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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

INFLUENCE OF SCALES ON STRUCTURE AND PROPERTIES OF LOW DENSITY POLYETHYLENE MATRIX COMPOSITES

Speciality:	3361.01 – Solid state electronics, radioelectronic components, micro and nanoelectronics (in physics)
Field of science:	Physics
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The dissertation work was performed at the Ecology Institute of the National Aerospace Agency

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GENERAL CHARACTERISTICS OF THE WORK

Research issue rationale and development rate. Polymers are widely used as dielectric and electret materials in various fields of technology. Their application-relevant physical properties such as dielectric permittivity, dielectric loss, specific electrical resistivity, electrical and mechanical strength, and others make them essential components in insulating materials, electronic devices, capacitors, and high-voltage machines, generators, as well as many other devices. Composite materials obtained by adding various organic or inorganic additives to polymers to meet the ever-increasing demand allow further expansion of the practical capabilities of the above-mentioned areas of application. The wide range of applications of polymer composites and the ability to obtain materials with controllable physical and chemical properties on their basis opens up a wide range of applications in microelectronics and radio engineering, as well as in all areas of technology as construction materials. For example, the development of different types of capacitors with high specific capacity for microsystem technology is carried out by increasing the operating voltage and dielectric constant of the polymer layers placed between their shirts.

The main requirements for composite materials based on polymers are high electrical and mechanical resistance, resistance to thermal energy and electric fields, mechanical strength, resistance to various chemical influences and radiation, and most importantly, their low cost. Since polymers are materials obtained from the chemical bonding of long molecules consisting of a large number of identical atomic groups, the high quality of products based on them depends on the choice of material and the level of technological processes.

Polymers used in the manufacture of parts for nuclear reactors, electrical insulation materials, conductors, energy converters and their composite materials based on them are exposed to various effects in spacecraft. However, most dielectric materials are made of highly motivated materials, ie., polymers. These materials have high dielectric properties, as well as high physical, mechanical and chemical properties. These materials are more efficient for the manufacture of products than inorganic dielectrics. Specific masses are lighter than inorganic substances and, finally, many times cheaper than them. This set of features opens wide opportunities for the use of polymers as a whole (polyethylene, polystyrene, polypropylene, fluoroplastics, polymethyl methacrylate, etc.) in various industries. In the development of modern devices and the further improvement of existing ones, the search for materials whose properties can be controlled has always been relevant, as pure polymers cannot fully meet the requirements for them. Thus, it is not always possible to solve existing problems using only polymers. For this reason, it is necessary to modify polymers to increase their resistance to heat and radiation, to improve their physical and mechanical properties to increase their resistance to frost and moisture. The most convenient of the available modification methods is the use of fillers of different origins. Despite the acquisition of new types of composites with high physical-mechanical, dielectric characteristics with the use of such fillers, there is always a need to obtain materials that can meet the ever-increasing scientific and practical needs.

A study of the literature has shown that biocomposites obtained using biological fillers have superior properties over other analogues. It is widely used in everything from biocomposites to the manufacture of household items, food products and their preservatives to modern spacecraft, which stimulates research in the field of biocomposites. Biocomposites can be used both separately and as an adjunct to various materials. Biocomposites have a number of properties; it is not harmful to health, it is not dangerous, it is light weight, it has practically no impact on environmental pollution, which shows that the use of biocomposites is more efficient. Biocomposites are composite materials obtained by adding natural fibres. These types of materials are often perceived as structures present in living organisms. The main components of biocomposites are materials from biological sources, which are rich in natural resources, cheaper and easier to process.

Thus, the analysis of the literature shows that the new composite materials obtained by making biological additives to low-density polyethylene can be considered interesting research objects. Given the above, the relevance of the study of composites obtained by adding fish scales to low-density polyethylene is undoubted.

The object and subject of research. The object of the research is low-density polyethylene-based biocomposites and bionanocomposites modified with fish scales, and the subject is the study of the dielectric, mechanical and electrical properties of these materials, as well as their behavior under the influence of a strong electric field, X-ray phase analysis, surface morphology and UV-IR radiation.

The purpose and objectives of the study. This study focuses on the development of polymer-based composite materials modified with fish scales, the comprehensive analysis of their structural and physico-chemical properties, and the assessment of their potential applications across various technological fields.

To achieve this goal, it was necessary to resolve the following issues.

1. Obtaining biocomposites LDPE + x vol.% FS using special technological methods;
2. Study of the surface micro relief of LDPE + x vol.% FS biocomposites using an atomic force microscope (AFM), depending on the number of fish scales;
3. Phase analysis of biocomposites LDPE + x vol.% FS using X-ray analysis;
4. Investigation of frequency and temperature dependences of dielectric parameters - the tangent of dielectric penetration and dielectric loss angles in a wide range of frequencies and temperatures of biocomposites LDPE + x vol.% FS;
5. Investigation of the effect of infrared (IR) and ultraviolet (UV) radiation on the structure and properties of LDPE + x vol.% FS biocomposites;
6. LDPE + x vol.% Study of electrical and mechanical tolerances, volume specific resistances of FS biocomposites depending on the influence of external factors;
7. To study the dependence of the effective values of the surface densities of electric charges on the amount of flake additives;
8. To study the effect of the electric field on the properties of LDPE + x vol.% FS biocomposites;
9. Study of the spectra of thermally stimulated depolarization in biocomposites LDPE + x vol.% FS;
10. Study of the luminescence spectra of bio- and bionanocomposite based on LDPE;
11. Investigation of the possibility of application of LDPE + x vol.% FS biocomposites.

Research methods. In the course of the research, the samples were subjected to ultraviolet (UV) irradiation using a DRSh-500 lamp, while infrared (IR) spectroscopy was performed with a UR-20 spectrophotometer. X-ray phase analysis of the composites was carried out using the Dunker 2D system, which enabled the determination of their degree of crystallinity. The surface morphology of the bio and bio-nanocomposites was investigated using an AFM of the Solver Next brand. Luminescence spectra of the bio and bio-nanocomposites were recorded with a CARU ECLIPSE fluorimeter. The dielectric strength of the composites under electric field was studied using a C-96 kilovoltmeter and an АИИ -70 setup.

