St. Petersburg State Polytechnical University Journal. Physics and Mathematics. 2025. Vol. 18. No. 2
Научно-технические ведомости СПбГПУ. Физико-математические науки. 18 (2) 2025

Original article
UDC 538.915
DOI: https://doi.org/10.18721/JPM.18208

THE EFFECT OF PLA-BASED COMPOSITE MATERIAL HISTORY ON ITS ELECTRET PROPERTIES

I. A. Zagidullina [1], **A. A. Guzhova** [1], **E. A. Karulina** [2] ✉, **S. M. Kulemina** [2]
[1] Kazan National Research Technological University, the Republic of Tatarstan, Kazan, Russia;
[2] Herzen State Pedagogical University of Russia, St. Petersburg, Russia
✉ karulina@mail.ru

Abstract. This study reveals the effects of manufacturing methods and composition of the films based on polylactic acid (PLA) and fine fillers of different nature (aerosil, white silica and starch) and concentration on the electret properties (EP). The methods were solution casting (I) and compression molding (II). PLA films manufactured via method II exhibited a significantly higher electret effect when subjected to unipolar corona discharge than the films obtained by method I. The difference in the EP was proved to be attributed to the presence of polar liquid molecules (trichloromethane, water) within the samples produced using method I enhancing the electrical conductivity of the samples. The samples showed a tendency for the EP parameters to increase when the dispersed particles of fine fillers were added to the pure PLA. An explanation for this phenomenon has been proposed. The composition of PLA with 4% white silica had the best electret properties.

Keywords: electret, polylactic acid, polymer, dispersed filler, white silica, aerosil, starch

Funding: The research was carried out using a grant from Academy of Sciences of the Republic of Tatarstan provided to young candidates of science (postdocs) for the purpose of presentation of a doctoral thesis, carrying out research work, as well as performing labor functions in scientific and educational organizations of the Republic of Tatarstan within the framework of the State Program “Scientific and Technological Development of the Republic of Tatarstan” (Agreement No. 114/2024-PD).

For citation: Zagidullina I. A., Guzhova A. A., Karulina E. A., Kulemina S. M., The effect of PLA-based composite material history on its electret properties, St. Petersburg State Polytechnical University Journal. Physics and Mathematics. 18 (2) (2025) 82–92. DOI: https://doi.org/10.18721/JPM.18208

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Научная статья
УДК 538.915
DOI: https://doi.org/10.18721/JPM.18208

ВЛИЯНИЕ ПРЕДЫСТОРИИ КОМПОЗИЦИОННЫХ МАТЕРИАЛОВ НА ОСНОВЕ ПОЛИЛАКТИДА НА ИХ ЭЛЕКТРЕТНЫЕ СВОЙСТВА

И. А. Загидуллина [1], **А. А. Гужова** [1], **Е. А. Карулина** [2] ✉, **С. М. Кулемина** [2]
[1] Казанский национальный исследовательский технологический университет,
г. Казань, Республика Татарстан, Россия
[2] Российский государственный педагогический университет им. А. И. Герцена,
Санкт-Петербург, Россия
✉ karulina@mail.ru

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Аннотация. Исследовано влияние метода изготовления и состава пленок из композиций полилактида (ПЛА) с дисперсными наполнителями (белая сажа, аэросил и крахмал в разной концентрации) на их электретные свойства (ЭС). Это методы полива из раствора (I) и прессования (II). Установлено, что пленки ПЛА, полученные методом II, гораздо лучше электретируются в униполярном коронном разряде и сохраняют ЭС, чем изготовленные методом I. Доказано, что разница в ЭС различных образцов обусловлена содержанием в их объеме полярной жидкости (хлороформ, вода), молекулы которой повышают электропроводность объектов. У образцов прослеживалась тенденция повышения параметров ЭС при добавлении дисперсных частиц к чистому ПЛА. Предложено объяснение этого эффекта. Лучшими ЭС обладала композиция ПЛА с 4 %-м содержанием белой сажи.

