When Ag-NPs is added to the package, it is necessary to take into account the possible agglomeration of nanoparticles, which will lead to the increase in their size and the decrease in antimicrobial activity, since there is the dependence of the antimicrobial activity of Ag-NPs on their size [18]. Thus, in the manufacture of modified polymer materials, it is advisable to apply the ultrasonic treatment of the melt to obtain packaging materials with uniformly distributed components and stable properties.

However, the environmental factor should be taken into account during the development of packaging materials. Currently, the issue of recycling polymeric materials, including packaging, is very acute [19]; therefore, the developed packaging materials must not only have the necessary set of properties that ensure the quality and safety of food products but also decompose under the influence of the environment after use. Considering this issue, the purpose of this work was to study the effect of the ultrasonic treatment on the physicochemical properties of biodegradable polyethylene compositions containing silver nanoparticles.

Materials and methods

Materials

The following materials were applied in this study: High-pressure polyethylene (PE) Kazpelen 15813-020 (Kazanorgsintez, Kazan, Russia), silver nanoparticles (Ag-NPs) in the form of the paste, brand XFNANO XFJ14 Jiangsu XFNANO Materials Tech Co., Ltd. (purity 99.9%, size 60-80 nm, diameter 80-90 nm), thermal stabilizer (plasticizer) Irganox 1010 (BASF, The Chemical Company, Basel, Switzerland); thermoplastic starch with the particle size of 30-50 microns. Nutrient media for microbiological research: dry nutrient medium Saburo (State Scientific Center for Nuclear Medicine, Obolensk, Russia), dry culture medium Chapeka-Doxa (State Scientific Center for Nuclear Medicine, Obolensk, Russia), meat peptone agar (State Scientific Center for Nuclear Medicine, Obolensk, Russia). All other reagents, used to analyze the properties of materials, had analytical purity.

Polymer materials production technology

To obtain polymer compositions, we used the basic principle described in previous research [20]. The production of test samples of films was carried out in two stages: The first stage was the production of granules (diameter 3 mm, length 6 mm), and the second stage was the production of film materials. The ultrasonic treatment (US) of the melt of polymer compositions was carried out at 22.4 kHz. General temperature conditions of polyethylene compositions are presented in Table 1.

TABLE 1 Temperature conditions for processing polyethylene compositions in the extruder

Polymer compositions [1]	Temperature conditions for processing polyethylene compositions in the extruder, °C			
	1 zone	2 zone	3 zone	4 zone
PE without US	120	180	190	200
PE _Ag-NPs without US	120	180	190	200
PE with US	120	170	180	190
PE _Ag-NPs with US	120	170	180	190
PE _Ag-NPs_S without US	120	140	160	170
PE _Ag-NPs_S with US	120	130	140	160

Composition and conditions for obtaining polymer compositions

The compositions obtained were as follows: PE without the ultrasonic treatment - pure PE obtained without the ultrasonic treatment of the melt; PE_Ag-NPs without the ultrasonic treatment - PE with silver nanoparticles, obtained without sonication; PE with the ultrasonic treatment - pure PE obtained with the ultrasonic treatment of the melt, PE_Ag-NPs with the ultrasonic treatment - PE with silver nanoparticles, obtained with the ultrasonic treatment; PE_Ag-NPs_S without the ultrasonic treatment - PE with silver nanoparticles and starch, obtained without the ultrasonic treatment; PE_Ag-NPs_S with the ultrasonic treatment - PE with silver nanoparticles and starch, obtained with the ultrasonic treatment. For this research, samples of polymer materials were obtained using the technology described above, the component composition of which are presented in Table 2. Polymeric compositions, obtained without the influence of the ultrasonic treatment on their melts, were used as control samples.