Basic theses for defense:

1. Changes in the micro relief of the surfaces LDPE-based composite materials depending on the amount of flake filler.
2. Investigation of the dependence of the tangent of the dielectric permittivity and tangent of dielectric loss angle LDPE + x vol.% FS biocomposites on the frequency, temperature and amount of additives.
3. Investigation of the effect of fish scales filler on the electret properties LDPE + x vol.% FS.
4. Regularities of change LDPE + x vol.% FS biocomposites depending on the amount of filler in the depolarization spectra.
5. Volume dependence of luminescence spectrum filler in LDPE + x vol.% FS biocomposites.
6. Investigation the resistance of scaly composite materials based on LDPE to the effects electric fields and mechanical forces.
7. Determining the applicability LDPE + x vol.% FS biocomposites.

Scientific novelty of the study:

1. For the first time, new biocomposite materials have been produced by adding varying amounts fish scales to LDPE.
2. The study of the micro relief of the surface LDPE + x vol.% FS biocomposites using AFM showed that the defects on the surface gradually disappear as the amount bioadditives in biocomposites increases.
3. An increase in the value of dielectric permittivity of biocomposites was revealed by studying the dielectric permittivity and tangent of dielectric loss angle LDPE + x vol.% FS biocomposites, frequency 100 Hz ÷ 1 MHz and temperature in the intervals 300 ÷ 450 K. Based

on the obtained experimental results, the optical functions of biocomposites were calculated.

The theoretical and practical essence of the research. The investigation of the fundamental properties of LDPE + x% Fs bio-composites has shown that, compared to pure ASPE, these materials exhibit an approximately 18 times increase in average lifetime in the electret state, making them suitable candidates for long-lasting electret applications. Moreover, due to their high dielectric constant and low loss tangent at optimal concentrations of bio-additives, they are promising for use as high-quality insulating materials. Additionally, their luminescent properties under certain compositions and conditions indicate potential applications in the field of optoelectronics.

Approbation and implementation. The main theses and results of the dissertation have been discussed at several international and national scientific conferences and published in their respective proceedings.

Based on the materials of the dissertation, 9 articles were published in high-ranking foreign journals and 5 abstracts in international conference materials, 5 articles in national journals and 3 abstracts in national conferences.

- –V International Scientific-Practical Conference on Modern Problems of Metal Physics (Baku, June 10-11, 2016);
- –I scientific-practical conference of young researchers (Baku, May 05-06, 2017);
- –II International scientific-technical conference on problems of metallurgy and material science (Baku, November 28, 2017);
- –International Scientific and Technical Conference on Topical Issues of Applied Physics and Energy (Sumgayit, May 24-25, 2018);
- –Topical issues of staff training in energy specialties. Republican Scientific Conference (Sumgayit, 30-31 May, 2019);
- –Dedicated to the 97^1 Anniversary of the National Leader of Azerbaijan, Heydar Aliyev IV International Scientific Conference of young researchers (Baku, 24 November, 2020);
- –XXVIII Modern methods of electron, probe microscopy and complementary methods in the study of nanostructures and

nonmaterial EARCHERS (Chernogolovka, 7-10 September, 2020);

–IX International Conference "Crystal Physics and Deformation Behavior of Prospective Materials" (Moscow, 22-26 November, 2021).

The name of the organization conducting the research work. The dissertation work was performed at the Institute of Ecology of the National Aerospace Agency.

The total volume of the dissertation in characters with indication of the volume of its structural sections separately. The dissertation consists of an Introduction (11121 characters), 4 chapters (Chapter I – 42194 characters, Chapter II – 30299 characters, Chapter III – 51002 characters, Chapter IV – 40209 characters), Conclusion (3689 characters), a List of References covering 150 sources, and a List of Abbreviations and Conventions. The total length of the dissertation (without spaces in the text, without tables, without figures, without abbreviations and without a list of references) is 178514 characters.

DISSERTATION CONTENT

In the “Introduction” the importance and relevance of the dissertation topic is justified from both scientific and practical points of view. The purpose of the dissertation and the research task to achieve the goal, the novelty and practical significance of the results obtained, the main provisions of the defense, approbation, information on publications are given in the introductory part.

In I chapter of the dissertation, the data on the characteristics and practical possibilities of low, medium and high density polyethylene used as a matrix in the research work were collected and analyzed, and the use of LDPE in the research work was substantiated. This chapter also explains the structure, properties and applicability of polymers based on polymer-based fillers of various origins and substantiates polymer-based composite materials and the advantages of their use. This chapter also summarizes the work on the development of technology for the production of polymer composites of different compositions, the study of dielectric, electrets and other properties depending on temperature, frequency, radiation, type and amount of fillers and the feasibility of these materials. This chapter explains the selection of fish scales used as filler in the study.

In II chapter of the dissertation is dedicated to solving technology problems. At the beginning of the chapter, the analysis of research works devoted to the study of the structure and properties of fish scales used as fillers in the research work and the properties of the substances obtained on its basis was carried out. The use of this biocomposite as a filler is justified. It has been shown that the parameters of biocomposite materials based on this type of fillers are very reliable and durable.

The method of obtaining bionanocomposite by grinding used fish scales is explained- heating to a certain temperature and powdering in a mortar, mixed with LDPE in accordance with the stoichiometry and by hot pressing method LDPE + x vol.% FS biocomposites, LDPE + x vol.% FS + y vol.% Fe.

The composites were obtained by hot pressing. The LDPE + x vol.% FS was pressurized to 5 MPa at room temperature and the mixture was raised to the melting temperature of ASPE, then the pressure was gradually increased to 15 MPa, under which the sample was kept for 3 min and then cooled in water. As a result, samples with a thickness of 200 microns and a diameter of 4 cm were obtained.

The following sections of the chapter briefly explain the method for investigating electrical resistance and mechanical properties. This chapter also describes the method of aging of biocomposites in the electric field, the method of UV radiation, the method of IR research of biocomposites. The next section provides information on the method for determining the difference in electrical potentials of biocomposites, a method for determining the effective surface density of loads, a method for studying the spectrum of thermally stimulated depolarization of biocomposites, as well as a method for studying the mechanical resistance of biocomposites.

In III chapter of the dissertation summarizes the main experimental results of the work, and at the beginning of this chapter the frequency and temperature dependencies of the dielectric permittivity and tangent dielectric loss angle LDPE + x vol.% FS biocomposites are investigated. The results of the study were given in the temperature range 300 ÷ 380 K in samples with fillers $x = 1; 3; 5; 7; 10$ and 15 vol.% (Figure 1).

