

The ion-conducting composites based on the aliphatic and aromatic epoxy oligomers and the lithium perchlorate salt

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The purpose of this study was investigation of the polymer composites based on the aliphatic epoxy oligomer – diglycidyl ether of polyethylene glycol DEG-1, aromatic diene-epoxy resin DER-331 and different content of the perchlorate lithium salt. Structure and properties of composites were characterized by means of the wide angle X-ray scattering (WAXS), the thermogravimetric analysis (TGA), the differential scanning calorimetry (DSC) and the broadband dielectric spectroscopy (BDS). The results show that the synthesized composites based on the aliphatic and aromatic oligomers in the presence of lithium perchlorate salt are amorphous with the ionic conductivity $\sim 4 \cdot 10^{-4}$ S/cm at 200 °C. An increase of the glass transition temperature of the synthesized composites has been observed with increasing of the salt concentration that indicates the influence of lithium perchlorate on the molecular structure of composites.

Keywords: aliphatic and aromatic oligomers, lithium perchlorate salt, structure, ion-conductivity.

Introduction.

Up to nowadays broad efforts have been devoted to multifunctional materials [1] with a reduced cost, increased performance and lifetime, which are needed for electric vehicles and stationary energy storage systems [2]. Solid state batteries use solid electrolytes to overcome the safety issues, such as a risk of leakage, volatilization and flammability of liquid electrolytes [3–10]. Furthermore, solid polymer electrolytes (SPE) show excellent electrochemical stability and favorable mechanical characteristics, and can perform over a wide temperature window [11], high energy density as well as memory effect [12], avoid an internal shorting [5], and also a reduction of the net weight and volume of a battery [3, 10]. In addition, flexibility of SPE provides production of the flexible batteries for emerging applications, such as wearable electronic devices and flexible sensors [6]. An electrolyte is a key for the performance of a battery [2] and plays a critical role in providing high ionic conductivity and transferring mechanical loads [13]. Except the usual research approaches of polymer matrix, the investigations of the ionic conductivity and dielectric properties are also used for the development of electrolytes [1].

Thermosetting polymers such as epoxy resins are materials for extensive use in the aerospace industry, automobile and marine applications, in microelectronics and food-packaging industries [14–16]. Properties of epoxy resins depend on specific combination of the used type

of epoxy resins and curing agents [17]. Epoxy resins are important thermoset materials due to their excellent thermomechanical, barrier, chemical and electrical properties, low shrinkage upon cure and outstanding adhesion to various substrates [15, 16, 18–20]. They are highly used as matrix in conducting polymer composites [19]. Versatility is one of the strong features of epoxy polymers [15].

Diene-epoxy resin has been studied and extensively used in industry, however, it was usually brittle, easy peeling and with poor impact resistance [21]. These characteristics decreased the value and scope of their application. Molecular structure of epoxy resin can be easily modified for various applications considering its versatile properties [22]. There are many approaches to improve characteristics of diene-epoxy resin, such as modification by vegetable oils, adding the plasticizers [21], elastomers [16] etc. Polymer blending technique is a quick and economic alternative, which has been widely used for obtaining materials with optimized properties and potentially can offer easy control of physical properties by compositional change for a wide variety of application prospects [5, 23]. Adding a more flexible aliphatic epoxy oligomer while mixing allows changing of thermal and mechanical properties. Copolymers, which have similar functional epoxy groups, enable formation a cross-linked network (even semi-IPN) due to the similar reaction with the diamine curing agent [24].

Table 1. The chemical structure and the ratios (%) of matrix components

Code	Oligomer name	Oligomer structure	Ratio, %
DEG-1	The diglycidyl ether of polyethylene glycol		67,5
DER-331	The aromatic-diane- epoxy resin		22,5
PEPA	Polyethylene polyamine	$H_2N(CH_2CH_2NH)_nH$	10,0

A great diversity of solid polymer electrolytes has been developed by different combinations of salts and host polymer matrixes [1]. Doping with lithium salts is an important criterion to realize the final application of SPE [5], owing the specific properties of the lithium ion (e.g., its small size) in consequence of the easier migration of Li^+ [25].