TABLE 2 Component content of polyethylene compositions

Polymer compositions	Component content of polyethylene compositions, %			
	PE	Ag-NPs	Irganox 1010	thermoplastic starch
PE	100,00	0	0	0
PE_Ag-NPs-0,25	99,75	0,25	0	0
PE_Ag-NPs-0,5	99,50	0,50	0	0
PE_Ag-NPs-0,75	99,25	0,75	0	0
PE_Ag-NPs_50-0,25S	48,75	0,25	1,0	50
PE_Ag-NPs_50-0,5S	48,50	0,50	1,0	50
PE_Ag-NPs_50-0,75S	48,25	0,75	1,0	50

Characteristics of polymeric materials

Rheological studies

The studies of polymer melts were carried out by the standard method of capillary viscometry [17]. The studies were carried out in five replicates.

Determination of the thickness of the films obtained

The thickness of the obtained materials was measured with a micrometre (Manufacturer). The measurement was carried out at five different locations with the average calculation.

physicomechanical properties

Determination of the breaking stress and relative elongation was carried out on the Instron tensile testing machine (Switzerland). For testing, the samples were cut in the form of strips with a size of 1.0 × 10.0 cm. The tensile speed of the materials was 10 mm / s. The studies were carried out in five replicates.

Study of antimicrobial properties

Studies of the antimicrobial properties of polymeric materials were carried out according to the method [20]. Cultures of microorganisms used as follows: Bacteria Escherichia coli M 17 (E. coli), Bacillus subtilis ATCC 6633 (B. subtilis), yeast fungus Candida albicans (C. Albicans), moulds Penicillium brevicompactum F-4481 (P. brevicompactum niger), Aspergillis 82 (A. niger). The assessment of the antimicrobial activity of the samples of polymer compositions was assessed visually by the presence of the inhibition zone, inhibition of the culture growth under and on the surface of the studied materials.

Sanitary-chemical research

To carry out sanitary-chemical studies, water extracts were obtained from material samples. The volume of the model medium (distilled water) was 1 cm3 per 2 cm2 of the surface of the sample under study. The exposure time was ten days at a temperature of 23 ± 2 °C. Then, the obtained extracts were analyzed.

The organoleptic indicators of the extracts were carried out by visual expert assessment according to the following indicators: Color, transparency, turbidity, sediment, odor. The smell was assessed on a five-point scale, where 0-1 points was the absence of smell and/or the presence of the weak odor characteristic of the material, determined not by all experts, and 5 points was the presence of the sharp, specific smell, determined by all experts. The number of independent experts was ten people.

The quantitative assessment of migrated low-molecular substances from the material samples into the model medium was carried out by the standard gas chromatography method using the high-performance liquid chromatography (Agilent 1200, Agilent Technologies Inc., USA).

The concentration of migrated silver nanoparticles into the model environment was determined by the method of atomic absorption spectrometry on the Spectr-5 atomic absorption spectrometer (Manufacturer). The calculation of the magnitude of migration of nanoparticles was carried out by the formula:

$m=c/v$, where

m - migration of silver nanoparticles (mg / dm3)

c - the quantity of nanoparticles found in the aliquot of volume V.

Assessment of biodegradability of the materials

The studies were carried out in accordance with the method [20]. Samples of polymeric materials were cut out 1 × 10 cm in size and placed in a container with soil with the moisture content of at least 50% of its maximum moisture capacity. A layer of soil 1.5±0.5 cm thick was poured over the samples, and loosely closed containers were placed in a chamber at the temperature of 23±2 °C and the humidity of 70±5%. Temperature and humidity were monitored throughout the composting process. The composting time for polymeric materials was six months. The degree of biodegradation of the materials was determined by the change in physical and mechanical properties according to paragraph 2.3.3.

Statistical processing

The results were statistically processed using the IBM SPSS Statistics Ver. 20 (SPSS Inc. USA)

Results and discussion

The developed technology for producing polymer materials with the ultrasonic treatment of melts made it possible to reduce the temperature of extruder zones 2, 3, and 4 by 10 ° C. The decrease in the processing temperature of polymer compositions can potentially lead to a decrease in the formation of degradation products, both of the polymer base and of the introduced components.

