

Original article

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## ELECTRIC AND THERMOELECTRIC PROPERTIES OF COORDINATION POLYMER BASED ON PHENAZINE LIGANDS AND SILVER

A. A. Tretyakov<sup>1</sup>, V. M. Kapralova<sup>1</sup>✉, I. Yu. Sapurina<sup>2</sup>,  
N. T. Sudar<sup>1</sup>, M. A. Shishov<sup>1,2</sup>

<sup>1</sup> Peter the Great St. Petersburg Polytechnic University, St. Petersburg, Russia;

<sup>2</sup> Institute of Macromolecular Compounds of RAS, St. Petersburg, Russia

✉ [kapralova2006@yandex.ru](mailto:kapralova2006@yandex.ru)

**Abstract.** The paper presents the results of an experimental study of the electric conductivity and thermoelectric properties of a new coordination polymer (CP) based on phenazine ligands (Phz) and silver (Ag) synthesized by a one-step method. This method produces phenazine by oxidative dimerization of aniline under the action of  $\text{AgNO}_3$  followed by release of metal nanoparticles and subsequent self-organization of Phz with excess  $\text{AgNO}_3$  into the CP. The used method of the Phz-Ag synthesis was found to make possible obtaining CP with conductivity of about 1300 S/cm commensurable with the modern record value for CPs. The obtained experimental data led to the conclusion that the studied CP Phz-Ag possesses a metallic type of conductivity. A physical mechanism of forming this property was put forward.

**Keywords:** polymer composite, nanocomposite, thermal properties, coordination polymer, phenazine ligand

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## ЭЛЕКТРИЧЕСКИЕ И ТЕРМОЭЛЕКТРИЧЕСКИЕ СВОЙСТВА КООРДИНАЦИОННОГО ПОЛИМЕРА НА ОСНОВЕ ФЕНАЗИНОВЫХ ЛИГАНДОВ И СЕРЕБРА

А. А. Третьяков<sup>1</sup>, В. М. Капралова<sup>1</sup>✉, И. Ю. Сапурина<sup>2</sup>  
Н. Т. Сударь<sup>1</sup>, М. А. Шишов<sup>1, 2</sup>

<sup>1</sup> Санкт-Петербургский политехнический университет Петра Великого,  
Санкт-Петербург, Россия;

<sup>2</sup> Институт высокомолекулярных соединений РАН, Санкт-Петербург, Россия

✉ [kapralova2006@yandex.ru](mailto:kapralova2006@yandex.ru)

**Аннотация.** В статье представлены результаты экспериментального исследования электропроводности и термоэлектрических свойств нового координационного полимера (КП) на основе феназиновых лигандов (Phz) и серебра (Ag), синтезированного одностадийным методом. Этот метод позволяет получать феназин путем окислительной димеризации анилина под действием  $\text{AgNO}_3$  с выделением наночастиц металла и последующей самоорганизацией Phz с избытком  $\text{AgNO}_3$  в КП. Установлено, что использованный метод синтеза Phz-Ag позволяет получать КП с проводимостью примерно 1300 С/см, что соизмеримо с современным рекордным значением для КП. Полученные экспериментальные данные позволили заключить, что новый КП обладает металлическим типом проводимости. Предложен физический механизм формирования этого свойства.

**Ключевые слова:** полимерный композит, феназиновый лиганд, координационный полимер, термоэлектрические свойства

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### Introduction

In recent years, considerable attention has been paid to the study of coordination polymers (CPs), which are organized metal-organic frameworks [1–4] consisting of organic ligands and transition metal atoms or ions. Organic ligands contain various functional groups (carboxylate, pyridine, azole, etc.) that include electron-donor atoms (O, N, S), which, due to donor-acceptor bonding, provide the ligand interaction with the transition metal. CPs are capable of forming one-, two-, or three-dimensional crystalline structures, the design of which is very diverse.