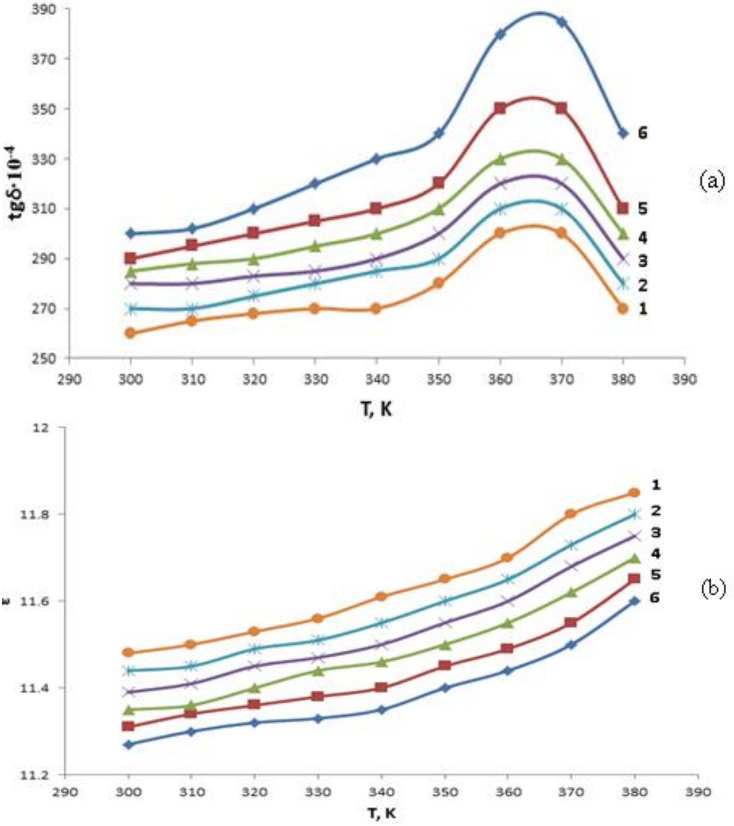

Figure 1. The dependence tangent dielectric loss angle (a) and dielectric permittivity (b) versus temperature biocomposites LDPE + *x* vol.% FS: 1 – *x* = 1; 2 – *x* = 3; 3 – *x* = 5; 4 – *x* = 7; 5 – *x* = 10; 6 – *x* = 15.

The results indicate that with increasing temperature, molecular mobility intensifies, allowing dipole structures to polarize more easily, which leads to an increase in the loss tangent $tg\delta(T)$. However, beyond a certain temperature (typically around 360–370 K), $tg\delta(T)$ reaches a maximum and then begins to decrease. This phenomenon is associated with the relaxation behavior of the material and signifies a transition of the structure to a locally stable state.

The addition of Fs to the composite not only increases the dielectric permittivity but also leads to an increase in the loss tangent $tg\delta(T)$. This behavior is attributed to the high polarization capability of Fs and the incorporation of a greater number of dipoles within the structure. However, as the Fs content varies, structural stabilization within the system is observed, resulting in a decrease in *tgδ*(*T*). This phenomenon is explained by enhanced interfacial compatibility and a reduction in the system's energy.[1]

It is evident that the initial increase followed by a decrease in *tgδ*(*T*) is indicative of a typical relaxation process, which holds significant importance for the use of composite materials in electromechanical applications.

It was found that as the amount of flocculent filler in the biocomposite increases, the dielectric permittivity increases, which is consistent with the Maxwell-Wagner theory. In other words, the change in the parameters of the studied biocomposites is associated with polarization processes occurring at the interface between the polymer matrix and the phases of the bio-filler. These processes are the result of the concentration of the iso-energetic surface density, the amount of filler, and the formation of deep traps.

The next section reflects the results of the study of the dependence of the mechanical strength of biocomposites with a volume of x = 1; 3; 5; 7; 10vol.% FS of the amount of filler (Figure 2).

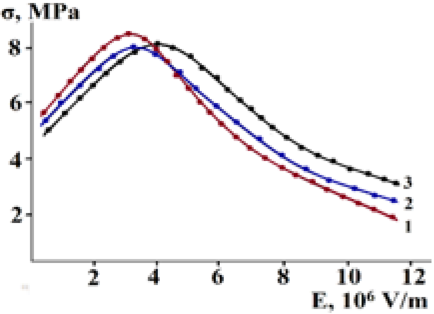

Figure 2. Dependence of the mechanical strength of LDPE + x vol% FS composites on the intensity of the external electric field: 1 – *x*=1, 2 – *x*=3, 3 – *x*=5

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[1] Gojayev, E.M. The dielectric properties and electrical conductivity of LDPE modified by fillers of biological origin / E.M.Gojayev, Sh.V.Aliyeva, X.S.Khalilova [et al.] // International Journal of Modern Physics B, – 2019. Vol. 33, No. 26, – pp.1950309-1-10

The study of the dependence of the mechanical strength of composites on the electric field strength was carried out and it was determined that an increase in the mechanical strength first to a certain value, and then a decrease can be explained as follows. This type of dependence σ(ε) is associated with a decrease in the curves around the slope approaching the maximum, as well as with forced elastic deformation in the samples. By its nature, forced elastic deformation, like plastic deformation, is the result of the sliding of one layer of material relative to another.

This is due to the beginning of the formation of thinned areas in the area corresponding to the maximum deformation. In the samples, where the maximum in the dependences σ(ε) is not observed, deformation occurs without the formation of thin spots. In the area of decreasing tension, the formation of thin spots occurs, and at the end of the decrease in stress, the formation of thin spots ends.

The tension in the smooth part of the tensile curve remains practically constant. At this stage of deformation, the cross-sectional area of the thinned areas changes little, and the elongation of the specimen is due to the spread of forced elastic deformation to adjacent parts of the specimen. During this process, the length of the thinned areas increases. The decrease in σ(ε) is the result of a decrease in the width of the sample at the thinned points. Of course, the deformation force decreases with decreasing cross-section of the sample, but if we divide the value of this reduction by the initial cross-section, then the tension will decrease [2].

In the next section of the dissertation, the results of a study of the dependence of the dielectric permittivity and the tangent dielectric loss angle on the frequency and amount of bio-filler of biocomposites with a volume of 3; 5; 7; 10 and 15 vol.% FS.

The studies were conducted out in the frequency range $10^{-2} \div 10^{-3}$ kHz. It was found that, as shown in Figure 3, the dielectric permittivity LDPE + x vol.% FS composites first increases in all studied samples with increasing frequency and decreases exponentially at high frequencies after reaching the maximum value.