When receiving polymeric materials, one of the main indicators is rheological characteristics. The nature of the change in the melt flow rate (MFR), depending on the investigated composition is presented in Figures 1 and 2.

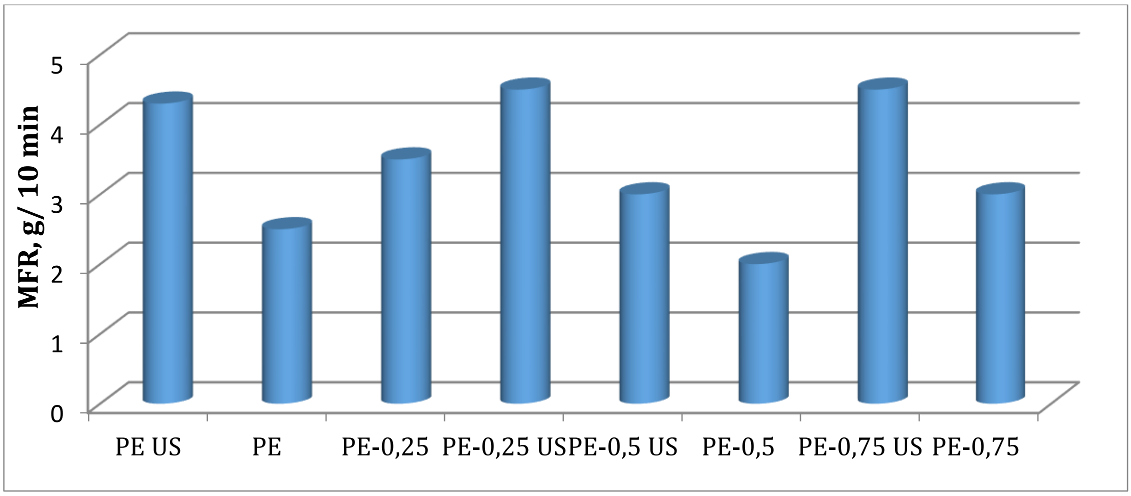

FIGURE 1 Graph of the dependence of the MFI on the composition of polymer compositions modified with silver nanoparticles

The introduction of silver nanoparticles in concentrations up to 3% does not significantly change the melt flow rate. The ultrasonic treatment of melts of polymer compositions in all cases leads to the increase in MFI, which is consistent with the data [17, 20] on the effect of the ultrasonic treatment on melts of polymers.

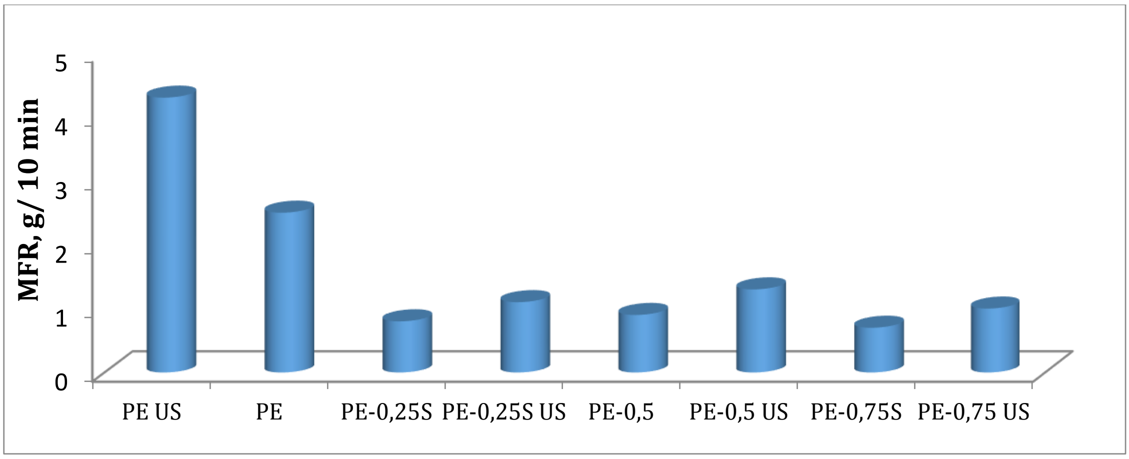

FIGURE 2 Graph of the dependence of the MFI on the composition of polymer compositions modified with silver nanoparticles and starch