The electrical conductivity of CP varies over a wide range. Most of them are dielectrics with conductivity of  $10^{-14}$ – $10^{-4}$  S/cm, but there are known CPs with significant conductivity of semiconductor and metallic types [3]. For example, the specific conductivity of a CP based on benzene-hexathiol ligands and copper, prepared as a film at the interface of two immiscible solvents, is approximately 1500 S/cm [5].

Elucidating the reasons for the high conductivity of CP is an important physical task. As noted above, CPs demonstrating metallic conductivity have now been synthesized. There is no band gap in these CPs and the charge carriers fill the states up to the Fermi level [6]. However, so far, the most effective way to increase the electrical conductivity of CPs is the inclusion of metal atoms into the intermolecular space of these polymers, which can form conducting clusters that provide the through conductivity of CP samples [7].

A variety of electrophysical properties of CPs determines a wide range of their practical applications. CPs can be used to produce porous materials with given physicochemical parameters of the available intermolecular volume [5], so CPs can be applied as organic frameworks for lithium storage in modern rechargeable batteries [8]. CPs with high electrical conductivity are considered to be promising materials for fabricating conducting channels in field-effect transistors [9]. The possibility of using CPs as detecting materials in new sensor devices is currently under discussion [10].

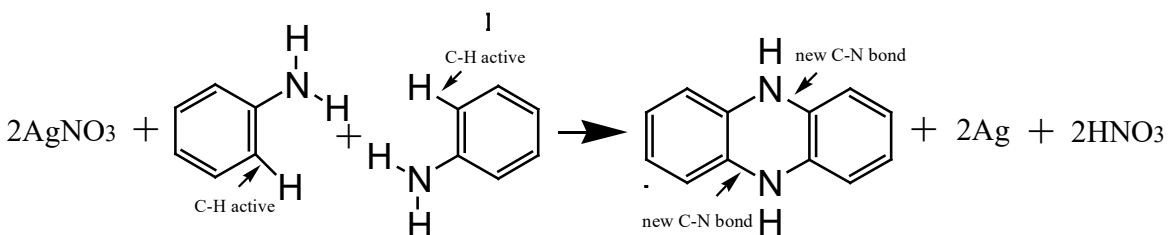
Thus, the synthesis of novel CPs with high conductivity and unique physical and chemical properties seems to be an urgent task, and the study of the electrophysical properties of such polymers is of significant scientific and practical relevance.

The authors of [11] developed a method for the one-step synthesis of a new CP based on phenazine ligands (Phz) and silver (Ag). It involved the synthesis of phenazine by oxidative dimerization of aniline under the action of silver nitrate and subsequent extraction of metal Ag-nanoparticles. As a result, the self-assembly of phenazine ligands into a crystalline organo-metallic framework occurred through interaction with an excess of  $\text{AgNO}_3$ . In the Phz-Ag combination silver performs two functions: silver ions serve as binding elements for the ligands, and metallic silver clusters play the role of guest elements, which, according to the authors of [11], will determine the high conductivity of the material.

This paper is aimed at finding out the nature of the conductivity of this CP synthesized by the one-step mechanism, which can be based on the electric impedance and thermoelectric data for this CP.

### Materials and Methods

The synthesis of the Phz-Ag composition was carried out under normal conditions in the volume of the aqueous-organic phase as a unified process. It began with the formation of the Phz molecule by oxidative dimerization of aniline under the action of silver nitrate  $\text{AgNO}_3$  with the subsequent release of two reduced silver atoms [12]. The relevant chemical reaction is as follows:



formation of a crystalline organometallic matrix and saturation of the matrix with silver metal nanoparticles. The resulting product was filtered out, washed with water, and dried under normal conditions. When dried, it was a yellowish-brown powder.

A study of the product composition showed that its organic part (60% C, 13% N, 3.3% H, 19.8% O) coincides well with the formula of the oxidized phenazine molecule containing  $\text{NO}_3^-$ -group as the counterion, and the silver content is 63–65 wt.%. The general formula of the product can be represented as  $\text{Ag}_{3.5}(\text{Phz}^+ \text{NO}_3^-)$ .