Ключевые слова: электрет, полилактид, полимер, дисперсный наполнитель, белая сажа, аэросил, крахмал

Финансирование: Работа выполнена за счет гранта Академии наук Республики Татарстан, предоставленного молодым кандидатам наук (постдокторантам) с целью защиты докторской диссертации, выполнения научно-исследовательских работ, а также выполнения трудовых функций в научных и образовательных организациях Республики Татарстан в рамках Государственной программы Республики Татарстан «Научно-технологическое развитие Республики Татарстан» (Соглашение №114/2024-ПД).

Для цитирования: Загидуллина И. А., Гужова А. А., Карулина Е. А., Кулемина С. М. Влияние предыстории композиционных материалов на основе полилактида на их электретные свойства // Научно-технические ведомости СПбГПУ. Физико-математические науки. 2025. Т. 18. № 2. С. 82–92. DOI: https://doi.org/10.18721/JPM.18208

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Introduction

Polylactic acid (PLA) is an aliphatic polyester derived from renewable sources such as corn starch or sugarcane. It has a number of valuable properties that determine its wide application: biodegradability, non-toxicity, relatively high tensile strength and elasticity modulus, easy processing by injection molding and extrusion, low hydrophilicity, ability to be modified physically and chemically, etc. [1]. It is a sustainable material that tends to reduce negative environmental impact, creating an alternative to traditional synthetic polymers. Due to this property, an application field of PLA is constantly expanding, which entails the growth of its global production volume. Traditional areas of PLA application are packaging (bags, containers, disposable tableware), 3D printing, medicine (surgical threads and pins, medical masks), textile industry (fibers and fabrics), agriculture (covering materials in the form of nonwoven fabrics), filtration and others [2 – 4]. Over the past decade PLA has been considered as a promising dielectric material with piezoelectric and electret properties for the production of elements of wearable electronics, highly sensitive sensors, ultrasonic transducers, sensors, etc. [5 – 7].

Electrets are dielectric materials that possess dipole polarization or quasi-constant electric charge ("quasi-constant" means that the lifetime of an electret is much longer than the time during which it is applied or studied) [8].

Electrets are often made of polymers or polymer composites, the advantages of which are ease of processing and manufacturing, although they have lower temperature stability compared to inorganic (ceramic) electret materials. The scientific world is taking a strong interest in study of the electret properties of PLA and its composites. This is due to the growing requirements for environmental friendliness and protection, as well as the need to produce biocompatible sensors and transducers. PLA is described as a promising dielectric material with electret properties, although data on their quality and stability vary. Research has shown that the charge stability of PLA depends on the form (L- or D-stereoisomer), supramolecular structure (degree of crystallinity), environmental conditions (temperature, humidity, pressure), the presence of impurities and chemical additives in its composition, geometric dimensions of samples, etc. [9 – 15].

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At the same time, attention is drawn to the fact that PLA-based electrets are produced by different manufacturers in various forms (films, sheets, fibers, coatings) and using all kinds of methods (pressing, extrusion, melt-blown, 3D printing, solution casting). Various methods of charging (thermal polarization, corona discharge) are also applied. It is clear that it is not worth comparing the values of electret characteristics of polymer samples obtained by different authors, because the prehistory of samples excludes the possibility of adequate comparison of the level of electret charge, their behavior during storage, research and operation.

The goal of the present study was to find the optimal method for preparing the PLA composition and the optimal nature of the filler (and its concentration) in order to obtain an electret with the best properties.

To achieve this goal the task was to compare the values and stability of electret parameters of films made of PLA and its compositions with fine fillers by different manufacturing methods.

Materials and methods

The subjects of research were polylactic acid and different fine fillers: pyrogenic silicon dioxide (aerosil), precipitated silicon dioxide (white silica) and starch. Their chemical structure, grades and properties are presented in Table 1.

Table 1

Characteristics of the materials under study

Material	Grade, standard	Chemical formula	Parameter value	
			ρ, g/cm^3	D, nm
Polylactic acid	Nature Works 40320	$(C_3H_4O_2)_n$	1.24	–
Aerosil	A-175, GOST 14922-77	SiO_2	2.15	5–40
White silica	BS-120, GOST18307-78			19–27
Starch	Corn starch GOST 32159-2013	$(C_6H_{10}O_5)_n$	1.5	$(5–20)\cdot 10^3$
Trichloromethane	GOST TU 20015-88	$CHCl_3$	1.483	–

Notations: ρ is the density, D is the particle size.