Similar data on the effect of the ultrasonic treatment on polymer melts were obtained for polymer compositions containing silver nanoparticles and thermoplastic starch. However, the introduction of starch into the composition of the compositions leads to the decrease in the melt flow rate, while the increase in the starch content in the polymer composition correlates with the decrease in the MFI, which is consistent with the data presented in previous research [21].

Changes in the physicomechanical properties of polymer compositions modified by Ag-NPs depending on the content of the modifier and the ultrasonic treatment are presented on Figures 3 and 4.

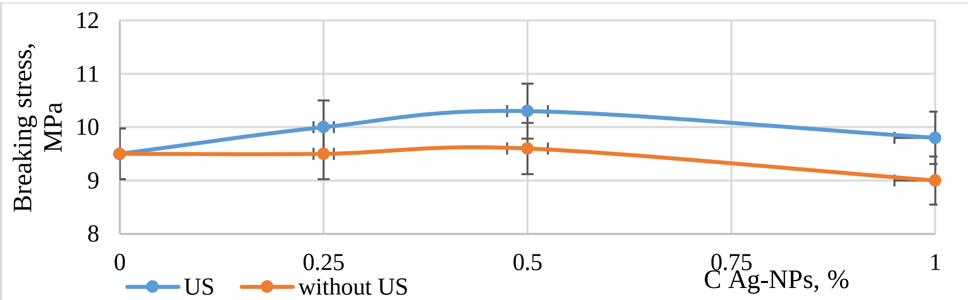

FIGURE 3 Dependence of the breaking stress on the content of Ag-NPs in polyethylene (PE) and ultrasonic (US) treatment

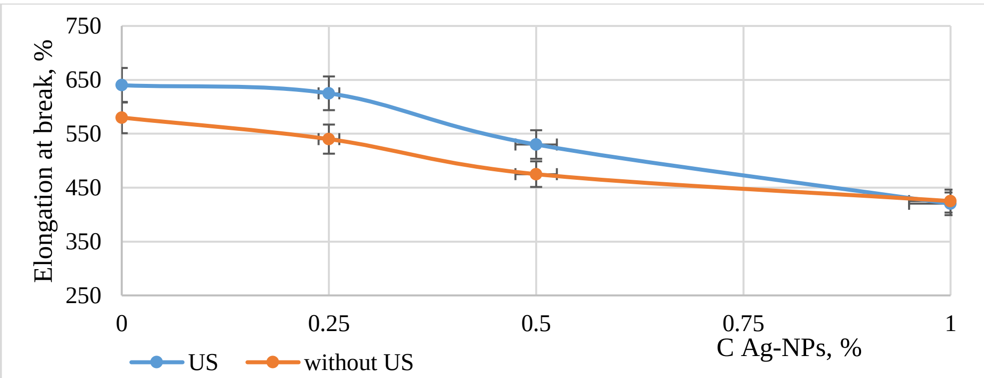

FIGURE 4 Dependence of elongation at the break on the content of Ag-NPs in polyethylene (PE) and ultrasonic (US) treatment

It was found out that the modification of polyethylene compositions with silver nanoparticles practically does not change the physicomechanical characteristics of the samples under study, while the ultrasonic treatment leads to an increase in the indicators relative to similar samples obtained without ultrasonic treatment of melts.