The composition and morphology of the Phz-Ag samples were examined using a Carl Zeiss Supra 55 VP scanning electron microscope (SEM).

Fig. 1 shows SEM image of Phz-Ag CP. We can see (Fig. 1,*a*) that the aggregate structure of the studied CP is characterized by the presence of thin two-dimensional plates (microcrystals) with lateral size of 5–8  $\mu\text{m}$ . Judging by the size of the end faces and transparency of the plates, their thickness does not exceed 5–10 nm, indicating that the microcrystals consist of only a few polymer layers. As the accelerating voltage of electron beam intensity increases (Fig. 1,*b*), silver



nanoparticles (light objects) localized both between microcrystals and on polymer planes become distinguishable. Ag particles are arranged in rows framing the edge of the polymer plates. Most Ag particles are tens of nanometers in size.

The conductivity and the Seebeck coefficient of the samples were measured simultaneously on the Netzsch SBA 458 Nemesis in the temperature range from 293 to 393 K. Electrical conductivity was measured using the four-point method. Specific electrical conductivity was determined using samples pressed into tablets with a diameter of 16 mm and a thickness of 1.5–2 mm using a hydraulic press. Three series of samples were made (three samples per each series) at different compressing pressures. Sufficiently strong and measurable samples were already obtained at a pressure of approx. 3 MPa.

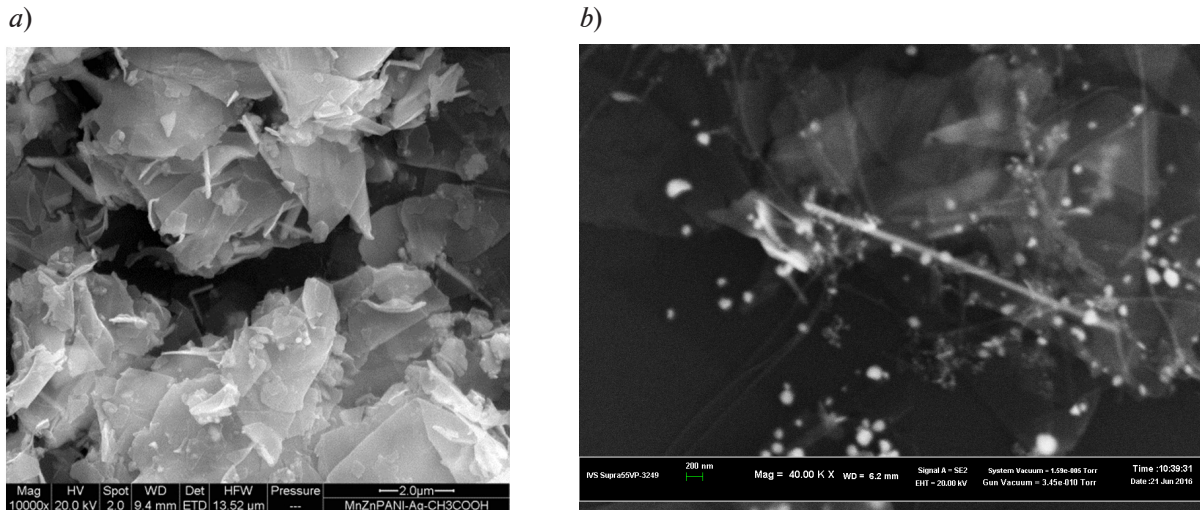


Fig. 1. SEM images of Phz-Ag morphology taken in standard mode (a) and at maximum electron beam intensity (b)

To measure the electrical impedance modulus  $Z$  and the phase angle  $\varphi$ , we used impedance meters E7-20 (in the frequency range from 25 to  $5 \cdot 10^4$  Hz) and E7-29 (in the frequency range from  $5 \cdot 10^4$  to  $1.5 \cdot 10^7$  Hz) impedance meters. Measurements were carried out at alternative voltage amplitude of 1 V using flat clamping electrodes made of polished copper.