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[2] Səlimova V.V. Biokompozitlərin mexaniki möhkəmlikləri // Energetika ixtisasları üzrə kadr hazırlığının aktual məsələləri. Respublika Elmi Konfransının materialları, – Sumqayıt: – 30-31 may, – 2019, – s. 103-105.

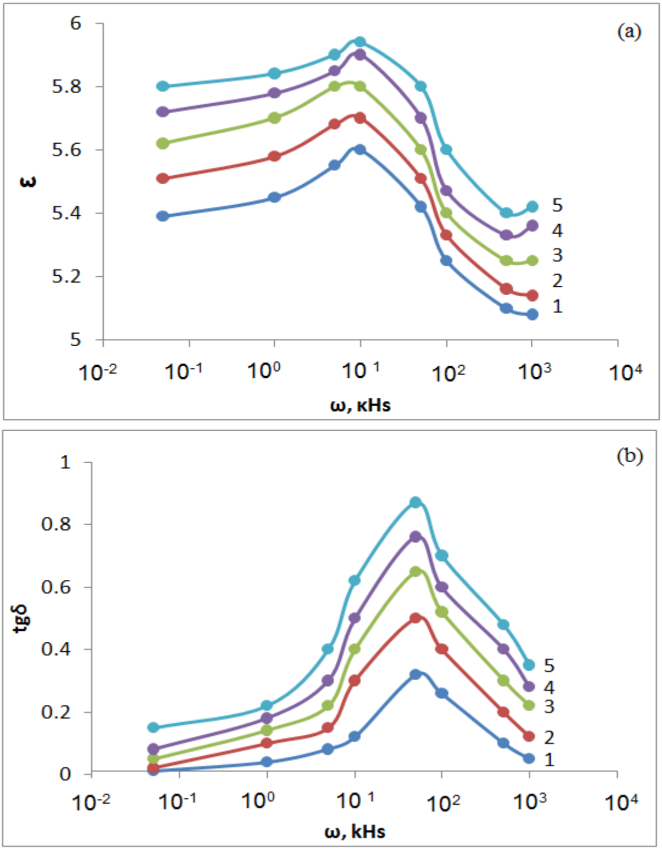


Figure 3. The dependence of dielectric permittivity (a) and tangent dielectric loss angle (b) on the frequency of biocomposites LDPE + x vol.% FS: 1 – *x*=3; 2 – *x*=5; 3 – *x*=7; 4 – *x*=10; 5 – *x*=15

An increase in dielectric permittivity also occurs with an increase in the amount of biodegradable composite. It should be noted that the studied composites show a slight increase in the dielectric permittivity at low frequencies and a sharp decrease at high frequencies. Dielectric losses initially rise sharply as they increase, reaching a maximum at 50 kHz and decreasing.

As the amount of FS additive increases in the graph, the dielectric constant of the material rises, indicating its enhanced ability to store electrical energy and increased polarization capability.

The common frequency spectrum of dielectric loss is due to the fact that the contribution of different types of dipole orientation-polarization to the total dispersion of the complex dielectric permittivity is different. This is the result of the presence of polar radicals and polar groups of plasticizer molecules in the studied compounds. In addition, the inclusion of modifiers in the polymer reduces its viscosity, weakens the intermolecular interactions, and the dipole orientation of polar groups and radicals alters the relaxation time of the polarization process. The results show that an increase in the concentration of fish scales fillers in the composite causes the relaxation frequencies to shift to the lower frequency range or the width of the complex dielectric permittivity to change.

Based on the obtained dielectric permittivity and frequency characteristic of the dielectric loss angle, their optical functions were determined: real and imaginary refractive index, absorption coefficient, reflection coefficient, real and imaginary part of optical electrical conductivity. The results are quite good.

In the next section, the effect of FS on LDPE in the dendritic process is described. Analysis of the obtained results shows that the dendritic resistance of the composites is high at low values of the filler. This can be justified by the fact that the increase in the dendritic resistance of the composite is due to the fact that the addition of flakes to its composition fills the gaps inside the composite. The chemical nature, concentration, physical and electro physical properties of the filler included in the composite have a significant impact on the dendritic resistance of crystalline polymers.

In general, the dendritic process in polymer composites occurs in two stages. In the first stage, dendrites are formed, a process that occurs from the moment the sample is exposed to an electric field until the moment the dendrites form. The formation of dendrites is visually recorded under a microscope. Dendrites appear in the form of an anal canal 10 ÷ 20 mkm long near the needle-like electrode. If the intensity of the electric field at the tip of the electrode is greater than the electrical resistance of the composite, then the perforation of the composite occurs instantly. If the stress applied to the composite is not sufficient to pierce it, then there is an induction period between the formation of the dendrites and the moment of stress transfer. During this time, the changes inside the composite increase, and after a certain period of time, it becomes perforated. In our case, the period from the development of dendrites to the complete perforation of the composite consists of the dendrites channel for the formation of dendrites and the development of dendrites. The duration of dendritic formation is maintained by increasing the amplitude of the 50 Hz voltage and the test temperature. In addition to the above, we can say that at present there are no experimental results and considerations that allow to fully clarify the causes of the emergence and development of dendrites. In polymer composites, different physical properties and different composition of fillers can be achieved to prevent or weaken the dendritic process.

The results of the study of spectra of thermo stimulated depolarization performed on samples of pure LDPE, containing 1, 3, 5, 7 vol.% FS (Figure 4).

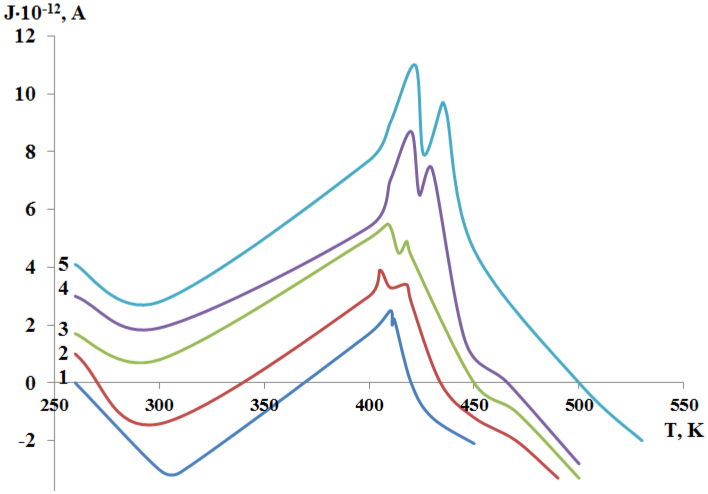


Figure 4. The dependence depolarization spectra versus temperature biocomposites LDPE + x vol.% FS: 1 – *x*=0; 2 – *x*=1; 3 – *x*=3; 4 – *x*=5; 5 – *x*=7

It has been found that TSD currents can be both positive and negative, depending on how different the dielectric permittivity of the polyethylene and the filler and the specific electrical conductivity are. This has been confirmed in research. The high-temperature parts of the TSD current spectra have a complex structure. Thus, depending on the amount of bio-filler, the maximums observed at high temperatures and their amplitudes change, as a result of which it is possible to control and control them by changing the amount of filler.