Thus, the aim of the research is investigation of thermal and electrical properties of the cross-linked amorphous polymer electrolyte based on the mixture of aliphatic (diglycidyl ether of polyethylene glycol) and aromatic (diane-epoxy resin DER-331) oligomers in ratio 67,5 % and 22,5 % respectively, which have similar functional epoxy groups (table 1), in the presence of the lithium perchlorate salt.

Experimental.

The diglycidyl ether of polyethylene glycol DEG-1 (Macromer, Vladimir, Russia, 26,1 % epoxy group), aromatic diene-epoxy resin DER-331 (DOW Chemical Company, Germany, 23,6 % epoxy group) and salt of lithium perchlorate ($LiClO_4$, Sigma-Aldrich, USA) were used for synthesis of ion-conductive epoxy polymer composites. Lithium perchlorate salt and DEG-1 were previously pre-dried in vacuum at 80 °C during 8 h. After drying, $LiClO_4$ was dissolved in oligomer DEG-1 by heating on a sand bath and DER-331 was added. Solutions of DEG-1/DER-331/ $LiClO_4$ were prepared with salt content from 0 to 30 phr (parts per hundred) on 100 parts of polymer matrix. Polyethylene polyamine (PEPA, Chimia, Kharkov, Ukraine) hardener was used as a curing agent. The chemical structure and the ratios (%) of matrix components (aliphatic DEG-1, aromatic DER-331 oligomers and PEPA) are shown in Table 1.

The structural organization and features of macromolecular ordering of the synthesized composites were investigated by wide-angle X-ray scattering (WAXS) using the X-ray diffractometer DRON-4.07. The X-ray optical scheme was performed using Debye-Scherrer method by passing the primary beam through the polymer sample using $Cu K\alpha$ emission ($\lambda = 1,54 \text{ \AA}$) that was made monochromatic using Ni filter. The X-ray tube BSV27Cu ($U = 30 \text{ kV}$, $I = 30 \text{ mA}$) was used as a source of characteristic X-ray irradiation. The investigations were carried out by automatic step scanning in the range of scattering angles (2θ) from 2,6 to 40,0° and the exposure time was 5 s.

Thermogravimetical analysis (TGA) was carried out in order to examine thermal stability of the composites. TGA was performed using thermogravimetric analyzer TA Instruments TGA Q50 in the temperature range from +25 to +700 °C with the linear heating rate of 20 °C/min in helium atmosphere. Research scattering is 0,01%.

The thermal characteristics were studied by differential scanning calorimetry (DSC) at TA Instruments DSC Q2000 in the temperature range from -70 to +200 °C with the heating rate of 10 °C/min.

The dielectric characteristics of the synthesized composites were investigated by the broadband dielectric analyzer 'Novocontrol Alpha' with Novocontrol Quatro Cryosystem in the frequency range from 10^1 to 10^7 Hz and the temperature range from -60 to +200 °C. All experiments were carried out upon heating up to +200 °C and cooling to -60 °C at 2 C/min under controlled nitrogen flow. The voltage applied to a sample was equal to 0,5 V. The test samples had 30 mm in diameter and a thickness of 0,5 mm and were previously coated by aluminum layer (serving as electrodes) under vacuum. The obtained data was analyzed using the software 'Novocontrol WinDeta 3.8'.

Results and discussion.

The wide-angle X-ray diffraction patterns of pure $LiClO_4$ and the polymer composite with 30 phr of $LiClO_4$ are shown in Figure 1. The WAXS of $LiClO_4$ (curve 1) has a

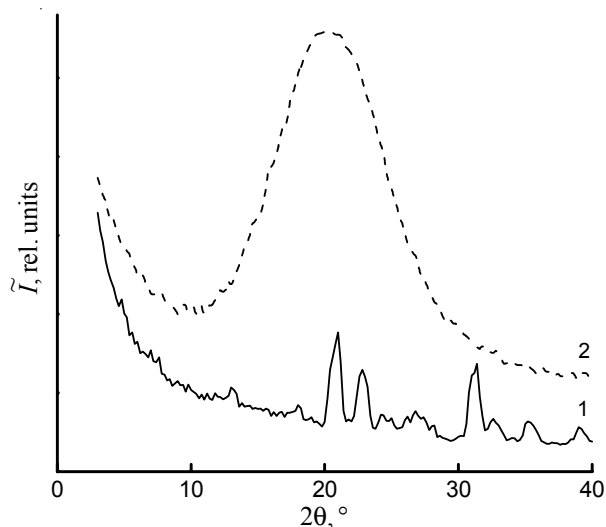


Fig. 1. Wide-angle X-ray diffraction patterns of $LiClO_4$ (1) and the composite with 30 phr of $LiClO_4$ (2)