### Results and discussion

The table shows the average values of density  $\rho$ , conductivity  $\sigma$  and Seebeck coefficient  $S$  measured at room temperature for the Phz-Ag samples made at different pressures. As follows from the presented data, with increasing compression pressure the Seebeck coefficient tends to decrease insignificantly, and the conductivity of samples increases in proportion to increasing pressure, however, even at the highest pressure used in this experiment, the conductivity of Phz-Ag samples is  $\sim 500$  times lower than that of pure silver ( $6.7 \cdot 10^5$  S/cm). The density of the samples increases until the pressing pressure exceeds 17.7 MPa. At higher pressures, its value seems to stabilize and is  $2.4\text{--}2.5$  g/cm<sup>3</sup>. This density value corresponds to the theoretically calculated Phz-Ag density if we take the silver density to be 10.5 g/cm<sup>3</sup> at 63 wt. % and the phenazine density to be 1.2 g/cm<sup>3</sup> at 37 wt. %.

Table

Dependence of Phz-Ag parameter values on pressure

Pressure, MPa	$\rho$ , g/cm <sup>3</sup>	$\sigma$ , S/cm	$S$ , $\mu$ V/K
38.0	$2.4 \pm 0.1$	$1300 \pm 10$	$2.1 \pm 0.1$
17.7	$2.5 \pm 0.1$	$550 \pm 4$	$1.9 \pm 0.1$
1.9	$1.9 \pm 0.1$	$30 \pm 1$	$2.8 \pm 0.1$

Fig. 2,*a* shows the temperature dependence of specific conductivity for two series of Phz-Ag samples prepared at pressures of 1.9 and 17.7 MPa. It can be seen that with increasing temperature their conductivity decreases approximately by a linear law, i.e.,  $d\sigma/dT = \text{const}$ , which allows us to simply calculate the temperature coefficient of resistance (TCR), defined as

$$\text{TCR} = (1/\rho_0)d\rho/dT,$$

where  $\rho_0$  is the specific resistance of the studied samples at  $T = 296$  K. It turned out that its values are  $1.6 \cdot 10^{-3}$  and  $2.0 \cdot 10^{-3} \text{ K}^{-1}$ , for samples pressed at lower and higher pressures, respectively. The TCR of pure silver is  $4.3 \cdot 10^{-3} \text{ K}^{-1}$  [13], so the TCRs of Phz-Ag and Ag are values of the same order, while the specific conductivities of these materials differ by 2–3 orders of magnitude.

Fig. 2,*b* presents the temperature dependence of the Seebeck coefficient for the studied samples. From the data presented, we can see that at room temperature, it is a small positive value, and it increases linearly with temperature. The determined values of  $S$  and the linear character of the dependence  $S(T)$  for Phz-Ag correspond to  $S$  and  $S(T)$  for pure silver [14].