As a result, by varying the filler content, it is possible to control and manage these properties. The peaks are concentrated around 400–450 K, indicating the release of thermally activated traps or dipole structures within the material.

With an increase in the FS content from x = 0 to x = 5 and beyond, both the intensity and the temperature position of the TSD peaks change. This suggests that Fs enhances the internal polarization capability of the material as well as the number of energy traps.

The influence of external factors on the electrical properties of biocomposites has been studied. Fillers embedded in a polymer significantly affect their dielectric properties, including dielectric losses. It is not enough to use composite materials as dielectrics and to know only the dielectric losses of the molecular structure of the thermally acting polymer. For this, it is necessary to study their dependence on temperature, electric field and other factors. For this, the temperature dependence of the dielectric loss angles of biocomposites containing small amounts of bio-fillers was studied (Figure 4). At the same time, a plot of the dielectric loss tangent versus the number of attached flakes was plotted (Figure 5).

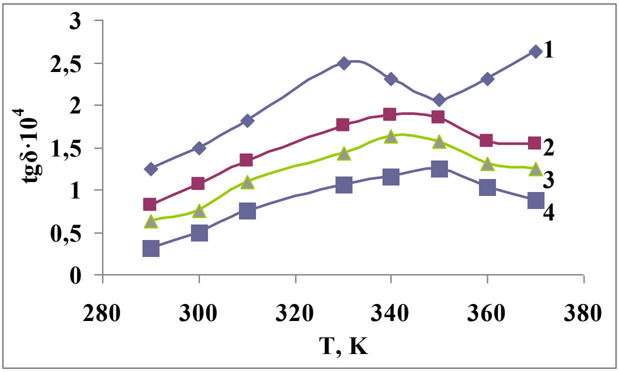


Figure 5. The effect of FS on the temperature dependence of the *tgδ*(*T*) of the dielectric loss angle

Weak maxima in a crystallizing polar polymer due to the dependence of *tgδ*(*T*) on the presence of weak additive polar groups. As seen in Figures 5 and 6, an increase in *tgδ*(*T*)corresponds to a large temperature value. In this case, the crystallization kinetics changes and the arrangement of macromolecules become dense. As a result, molecular interactions occur in polymer chains. The movement of the kinematic segments becomes more difficult, that is, some of the additive polymer radicals fall into the region of the crystal and reduce their mobility.

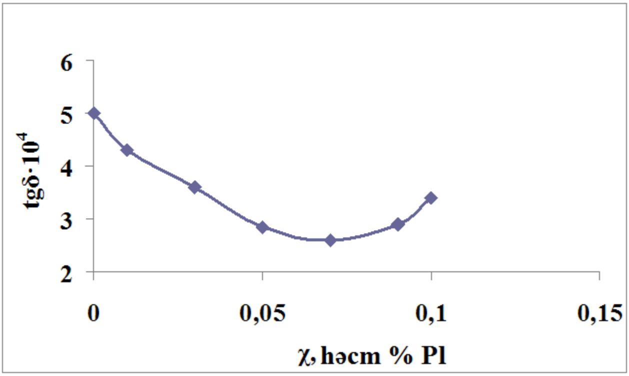
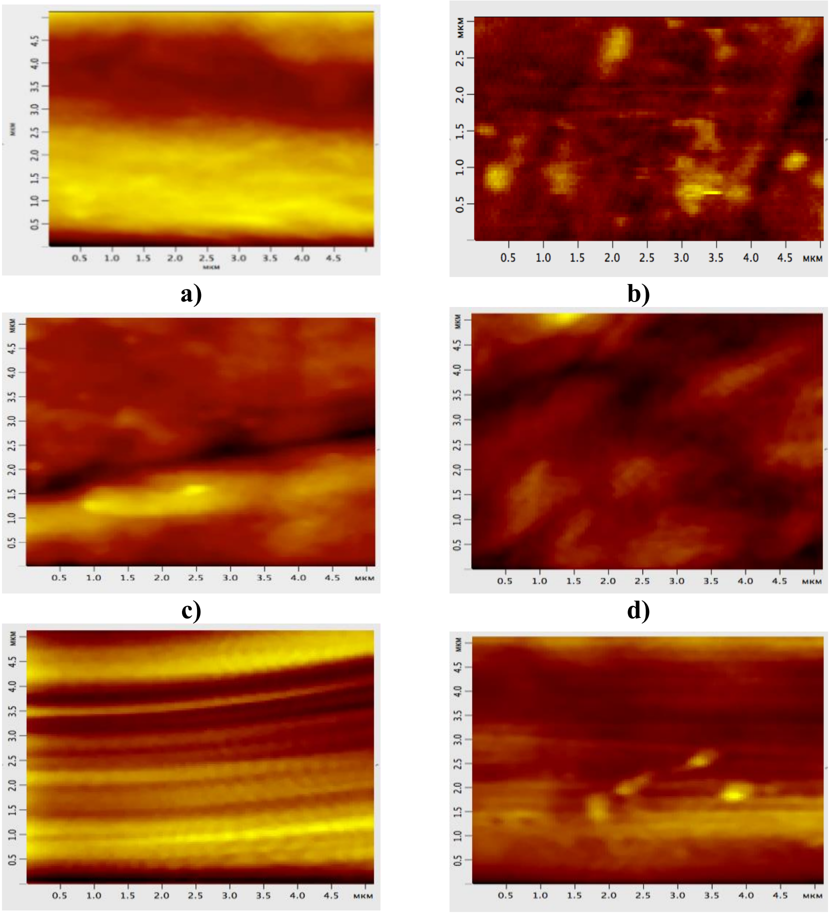


Figure 6. LDPE +x vol.% FS The dependence of the dielectric loss tangent of biocomposites on the content of scales

In IV chapter of the dissertation the results of the study of the microrelief of surface structures in 2D and 3D modes using AFM bionanocomposites LDPE +x vol.% FS and LDPE +y vol.% Fe.