Table 2. The Bragg distance between the molecular segments

Sample	$2\theta_m$, degrees	d , Å
LiClO ₄ (pure)	20,0	4,44
DEG-1 /DER-331/ LiClO ₄ (30 phr)	20,6	3,92

maximum at $2\theta_m = 20,0^\circ$ indicating the high extent of crystallinity of the ionic salt. Whereas, DEG-1/DER-331 matrix with 30 phr of LiClO₄ (curve 2) is amorphous in contrast to the pure salt. Amorphous state of the salt in the composite can be achieved by interactions between the salt and the matrix. Similar effect was found in polymer system based on polyacrylonitrile where polymer is active in a salt solution and, hence, suppresses the precipitation process of a salt [26].

The epoxy oligomers DEG-1 and DER-331, which were cured by PEPA, are characterized by short-range ordering in a space translation of molecular fragments of their cross-site links. This is confirmed by the presence of one diffraction peak (calculated from the angular half-width) of the diffusion type (amorphous halo), in which the angular position ($2\theta_m$) is about $20,6^\circ$. The average value of the period (d) of a short-range molecular ordering of DEG-1 and DER-331 internodal molecular segments in a polymer volume can be calculated using Bragg equation:

$$d = \lambda(2\sin\theta_m)^{-1},$$

where: λ is the wavelength of the characteristic X-ray emission ($\lambda = 1,54$ Å for Cu K α emission) and it equals to 3,92 E.

This indicates a tendency to decrease the Bragg distance between the molecular segments (Table 2).

The results of the thermogravimetric analysis are given in Fig. 2. From these results, it can be seen that LiClO₄ slightly influences on the thermal stability of the epoxy composite doing it less thermostable. A similar trend of slight shift towards the lower temperature was observed on the weight loss curve with adding the LiNO₃ salt to the

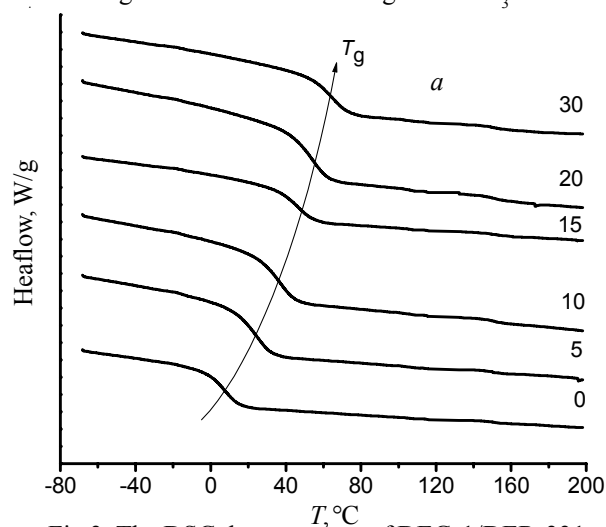


Fig 3. The DSC thermograms of DEG-1/DER-331 composites with different content (the numbers near curves, in phr) of LiClO₄ (a) and thermogram of perchlorate lithium salt (b)

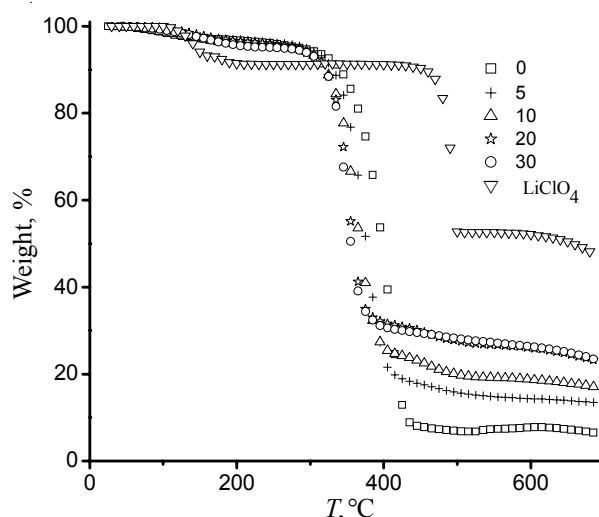


Fig 2. The TGA of pure LiClO₄ and DEG-1/DER-331 composites with different content of LiClO₄ (in phr)

PEO/PVP blend [5]. The weight loss of the composites is negligible up to 200 °C and, probably, it is related to the loss of moisture, and this, in turn, suggests the suitability of composites to operate at elevated temperatures.