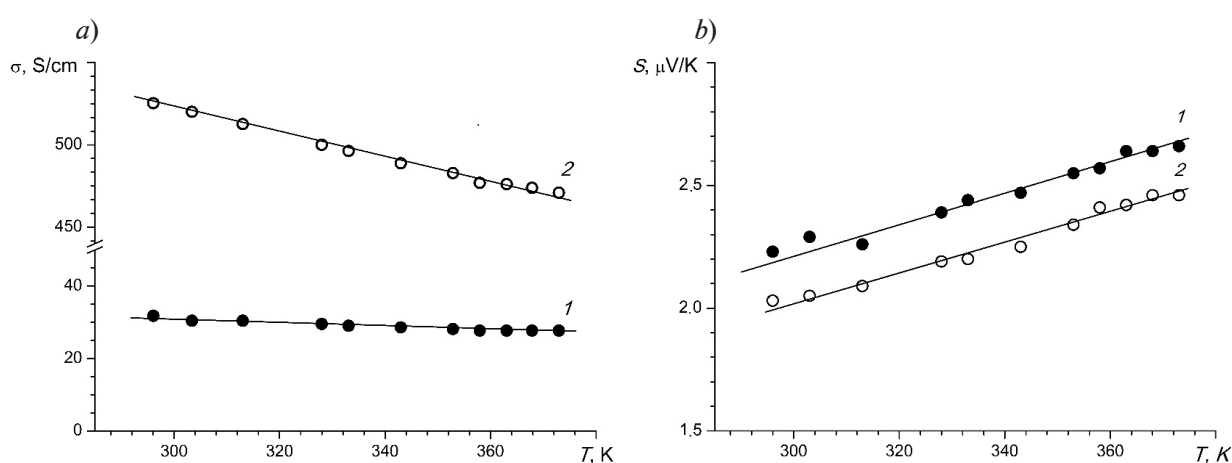


Fig. 2. Temperature dependence of electrical conductivity (*a*) and Seebeck coefficient (*b*) for the samples of Phz-Ag metal-organic coordination polymer pressed at 1.9 MPa (*1*) and 17.7 MPa (*2*)

Thus, the decrease of the specific conductivity with increasing temperature, the value of the TCR of Phz-Ag close to the TCS of silver, as well as the value of the Seebeck coefficient and its temperature dependence give reason to assume that in the investigated Phz-Ag CP the metallic type of conductivity is realized.

To clarify its features, impedance spectroscopy was used, which is sensitive to charge transfer in heterogeneous systems including phase boundaries, electrode interfaces and microstructure elements.

Fig. 3 shows the frequency dependences of the phase angle  $\varphi$  (*a*), the electrical impedance modulus  $Z$  (*b*), and the impedance diagram (*c*) of the Phz-Ag CP sample compressed at 38 MPa. It can be seen that in the entire frequency range studied  $0 \leq \varphi \leq 90^\circ$ . At a frequency of less than 1 kHz the value of  $\varphi \approx 0^\circ$ , and the electric impedance modulus is nearly independent of frequency and equal to  $\sim 8$  mOhm, i.e. the conductivity of the sample is ohmic and is  $\sim 125$  S. The frequency range from 1 to 100 kHz can be considered an intermediary. There is a sharp and non-linear increase in  $\varphi$  and  $Z$ , and, as it follows from Fig. 3,*c*, the increase in  $Z$  with rising frequency is mainly due to the increase in the imaginary part of the impedance  $\text{Im}Z$ . Finally, in the high-frequency region, the value of  $\varphi$  stabilizes near  $90^\circ$ , but the value of  $Z$  grows linearly with increasing frequency. This behavior of the  $Z(f)$  dependence in the high-frequency region indicates the inductive type of conductivity, because  $Z = 2\pi fL$ , where  $L$  is the inductance of the sample. Using this relation, we obtain that at frequencies above 100 kHz the value of  $L$  will be  $\sim 60$  nH. The linear, close to vertical shape of the impedance diagram of the studied sample indicates that its conductivity is not related to any diffusion processes or to the influence of interphase boundaries (in this situation, these are the boundaries of microcrystallites in contact with each other) [15].