The research was carried out on samples with fillers *x* = 1; 5; 7; 10; 15; 20; 30 and 50 vol.% FS. Some of these results were obtained with composites taken from different parts of the same sample. Analysis of the results obtained shows that the surface of the samples becomes smoother as the amount of RF filler in the composites increases (Figure 7, 8).

The composite containing *x* = 5 vol% FS exhibits well-defined, regular stripe-like surface structures, indicative of the uniform dispersion of Fs within the polymer matrix and the nucleation of primary crystalline domains. This structural organization contributes to enhanced mechanical stability of the surface as well as improved overall structural integrity of the composite.



a) b) c) d) e) f)

Figure 7. 2D description of the microrelief of the surface of bionanocomposites such as LDPE + x vol.% FS + y vol.% Fe in an AFM: a) *x*=20, *y*=20; b) *x*=15, *y*=15; c) *x*=10, *y*=10; d) *x*=3, *y*=7; e) *x*=7, *y*=3; f) *x*=5, *y*=5

At a higher filler concentration of *x* = 7 vol% FS the composite surface demonstrates increased density and noticeable structural symmetry. This concentration is identified as optimal, providing a favorable balance of crystallinity, mechanical strength, and thermal stability within the composite matrix.

In the next section of the chapter, an X-ray phase analysis of the objects of study was carried out and, based on the results obtained, the number of phases included in the biocomposites was determined.

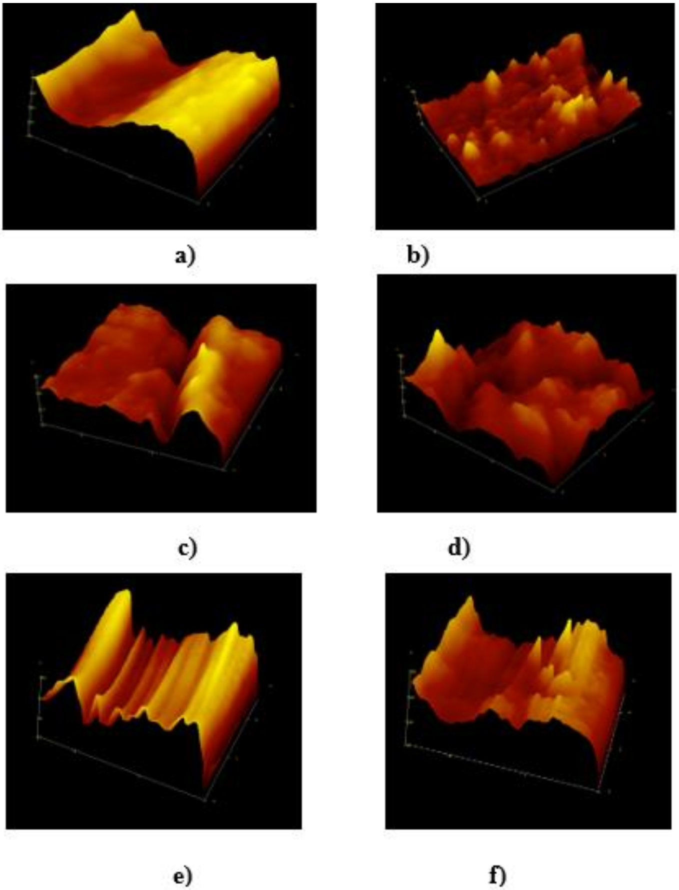


Figure 8. 3D description of the microrelief of the surface of bi-onanocomposites such as LDPE + x vol.% FS + y vol.% Fe in an AFM: a) x=20, y=20; b) x=15, y=15; c) x=10, y=10; d) x=3, y=7; e) x=7, y=3; f) x=5, y=5.

The relationship between the intensity of reflexes observed in X-ray diffraction patterns, the crystallization rate and the amount of filler can be explained as follows. Since fish scales are used as a filler in the production of composites, they cannot penetrate into the crystalline region of the composite; they only penetrate into the amorphous part of the material. As is known, the intensity of X-ray reflections is mainly determined by the densities of the polymer and crystal fields. The crystalline part of the polymer is impossible from the point of view of the inclusion of extraneous atoms. Consequently, extraneous atoms can form in the amorphous polymer field.

It is well established that the relationship between the densities of the amorphous and crystalline regions and the intensity of X-ray scattering is described by the following mathematical expression.

$$J \sim A(\rho_k + \rho_a)^2$$

In this equation, A is a constant, while ρ_k and ρ_a represent the densities of the crystalline and amorphous regions of the polymer respectively.

The next section of the fourth chapter describes the results of studying the IR absorption spectrum of composites. An analysis of the results obtained showed that a comparison of the obtained spectra shows that the inclusion of scale modifiers and metal nanoparticles in low-density polyethylene does not lead to the formation of new, sharp lines of the absorption spectrum, bands, does not change the shape of the IR absorption spectrum. This means that the modifiers we use and their amounts are well compatible with low-density polyethylene.

As can be seen from Figure 9, the maximum observed at very high frequencies (3857 cm^{-1}) corresponds to the bands of free stretching vibrations, and does not participate in hydrogen bonds.

O-H ... O, bands formed by hydrogen bonds are clearly pronounced in the IR spectrum. There are wide bands in the range 3000-2800 cm^{-1}, which is associated with the formation of a large number of small structures of different compositions. The position of the hydroxyl bands depends on the position of the hydrogen bands, and the frequency can be reduced to 2400 cm^{-1}, especially for strong bonds. Note that the intense bands observed in the range 2800-3000 cm^{-1} correspond to stretching vibrations of hydrogen CH, CH_2 and CH_3. Hydrogen atoms associated with carbon sp2-hybridization have a higher frequency of stretching vibrations (3000 ÷ 2800 cm^{-1}). Here it is necessary to single out the CH groups at frequencies of 2928,407 cm^{-1} and 2853.4 cm^{-1}. Compounds containing a vinyl group -CH = CH_2 have every 2 bands in the spectrum. This fragment also contains the higher valence bands of the acidic ≡C-H C atoms. The deformation vibrations have frequencies much less than the frequencies of valent vibrations of the same groups.