The DSC thermograms at the second heating are presented in Fig. 3. In the plot of DEG-1/DER-331 with different content of LiClO₄ a glass transition is only present, thus the composites are amorphous (Fig. 3a). Salt of lithium perchlorate (Fig. 3b) has endothermic peaks at 79, 95, 133 and 150 °C, which correspond to different melting of its crystalline structure. The introduction of LiClO₄ from 0 to 30 phr content into the DEG-1/DER-331 composites leads to a significant increase of their glass transition temperature T_g from 7 to 65 °C. Some polymer electrolytes reported in literature showed similar trend with adding the lithium salt [7–9]. At the same time, according to [26], in the poly(acrylonitrile-co-butyl acrylate) - LiTFSI systems the T_g is lowered with the increased salt content:

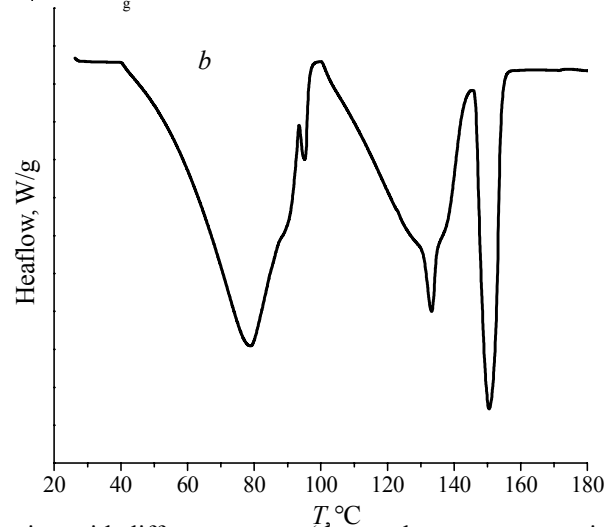


Table 3. Dependences of the glass transition temperatures of the DEG-1/DER-331 composites on LiClO_4 content

Matrix DEG-1/DER-331	Content LiClO_4 , phr					
	0	5	10	15	20	30
T_g , °C	6,5	24,5	37,2	46,5	54,4	64,7

from +48 °C for pure copolymer to -44 °C for the electrolyte containing 95 wt.% of the salt. That can be a result of different strength of interactions between polymer matrices and salts with no stiffening of chains due to physical crosslinking that occurs because the solution of salt acts as a plasticizer.

In our case, the increase of T_g as well as absence of LiClO_4 melting peaks on the DSC thermograms of the composites can be explained by the formation of coordination complexes, which were investigated in detail in our previous work [27] for the composites DEG-1/PEPA with different content of LiClO_4 . The T_g of the DEG-1/DER-331 compositions depending on LiClO_4 content have been determined from the DSC curves (Table 3).

Ionic conductivity of materials depends on the concentration of conducting species and their mobility [5]. It is also known that temperature is one of important factors for the ion mobility [12]. Fig. 4 shows the isothermal spectra of the real part of the conductivity (σ') for different concentrations of LiClO_4 salt in the synthesized composites in the temperature range from "60 to 200 °C in order to study the conducting features of the polymer electrolyte.

As one can see, the conductivity does not vary linearly with the increasing lithium salt concentration, the plateau of conductivity appears in defined frequency region and can be associated with dc conductivity. The maximum value observed for the composites with 10 phr of LiClO_4 equals to $4,1 \cdot 10^{-4}$ S/cm. In [5, 28] the ionic conductivity of polymer systems increases up to optimal concentration, but the further increase of the salt content

Table 4. The level of conductivity of the synthesized composites depending on the LiClO_4 content

Conductivity σ_{dc} , S/cm	Content LiClO_4 , phr		
	0	10	30
60 °C	$3,4 \cdot 10^{-8}$	$1,6 \cdot 10^{-8}$	$1,3 \cdot 10^{-8}$
100 °C	$7,0 \cdot 10^{-7}$	$2,5 \cdot 10^{-6}$	$2,2 \cdot 10^{-6}$
200 °C	$1,8 \cdot 10^{-5}$	$4,0 \cdot 10^{-4}$	$4,1 \cdot 10^{-4}$

leads to decrease of ionic conductivity level. That is because of a consequence formation of ion pairs and ion aggregates, which cause constrains to ionic and polymeric segmental mobility [5].