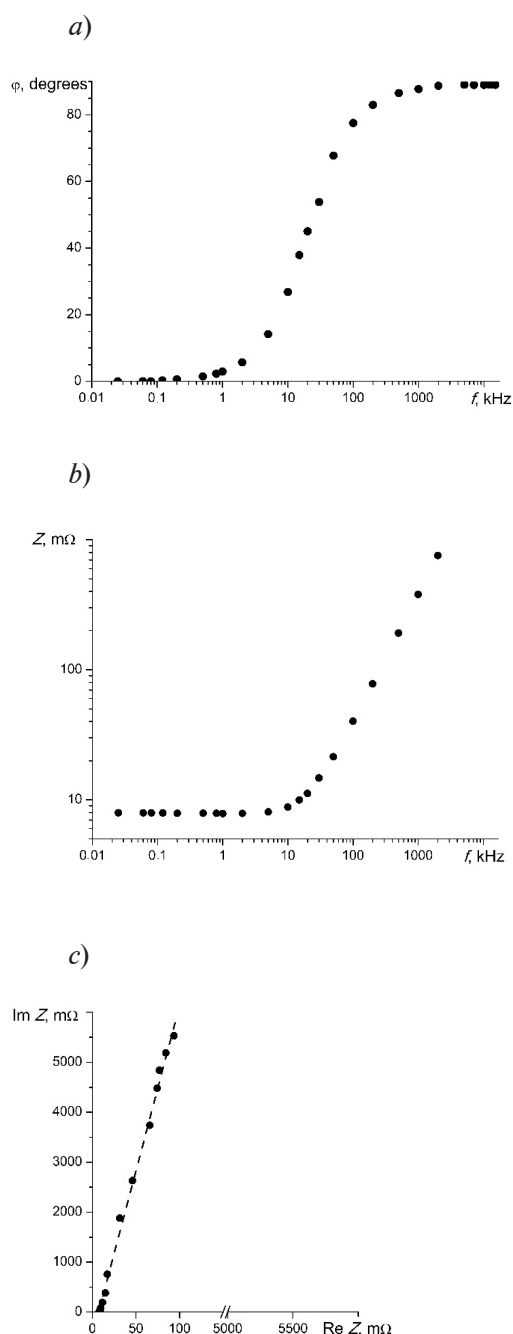


Fig. 3. Frequency dependences of electrical impedance modulus  $Z$  (a), phase angle  $\varphi$  (b) and impedance diagram (c) of Phz-Ag sample produced at a pressure of 38 MPa (data at room temperature)

It was noted above that conglomerates of silver metal about 10 nm in size are present on the surfaces of Phz-Ag microcrystals. Therefore, the presence of smaller silver clusters that are not fixed by SEM but form conductive metal films in the form of individual stripes seems very likely. It should be noted that the authors of [16] point out that silver interlayers can also occur between the individual phenazine ligands that form the microcrystallite. It should be emphasised here that the conductive properties of metal clusters are retained when their size is reduced down to 1 nm [17]. It is known that the specific electrical conductivity of metal films formed from nanoscale clusters is significantly lower than that of their bulk counterparts. For film

The electrical equivalent circuit of this sample can be represented as a series-connected active resistance and inductance.

Let us consider the possible reasons for the appearance of metallic conductivity in the studied Phz-Ag CP samples, as well as the ohmic and inductive nature of their conductivity in the low- and high-frequency regions, respectively. In our opinion, the formation of thin silver films on the surface of microcrystals can be considered as one of the reasons.

We noted above that there are conglomerates of metallic silver  $\sim 10$  nm in size on the surface of Phz-Ag microcrystals. Therefore, the presence of smaller silver clusters, not recorded by SEM, but forming conductive metal films in the shape of individual strips, seems very likely. Note that the authors [15] point to the possibility of formation of silver interlayers also between individual phenazine ligands that form a microcrystallite. Here it should be emphasized that the conductive properties of metal clusters are retained even when their size is reduced down to 1 nm [16]. It is known that the conductivity of metallic films formed from nanoscale clusters is significantly lower than that of their bulk analogues. For film thickness less than 40 nm, it is estimated to be no more than  $10^4$  S/cm and weakly dependent on the film thickness [17, 18]. With regard to the considered situation, we can assume that during pressing the plates of Phz-Ag microcrystals are stacked laterally on each other, providing a good overlap of the metal strips and a sufficiently high level of conductivity. As the compression pressure increases, the number of overlaps increases, which leads to the emergence of conductive wire-like microchannels piercing through the polymer sample. Such channels can be considered as microwires connecting the opposite sides of the tablet. They are characterized by a finite value of conductivity and inductance.

Let us consider the possible causes of metallic conductivity in the investigated Phz-Ag samples, as well as the ohmic and inductive nature of their conductivity, respectively in the low- and high-frequency regions.

The most likely cause is the formation of thin films of silver on the surfaces of the microcrystals

thicknesses below 40 nm, it is estimated to be no more than  $10^4$  S/cm and weakly dependent on film thickness [18, 19]. In the situation under consideration, it can be assumed that during the pressing process the plates of Phz-Ag microcrystals stack laterally on top of each other, ensuring good overlapping of the metal stripes and a sufficiently high level of conductivity. As the pressure increases, the number of overlaps also increases, creating conductive filament-like microchannels that penetrate the polymer sample. Such channels can be thought of as microwires connecting opposite sides of the tablet, and it is these that determine the conductivity and inductance of the samples under investigation.