Footnote: Polylactic acid has the melting temperature T_m = 155 – 170°C, glass transition temperature T_g = 54 – 58 °C.

The polymer and the fillers were mixed in a Brabender Mixer W 50 EHT at 180°C for 300 s at a roller speed of 150 rpm. The fillers were brought in the polymer in the ratio of 2, 4 and 6 wt. %. After that, films were produced by two methods:

Method I. Films 50 μm thick were manufactured by solution casting. For this purpose, PLA compositions were dissolved in the chloroform in the ratio of 1:20 and thoroughly stirred for 60 min; then they were left for 24 hrs until completely dissolved. Then, after another thorough stirring for 10 min, the 5 % (by mass) solution of PLA and its compositions were poured into specially prepared glass molds, dried under a hood for 1 hour, then drying was carried out under ambient conditions for three days, after that the films were removed from the substrates.

Method II. Samples in the form of films 500 μm thick were produced on a Gotech GT-7014-H10C press in accordance with GOST 12019-66. Compression molding parameters were as follows:

the heating temperature .... 200 °C,
the pressure .... 35 MPa,
the preheating time .... 20 min,
the pressure holding time ... 5 min,
the cooling time .... 2 min.

The films were charged with a two-electrode corona discharge film processing unit (Fig. 1). Processing parameters were as follows:

the distance between the sample and the electrode ... 20 mm,
the polarization voltage .... 30 kV,
the polarization time .... 30 s.

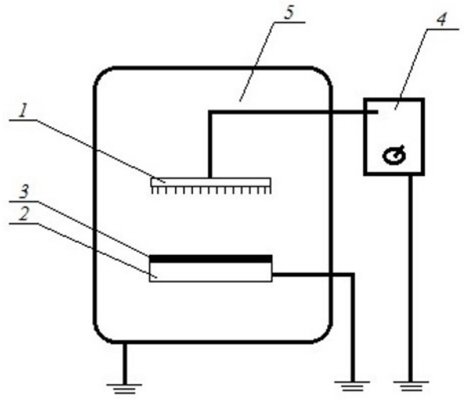

Fig. 1. Two-electrode charging unit with unipolar corona discharge:
a corona electrode (*1*), a grounded electrode (*2*), a sample (*3*), a power supply of the corona electrode (*4*), a heating chamber (*5*)

The electret properties of the samples, such as the surface potential V_s, the electric field strength E and the effective surface charge density σ_{eff} were measured with the IPEP-1 fieldmeter.

The FTIR spectrometer Agilent Cary 600 Series was used to record the infrared (IR) spectra of the samples.

Results and discussion

At the first stage of the study, PLA films were made from solution (casting) and melt (compression molding) of the polymer followed by the charging of the films in negative corona discharge to produce electrets. The results were quite unexpected. The PLA films obtained by solution casting had the following characteristics one hour after being charged:

$$V_s = 0.29\ \text{kV},\ E = 9.7\ \text{kV/m},\ \sigma_{eff} = 0.11\ \mu\text{C/m}^2,$$

while the compression-molded PLA films had the following characteristics:

$$V_s = 2.73\ \text{kV},\ E = 170.3\ \text{kV/m},\ \sigma_{eff} = 1.51\ \mu\text{C/m}^2,$$

i. e. by 10 times higher.

However, after 20 days of storage, the parameter values of electret properties of samples obtained by solution casting and compression molding became of the same order:

$$V_s = 0.14\ \text{kV},\ E = 8.7\ \text{kV/m},\ \sigma_{eff} = 0.09\ \mu\text{C/m}^2;$$

$$V_s = 0.19\ \text{kV},\ E = 17.0\ \text{kV/m},\ \sigma_{eff} = 0.08\ \mu\text{C/m}^2,$$

respectively.