The introduction of starch into the composition of the polymer matrix leads to the decrease in physicomechanical parameters; this is due to the poor compatibility of starch with polyethylene [22]. The use of thermoplastic starch improves the compatibility of the components in polyethylene compositions by enhancing interfacial adhesion [23]; however, according to the data obtained, with the increase in the starch content in the composition, the mechanical properties decrease. The use of the ultrasonic treatment of melts leads to the improvement in the physicomechanical parameters of the film samples, which is most likely associated with the better dispersion of starch in the polyethylene polymer matrix and, as a consequence, the increase in the interfacial adhesion between PE and starch. On the basis of rheological and physicomechanical parameters for modifying the properties of polyethylene films, it is advisable to use the ultrasonic treatment of the melt and the content of Ag-NPs modifiers from 0,25% to 0,75% and starch not more 50 %. With such a ratio of components, satisfactory values of strength characteristics are retained, and potentially materials have antimicrobial properties due to the content of Ag-NPs and are biodegradable due to starch. Further studies were carried out to determine the antimicrobial properties and biodegradability of the selected modified compositions.

Table 5 shows the results of the study of the antimicrobial activity of the studied samples of polymer compositions containing Ag-NPs.

TABLE 5 Results of visual assessment of the surface of polymeric materials based on polyethylene (PE) with silver nanoparticles (Ag-NPs), obtained with and without using the ultrasonic treatment, inoculated for 24–48 hours

Polymer composition	**Visual assessment**				
	E.coli	***B.subtilis***	***C. albicans***	***P. brevicompactum***	***A. niger***
PE US	Growth on the surface	Growth on the surface	Growth on the surface	Growth on the surface	Growth on the surface
PE	Growth on the surface	Growth on the surface	Growth on the surface	Growth on the surface	Growth on the surface
PE_Ag-NPs-0,25S US	Growth retardation on the surface	Growth retardation on the surface	Growth retardation on the surface	Growth retardation on the surface	Growth retardation on the surface
PE_Ag-NPs-0,25S	Growth retardation on the surface	Growth retardation on the surface	Growth retardation on the surface	Growth retardation on the surface	Growth retardation on the surface
PE_Ag-NPs-0,5 US	No growth on the surface	No growth on the surface	No growth on the surface	No growth on the surface	No growth on the surface
PE_Ag-NPs-0,5	No growth on the surface	No growth on the surface	No growth on the surface	No growth on the surface	No growth on the surface

PE_Ag-NPs-0,75S US	No growth on the surface	No growth on the surface	No growth on the surface	No growth on the surface	No growth on the surface
PE_Ag-NPs-0,75S	No growth on the surface	No growth on the surface	No growth on the surface	No growth on the surface	No growth on the surface
PE_Ag-NPs-0,75 US	Zone of inhibition 1,8±0,2 mm	Zone of inhibition 1,8±0,2 mm	Zone of inhibition 1,5±0,1 mm	Zone of inhibition 3,0±0,5 mm	Zone of inhibition 1,8±0,2 mm
PE_Ag-NPs-0,75	Zone of inhibition 1,7±0,2 mm	Zone of inhibition 1,7±0,2 mm	Zone of inhibition 1,5±0,2 mm	Zone of inhibition 3,1±0,5 mm	Zone of inhibition 1,7±0,2 mm

As a result of the studies, it was found that the introduction of silver nanoparticles into the polymer matrix of polyethylene leads to the production of a material with antimicrobial properties against the studied microorganisms, which is consistent with the data on the growth inhibition of various microorganisms presented in previous research [24]. It should be noted that the degree of inhibition of culture growth depended not only on the concentration of Ag-NPs but also on the strain of the microorganism. Similar phenomena have been described previously [25]. For the manifestation of antimicrobial activity, silver nanoparticles must be in contact with the surface of the food product or migrate from the package into the volume of the product. According to the European Food Safety Authority, (EFSA) Commission on Food Additives and Sources of Food Additives, the limit for the migration of silver from packaging to food is set at 0.05 mg/kg (or 0.05 mg/L) [26]. As a result of the studies carried out on the migration of silver nanoparticles, it was found that with the increase in the content of Ag-NPs in the composition of the polymer composition, the level of migrating particles increased and at the content of 1 % exceeds the permissible limit. Therefore, for food products packaging, it is advisable to use starch-filled polymer compositions with the Ag-NPs content of no more than 1 %. The results of evaluating the migration of normalized low molecular weight substances into the model environment from the selected modified polymer composition are presented in Table 6.