The exception is the bands of primary amino groups. Of the deformation vibrations of other hydrogen-containing groups, the antisymmet-

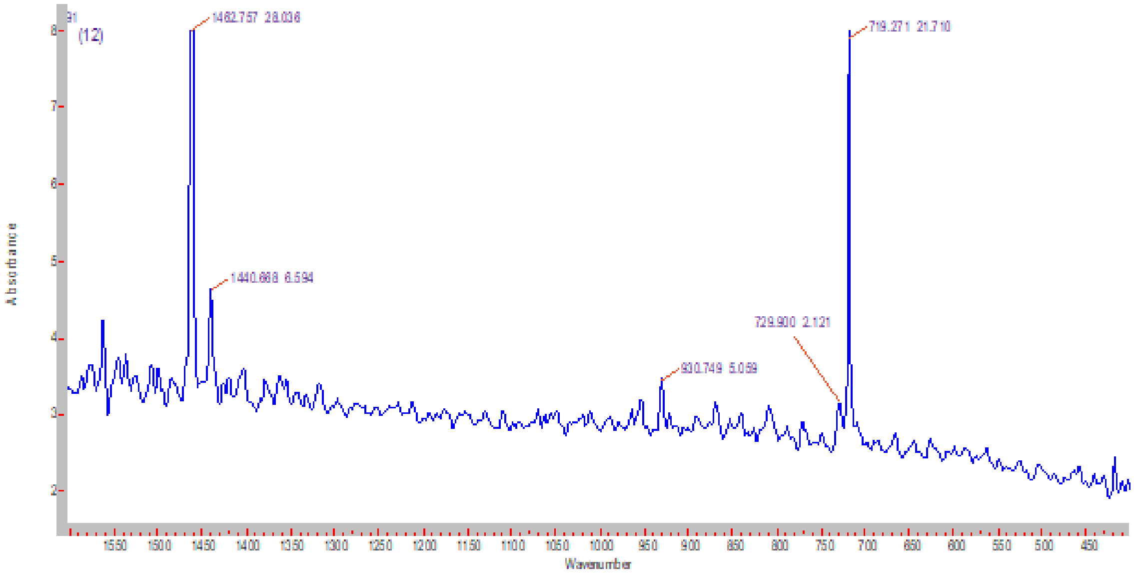


Figure 9. IR absorption spectra biocomposites of LDPE + x vol.% FS.

ric and symmetric deformation vibrations of the methyl groups (1471-1462 cm^{-1} and 1367-1352 cm^{-1}) of the scissors vibration of the methyl group are of greatest importance for structural analysis. The absorption band at 757 cm^{-1} is responsible for vibrations of methylene groups. In the C=N, O=N, C≡N and O=N groupings, the reduced masses are close but the force constants differ signifiantly depending on the number of bonded electrons and reveal a clearly pronounced dependence on conjugation effects leading to a decrease in the numerical values of the characteristic frequencies of conjugate multiples connections compared to isolated. The intensity of the corresponding bands in the IR spectra depends on the polarity of these bonds. The polar groups of different electronegativity atoms (C=O, N=O, S=O, etc.) are characterized by a very high intensity of stretching vibration bands, which, according to this feature, are easily detected in the spectra. But double and triple bonds between identical atoms (C-C, N-N, C-C) usually have medium and weak stretching vibration bands, and in symmetric structures such vibrations are generally inactive in the IR spectra.[3]

The IR spectra of the above biocomposites and bionanocomposite containing a flake filler and Fe have been investigated. In the IR spectrum of these bio- and bionanocomposite (1470 ÷ 1450 cm^{-1} and 750 ÷ 700 cm^{-1}) bands are observed in the frequency range. The experimental results obtained show that the optimal use of the additives we use is superior to other stabilizers, that is, the additives of biological origin we use allow us to obtain stronger materials with an optimal and strong structure.

The next section of the chapter examines the effect of UV radiation on the dielectric properties of biocomposites.

The study was carried out on samples exposed to irradiation for 70 and 100 hours and containing 3, 5, 7, 10 and 15 vol.% FS.

It was found that after UV radiation the dielectric permittivity increases, and the tangent dielectric loss angle decreases. This is due to the formation of new dielectric losses on the temperature dependence of the tangent of the loss angle of UV radiation of polyethylene.

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[3] Gojayev, E.M. IR absorption spectra of high-pressure polyethylene modified by fish scales / E.M.Gojayev, V.V.Salimova, S.H.Jabarov // Journal of World Scientific, Physics, – 2019. №33(33), – pp. 1950412-1-6.

However, under the same conditions, a very small change in the dielectric properties of the samples modified by the flakes is observed. In other words, the dielectric permittivity increases monotonically, but the tan δ(T) dependence takes on a very small value in the transition region. In addition, the addition of scales slows down the efficient oxidation process in the LDPE. Thus, the main losses of polyethylene occur during the relaxation of carbonyl groups. On the tan δ(T) dependence, new dielectric losses appear in the temperature range 320 ÷ 340 K after UV radiation. The loss of the dipole-segmental maximum of the tan δ(T) dependence increases and shifts towards higher temperatures.

Finally, it should be noted that if the degree of polymer crystallization does not exceed 70%, then this type of material exhibits maxima characteristic of amorphous substances. In addition, the observed maxima on the tan δ(T) dependence in crystalline substances are due to structural changes, local displacements of macromolecules in the regions of the crystal, and other factors. The losses observed in the maximum regions, which are important for determining the mechanism of molecular motion, depend on the degree of crystallization of the materials. [4,5]

In the last section of the chapter, a study of the luminescence spectrum in bionanocomposite LDPE + x vol.% FS and LDPE + x% FS + y vol.% Fe is given.

Luminescence spectra were studied in composites HDPE + x vol.% FS. The luminescence effect was also found in composites LDPE +7 vol.% FS and LDPE +7 vol.% FS+3 vol.% Fe (Fig.10). It was revealed that in the composite LDPE +7 vol.% FS, when excited with a wavelength of 466.06 nm, the intensity of the luminescence peak is 32 units of atomic energy, and when excited with a wavelength of 501.94 nm, the luminescence intensity is 9.2 units of atomic energy.

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[4] Gojayev, E.M. The effect of UV irradiation on the dielectric properties of bionano¬composite with fillers of biological origin and metal nanoparticles / E.M.Gojayev, Sh.V.Aliyeva, V.V.Salimova [et al.] // Journal of World Scientific, Physics, – 2020. Vol. 34, №17, – pp. 2050186-(1-13).

[5] Gojayev, E.M. The Influence of UV Irradiation on Dielectric Properties of Biocomposites / E.M.Gojayev, Sh.V.Aliyeva, V.V.Salimova [et al.] // Surface Engineering and Applied Electrohemistry, – 2020. Vol. 56, №6, – pp. 740-745.