### Conclusion

We carried out an experimental study of the effect of temperature on the specific conductivity and the Seebeck coefficient of a novel phenazine-silver coordination polymer, which demonstrates that the conductivity of this CP is of metallic nature. The conductivity value depends on the pressure at which the Phz-Ag tablet is prepared, and reaches a value of  $\sim 1300$  S/cm, which is comparable to the highest CP conductivity value achieved to date. The metallic nature of the conductivity is due to the release of atomic silver during the synthesis of CP, its deposition on the surface of microcrystals and the formation of metallized strips on their surface. When the samples are pressed, electrical contact emerges between the individual microcrystals and conductive channels are formed inside the CP sample.

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## THE AUTHORS

### TRETYAKOV Artem A.

*Peter the Great St. Petersburg Polytechnic University*  
29 Politechnicheskaya St., St. Petersburg, 195251, Russia  
tretartem@gmail.com  
ORCID: 0000-0001-9050-4453

### KAPRALOVA Viktoria M.

*Peter the Great St. Petersburg Polytechnic University*  
29 Politechnicheskaya St., St. Petersburg, 195251, Russia  
kapralova2006@yandex.ru  
ORCID: 0000-0001-9050-4453

### SAPURINA Irina Yu.

*Institute of Macromolecular Compounds of RAS*  
31 Bolshoy Ave. V.O., St. Petersburg, 199004, Russia  
sapurina@mail.ru  
ORCID: 0000-0001-9050-4453

### SUDAR Nicolay T.

*Peter the Great St. Petersburg Polytechnic University*  
29 Politechnicheskaya St., St. Petersburg, 195251, Russia  
sudar53@mail.ru  
ORCID: 0000-0001-9050-4453

### SHISHOV Mikhail A.

*Peter the Great St. Petersburg Polytechnic University*  
29 Politechnicheskaya St., St. Petersburg, 195251, Russia  
shv\_misha@mail.ru  
ORCID: 0000-0001-5100-2565

**СВЕДЕНИЯ ОБ АВТОРАХ**

**ТРЕТЬЯКОВ Артём Александрович** – аспирант Высшей школы электроники и микросистемной техники Санкт-Петербургского политехнического университета Петра Великого.

195251, Россия, г. Санкт-Петербург, Политехническая ул., 29

tretartem@gmail.com

ORCID: 0000-0001-9050-4453

**КАПРАЛОВА Виктория Маратовна** – кандидат физико-математических наук, доцент Высшей школы электроники и микросистемной техники Санкт-Петербургского политехнического университета Петра Великого.

195251, Россия, г. Санкт-Петербург, Политехническая ул., 29

kapralova2006@yandex.ru

ORCID: 0000-0001-9050-4453

**САПУРИНА Ирина Юрьевна** – доктор химических наук, старший научный сотрудник Института высокомолекулярных соединений РАН, Санкт-Петербург, Россия.

199004, Россия, г. Санкт-Петербург, Большой пр. В.О., 31

sapurina@mail.ru

ORCID: 0000-0001-9050-4453

**СУДАРЬ Николай Тобисович** – доктор физико-математических наук, профессор Высшей школы электроники и микросистемной техники Санкт-Петербургского политехнического университета Петра Великого.

195251, Россия, г. Санкт-Петербург, Политехническая ул., 29

sudar53@mail.ru

ORCID: 0000-0001-9050-4453

**ШИШОВ Михаил Александрович** – кандидат технических наук, старший научный сотрудник научно-исследовательской лаборатории «Полимерные материалы для тканевой инженерии и трансплантологии» Санкт-Петербургского политехнического университета Петра Великого.

195251, Россия, г. Санкт-Петербург, Политехническая ул., 29

shv\_misha@mail.ru

ORCID: 0000-0001-5100-2565

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