However, the electret properties of the molded samples are still slightly higher than those of the samples cast from the solution.

The authors of Ref. [15] explained a great difference in the level of PLA properties by the presence of bound water in the samples: they showed that pre-annealing of PLA films led to a significant increase in the level and stability of their surface potential values.

To explain the observed difference in the properties of our samples obtained by different methods, their IR spectra were recorded, but no significant difference was found.

Notice that a range of 3700 − 3745 cm^{-1} refers to isolated ("free") -OH groups, of 3650 − 3660 cm^{-1} does to isolated pairs of neighboring OH-groups (vinyl-OH-groups) connected by hydrogen bonds, of 3650 − 3660 cm^{-1} does to paired groupings of -OH-groups, with a hydrogen bond between. And so, judging by the absorption band group 3550 − 3750 cm^{-1}, water is present in both samples, but its amount is insignificant.

Migration of water molecules is known to occur into the polymer volume that during storage of PLA [1]. For the samples studied, this is evidenced by IR spectra of molded PLA films recorded immediately after preparation and after 90 days of storage (Fig. 2). It can be seen that the intensity of absorption bands at 3650 − 3660 cm^{-1}, corresponding to the vibration of water molecules, differ practically by a factor of 3, if calculated by the absolute value, and by a factor of 4, if calculated by the baseline. We suppose a significant drop in the values of V_s, E and σ_{eff} of electrets, which are inversely proportional to the values of electrical conductivity, to be related to this. The strong effect of polymer humidity on the value of its volume specific electrical conductivity (it can change by 1 − 3 orders of magnitude) is known [16], and it was shown that in this case water molecules were only a catalyst of electrical conductivity and did not participate directly in charge transfer [17].

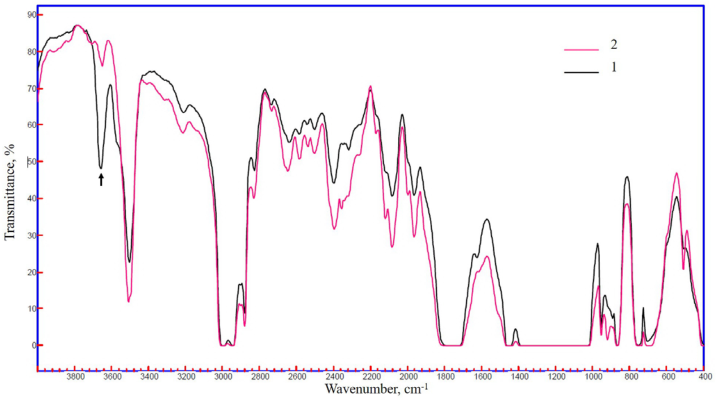

Fig. 2. IR spectra of the PLA film obtained by compression molding, immediately after preparation (*1*) and after 90 days of storage (*2*).
The arrow indicates the position of the H_2O vibration

Probably, a similar process of charge transfer (relaxation of the electret state) is also observed for PLA films obtained by solution casting. However, it is not possible to prove it by IR spectroscopy, since the absorption bands characteristic of chloroform (2800 cm^{-1} refers to valence vibrations of CH, 1220 cm^{-1} does to deformation vibrations of CH, 630 cm^{-1} does to valence vibrations of CCl_3) overlap with the characteristic bands of PLA in this region (Fig. 3).

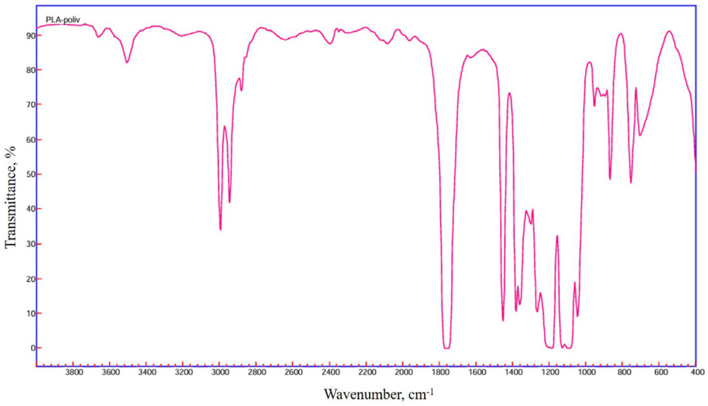

Fig. 3. IR spectrum of a solution-cast PLA film

Taking into account the results of the investigation aimed at increasing the charge stability of PLA by using fine fillers that cause structural defects in the polymer matrix and at the polymer – filler interface, which act as traps for charge carriers [12 – 14], at the second stage of study we created compositions of PLA with a number of dispersed fillers and studied their electret properties (see Fig. 4 and Table 2).