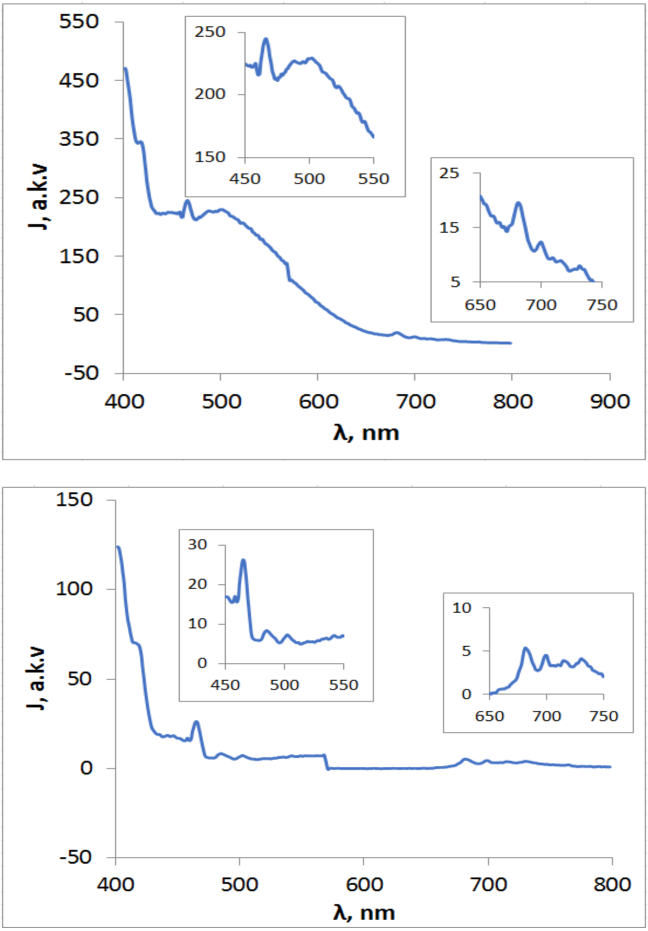

Figure 10. Luminescence spectrum of excitation with a wavelength of 342 nm of bionanocomposite LDPE + 7 vol.% FS (a) and LDPE + 7 vol.% FS + 3 vol.% Fe (b).

And in the bionanocomposite LDPE +7 vol.% FS +3 vol.% Fe when excited with a wavelength of 465 nm, the luminescence intensity is observed as a single relatively pronounced peak with an intensity of 20 atomic energy units. Comparing the intensities of the obtained luminescence peaks with the intensities of the excitation signals, it can be concluded that pronounced luminescence spectra are obtained in some of the studied biocomposites.

This allows us to conclude that the luminescent property, which is of practical importance, can be achieved by changing the composition of the studied biocomposites and bionanocomposite and choosing the appropriate excitation.

CONCLUSIONS

1. For the first time, LDPE + x vol% FS bio-composites were synthesized by incorporating fish scale waste into low-density polyethylene, and their electrophysical properties were systematically investigated.

2. It was established that the incorporation of fish scales into low-density polyethylene (LDPE)-based bio-composites significantly improves their electrical and dielectric properties. Specifically:

- –In the sample containing 1 vol% fish scales, the specific electrical resistivity increased by approximately ~45%, reaching ~1.6 × 10^9 Ω·cm.
- –In the sample with 0.7 vol% filler, the dielectric loss tangent (tg δ) decreased to 0.008, indicating minimal energy loss.
- –With increasing temperature, a general decrease in resistivity was observed in all samples; however, in the composite containing 1 vol% filler, the resistivity remained stable, indicating the formation of an optimal structure.
- –These results demonstrate that LDPE bio-composites containing 0.7–1 vol% fish scales possess optimal electrical insulation properties and are promising for application in energy systems.

3. The influence of infrared (IR) spectroscopy and ultraviolet (UV) irradiation on the structure of bio-composites modified with fish scales was investigated. It was determined that the optimal amount of fish scales and iron nanoparticles in the bio-composites significantly affects the dielectric properties and contributes to the stabilization of the structural characteristics of the polymer matrix

4.For the first time, the effect of ultraviolet (UV) irradiation of various durations (50–100 hours) on the dielectric properties of bio-composites and bio-nanocomposites based on an ASPE matrix, modified with 3–15 vol% fish scales and 1 vol% Fe nanoparticles, was systematically investigated. It was shown that UV irradiation alters the structure of the polymer matrix, leading to an increase in dielectric permittivity. The dielectric loss tangent, particularly in the amorphous–crystalline transition region, exhibits a decreasing trend. These changes are associated with the interaction at the polymer–filler interfacial phases and the dynamics of radical oxidation processes.

5. Based on the investigation of thermally stimulated depolarization (TSD) spectra, the activation energies of the bio-composites were calculated. It was determined that by varying the filler content, the high-temperature components of the TSD currents and their amplitudes can be controlled. This provides a basis for optimizing the electrical properties of the material. Furthermore, the study of the charge retention time as a function of surface charge density revealed that the bio-composite containing 3 vol% FS exhibits a long-lived electret behavior, making it a promising material for electret applications.

6. Analysis of 2D and 3D surface images obtained via Atomic Force Microscopy (AFM) for LDPE + FS (x = 1–50 vol%) and LDPE + FS + Fe (x = y = 5–20 vol%) bio-nanocomposites revealed that in the range of x = 5–7 vol% Pl, optimal surface structuring, uniformity, and periodicity were observed. The surface roughness at the microscopic level was found to vary between 45 and 130 nm, confirming that fish scales, as effective bio-fillers, have the ability to optimize the surface morphology of the composite matrix. X-ray phase analysis of the composites further demonstrated that increasing the content of fish scale and Fe particles leads to a rise in the degree of crystallinity from 42% to 68%, proving the influence of these bio-fillers on the formation of the crystalline structure within the composites.

7. For the first time, the luminescent properties of bio-nanocomposites based on an LDPE matrix, modified with fish scales (FS) and doped with Fe nanoparticles, were systematically investigated. It was demonstrated that the sample containing 7 vol% FS and 3 vol% Fe exhibits intense luminescence peaks within the wavelength range of 465–501 nm, with peak intensities of 32 and 9.2 energy units, respectively. This indicates that the incorporation of metal nanoparticles into the bio-nanocomposite matrix enables the development of optically active luminescent materials with high sensitivity.

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