TABLE 6 The results of migration of normalized low-molecular substances from the sample PE_Ag-NPs_ 0,75_S_ with the ultrasonic treatment into the model environment

Name of the indicator, mg / dm3	Norm according to TR CU 005/2011	Actual value, Exposure time ten days in the water
Acetaldehyde	No more than 0,2	Less than 0,05
Ethyl acetate,	No more than 0,1	Less than 0,05
Hexane, mg / dm3	No more than 0,1	Less than 0,005
Heptane, mg / dm3	No more than 0,1	Less than 0,005
Acetone, mg / dm3	No more than 0,1	Less than 0,05
Formaldehyde, mg / dm3	No more than 0,1	Less than 0,025
Methyl alcohol, mg / dm3	No more than 0,2	0,05
Butyl alcohol, mg / dm3	No more than 0,5	Less than 0,05
Isobutyl alcohol, mg / dm3	No more than 0,5	Less than 0,05
Propyl alcohol, mg / dm3	No more than 0,1	Less than 0,05
Isopropyl alcohol, mg / dm3	No more than 0,1	Less than 0,05

Based on the obtained results of migration of low molecular weight substances, the developed material can potentially be used as a packaging material for food products, since, according to the preliminary data, it complies with the standards. However, more research is required to confirm the safety of the developed packaging material more accurately.

At the final stage, the biodegradability of the developed starch-filled compositions, containing Ag-NPs, was evaluated by composting. The evaluation criterion was the degree of change in the physicomechanical properties of the films after six months of composting. The data obtained are presented in Table 7.

TABLE 7 Change in breaking stress and elongation at break of compositions containing Ag-NPs and starch after six months of composting

Polymer composition	Change breaking stress, MPa	Change elongation at break %
PE_Ag-NPs-0,5S US	28±2	40±5
PE_Ag-NPs-0,25S	20±2	32±4
PE_Ag-NPs-0,75S US	22±1	28±2
PE_Ag-NPs-0,75S	17±1	20±2

The content of Ag-NPs leads to the slowdown in the process of decomposition of materials, relative to materials that do not contain nanoparticles, which is associated with their antimicrobial activity; however, the strength characteristics for six months of composting significantly decreased by 30-40 %, which indicates the destruction of polymer compositions. It should be noted that the materials obtained with the ultrasonic treatment showed a more significant decrease in strength characteristics, which is associated with better dispersion of starch in the polymer matrix and the increase in the content of oxygen-containing groups in the polymer [27].

Conclusion

We investigated polymer compositions based on polyethylene, modified with thermoplastic starch and/or silver nanoparticles. Technology has been developed for producing starch-filled polyethylene compositions, containing silver nanoparticles with the ultrasonic treatment of a polymer melt. On the basis of studies of rheological and physical-mechanical characteristics, the optimal ratios of the components of PE-AG-NPs-starch () were established. The levels of migration of components from modified polymeric materials, which are normalized when used for contact with food, were identified, and their compliance was established. Based on the determination of the antimicrobial activity and biodegradability of the materials, the optimal ratios of the components were determined to obtain a polymer material based on polyethylene modified with starch and silver nanoparticles (PE-AG-NPs-starch), which in turn was meant to obtain a biodegradable material with antimicrobial properties. It is suggested that other antibacterial nanocomposites be used in future studies and the results be compared with the results of this study.

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Conflicts of Interest

The authors declare no conflicts of interest.

Orcid:
Kirsh Irina Anatolyevna:
https://orcid.org/0000-0003-4569-7035

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