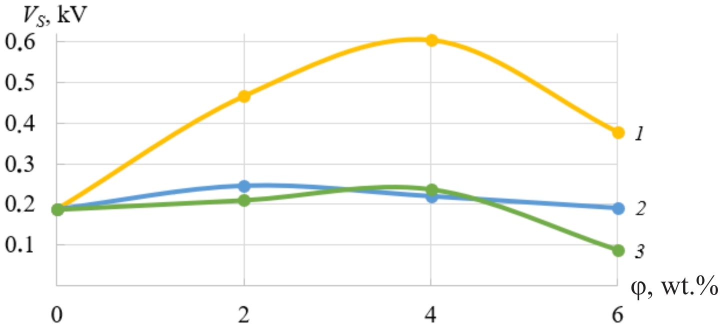

Fig. 4. A plot of surface potential values of PLA compositions with white silica (*1*), aerosil (*2*) and starch (*3*) versus the filler content after 20 days of storage

Table 2

Electret properties of PLA compositions (PLA + filler)

Filler	φ, wt.%	Method	Parameter value					
			Initial			After 20 days		
			V_s, kV	E, kV/m	σ_{eff}, $\mu C/m^2$	V_s, kV	E, kV/m	σ_{eff}, $\mu C/m^2$
No	0	I	0.29	9.7	0.11	0.14	10.9	0.07
		II	2.73	170.3	1.51	0.19	17.0	0.08
White silica	2	I	0.15	5.9	0.05	0.22	2.3	0.02
		II	2.42	153.7	1.37	0.47	22.4	0.09
	4	I	0.21	9.9	0.09	0.33	1.9	0.01
		II	2.45	153.3	1.36	0.61	28.4	0.12
	6	I	0.15	6.4	0.06	0.32	5.2	0.04
		II	3.86	241.0	2.13	0.38	18.3	0.10
Aerosil	2	I	0.13	5.3	0.05	0.28	2.2	0.01
		II	2.85	165.3	1.45	0.25	14.9	0.13
	4	I	0.08	1.6	0.01	0.30	2.3	0.08
		II	3.30	193.0	1.70	0.22	12.3	0.11
	6	I	0.06	6.2	0.05	0.21	2.5	0.02
		II	3.51	205.3	1.82	0.19	12.3	0.11
Starch	2	I	0.05	4.7	0.04	0.26	0.9	0.02
		II	3.35	197.3	1.73	0.21	10.6	0.10
	4	I	0.02	1.5	0.01	0.28	1.5	0.05
		II	2.51	143.7	1.27	0.24	12.6	0.11
	6	I	0.02	2.7	0.02	0.30	2.4	0.02
		II	2.81	164.3	1.43	0.09	4.8	0.05

Notations: φ is the filler content, V_s is the surface potential, E is the electric field strength, σ_{eff} is the effective surface charge density.

Footnote. Samples were manufactured either by solution casting (Method I) or by pressing (Method II).

If we consider compositions of PLA with fillers obtained by mixing filler with polymer melt, there is a tendency to increase the values of electret properties of PLA when adding fine particles. Composition of PLA with hydrated silicon dioxide has the best properties, and the highest values of V_s, E and σ_{eff} are observed at 4 % content of white silica (BS-120). If pyrogenic silicon dioxide is introduced into polylactide, then the optimal (for the manifestation of electret properties) formulation is a sample of polylactic acid with 2 % aerosil. The doping with starch, as well as aerosil, does not change the values of electret characteristics of the polymer significantly. An increase in the level of electret characteristics of PLA upon addition of dispersed particles is explained by the formation of structural defects acting as traps for charge carriers [12 – 14]. The solid surface of the filler reduces the segmental mobility of polylactic acid macromolecules due to the appearance of physical and hydrogen bonds between macromolecules and fillers. On one hand, this makes it difficult to polarize polylactide during corona discharge treatment. On the other hand, it complicates and even excludes the possibility of polarization relaxation. Especially it concerns dipole groups (for example, oxygen-containing), formed under the action of corona discharge and oriented along the field direction of corona discharge; the dipole appearing in the composition of macromolecule is fixed, the freedom of movement, oscillation of the macromolecule section in the areas adjacent to the surface is limited. That is, in filled polylactide near the surface of dispersed particles there is a layer of macromolecules with limited mobility, which slows down (excludes) relaxation of dipole polarization in polylactide compositions. The following mechanism is also quite possible: dispersed particles act as macrodipoles, in which a dipole moment is induced when the sample is treated in the electric field of corona discharge. Applied to aerosil or white silica, this mechanism can be described as follows. A silicon atom has an unsaturated valence bond, which is saturated by the addition of a hydroxyl group OH [18]. The loss or gain of a proton leads to formation of a negatively ($[OH]^-$) or positively ($[H_2O]^+$) charged group, respectively. When such a dispersed particle enters an electric field, the charged groups move along the surface of the particle toward the opposite electrode. As a result, macrodipoles are formed in the structure of composites with increased concentration of charged hydroxyl groups on both sides of the filler particle. An increase in the number of charged particles and energy traps of charges in composites compared to the original polymer positively affects the ability of samples to be charged in the corona discharge and increases the stability of their surface potential, effective surface charge density and electric field strength.

It should be noted that annealing of the polymer before polarization is an effective way to preserve high level of electret properties. Fig. 5 compares the temperature curves of surface potential drop of PLA samples annealed and not annealed immediately before polarization. The annealing was carried out at 130° for 30 min.

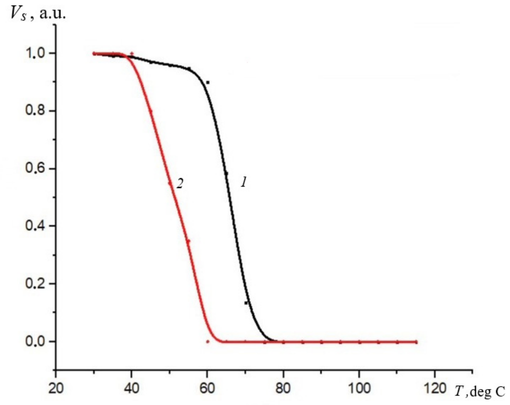

Fig. 5. A plot of surface potential versus temperature for the PLA samples with 4% starch polarized in the field of negative corona discharge. The samples were pre-annealed in a muffle furnace (*1*) or not annealed (*2*)

As can be seen from the presented graphs of the potential-temperature relation (see Fig. 5), the temperature and consequently the time stability of the surface potential increases significantly after pre-annealing.

Compositions of PLA with fillers manufactured via solution casting have a low level of electret characteristics. This result can be explained by poor dispersion of the filler and the presence of agglomerates with sizes exceeding the thickness of the samples. It is known that in production conditions polymer compositions cannot be obtained by simple addition of filler to the polymer solution, as this does not achieve a high degree of dispersion of filler agglomerates. Of course, it is possible to use special ball or bead mills, in which the filler agglomerates are grinded between hard surfaces (e.g., between the surface of metal balls), or ultrasonic dispersants. But the main limitation is due to the fact that polymer solutions have low concentration and solvents are very expensive. As a rule, compositions of polymers with dispersed fillers are obtained by this method only when the solvent is a component thereof (paints, enamels, etc.).

At the same time, mixing of filler with polymer melt is the most common in the polymer composites industry. High quality of filler dispersion in the absence of any additional auxiliary additives levels out the disadvantages in the form of high energy consumption and expensive high-tech equipment.

Summary

Thus, polylactide films obtained by compression molding are an order of magnitude better charged in unipolar corona discharge than films obtained by the solution casting. After 20 days of storage, the values of electret characteristics of samples obtained by different methods become of the same order, but the electret properties of molded samples are still slightly higher than those of samples cast from solution.

The difference in the electret properties of different PLA samples is due to the presence of polar liquid molecules (chloroform, water) in its volume, the presence of which increases the values of electrical conductivity of the samples.

The compositions of PLA with fillers obtained by the method of mixing the filler with the polymer melt show a tendency to increase the values of electret parameters of PLA when dispersed particles are added. The improvement in electret characteristics of PLA when bringing in the fine particles is explained by formation of structural defects acting as traps for charge carriers, decrease of segmental mobility of PLA macromolecules at the surface of filler particles and appearance of macrodipoles with increased concentration of charged hydroxyl groups, the role of which is played by dispersed filler particles.

The PLA-filler compositions obtained by solution casting generally exhibit low electret characteristics.

The PLA composition with 4 % content of hydrated silicon dioxide (white silica) exhibits the best electret properties. In order to create the preservation of high level of values of electret properties of polylactide, it is recommended to anneal the polymer before charging and to prevent the contact of the produced electrets from the action of air moisture during operation.

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THE AUTHORS

ZAGIDULLINA Inna A.
Kazan National Research Technological University
68 Karl Marx St., Kazan, the Republic of Tatarstan, 420015, Russia
zagidullina_inna@mail.ru
ORCID: 0000-0002-5736-6957

GUZHOVA Alina A.
Kazan National Research Technological University
68 Karl Marx St., Kazan, the Republic of Tatarstan, 420015, Russia
alina_guzhova@mail.ru
ORCID: 0000-0003-4354-8984

KARULINA Elena A.
Herzen State Pedagogical University of Russia
48 Moyka Emb., St. Petersburg, 191186, Russia
karulina@mail.ru
ORCID: 0000-0001-9604-4769

KULEMINA Sofya M.
Herzen State Pedagogical University of Russia
48 Moyka Emb., St. Petersburg, 191186, Russia,
sonia.kulemina@yandex.ru
ORCID: 0009-0002-3569-498

СВЕДЕНИЯ ОБ АВТОРАХ

ЗАГИДУЛЛИНА Инна Александровна – *кандидат технических наук, доцент кафедры технологии переработки полимеров и композиционных материалов ФГБОУ ВО «Казанский национальный исследовательский технологический университет», г. Казань, Республика Татарстан, Россия.*
420015, Россия, Республика Татарстан, г. Казань, ул. К. Маркса, 68
zagidullina_inna@mail.ru
ORCID: 0000-0002-5736-6957

ГУЖОВА Алина Альбертовна – *кандидат технических наук, доцент кафедры иностранных языков для профессиональной коммуникации ФГБОУ ВО «Казанский национальный исследовательский технологический университет», г. Казань, Республика Татарстан, Россия.*
420015, Россия, Республика Татарстан, г. Казань, ул. К. Маркса, 68
alina_guzhova@mail.ru
ORCID: 0000-0003-4354-8984

КАРУЛИНА Елена Анатольевна – *кандидат физико-математических наук, доцент кафедры общей и экспериментальной физики Российского государственного педагогического университета имени А. И. Герцена, Санкт-Петербург, Россия.*
191186, Россия, г. Санкт-Петербург, наб. р. Мойки, 48
karulina@mail.ru
ORCID: 0000-0001-9604-4769

КУЛЕМИНА Софья Михайловна – *аспирантка кафедры общей и экспериментальной физики Российского государственного педагогического университета имени А. И. Герцена, Санкт-Петербург, Россия.*

191186, Россия, г. Санкт-Петербург, наб. р. Мойки, 48
sonia.kulemina@yandex.ru
ORCID: 0009-0002-3569-498

Received 05.01.2025. Approved after reviewing 28.01.2025. Accepted 28.01.2025.

Статья поступила в редакцию 05.01.2025. Одобрена после рецензирования 28.01.2025. Принята 28.01.2025.

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