Fig. 4 demonstrates the heightened value of the ionic conductivity with the temperature increasing. This is mainly related with the increased ionic mobility and the concentration of charge carrier [5]. The higher Li^+ ion concentration and the segmental mobility of the polymer chains above T_g contribute to the high ionic conductivity at the elevated temperature. The level of conductivity of the synthesized composites depending on the LiClO_4 content is shown in the Table 4.

Conclusions.

The WAXS and DSC methods proved the amorphous structure of the synthesized composites. The synthesis of epoxy composites in the presence of lithium perchlorate salt made possible to obtain an ion-conductive polymeric material with a level of ionic conductivity $\sim 10^{-4}$ S/cm at the elevated temperature 200 °C. According to the results of TGA the weight loss in this temperature range is negligible.

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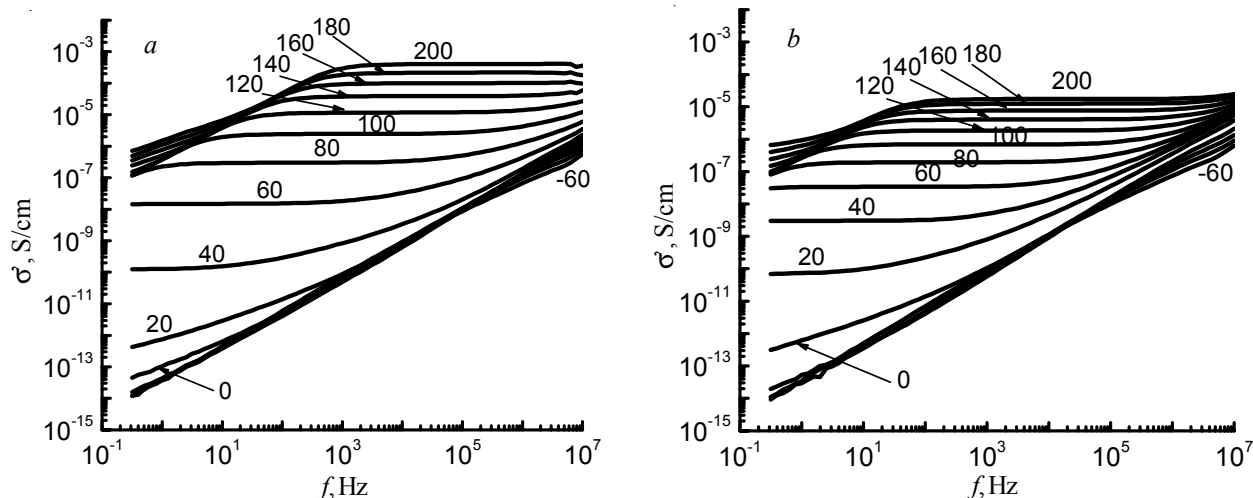


Fig. 4 Real part of complex conductivity spectra at different temperatures of the pure DEG-1/DER-331 composite (a) and the composite with 10 phr of lithium salt (b). The values of temperature are shown near curves

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Іонпровідні композити на основі аліфатичного і ароматичного епоксидних олігомерів та солі перхлорату літію

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Досліджено властивості полімерних композитів на основі епоксидного аліфатичного олігомеру – дигліцидилового ефіру поліетиленгліколю ДЕГ-1, ароматичної діан-епоксидної смоли ДЕР-331 та різного вмісту солі перхлорату літію. Структура і властивості композитів були охарактеризовані за допомогою ширококутового розсіювання рентгенівських променів, термогравіметричного аналізу, диференційної сканувальної калориметрії та ширококутової діелектричної спектроскопії. Результати показали, що синтезовані композити на основі аліфатичного і ароматичного олігомерів та солі перхлорату літію є аморфними з величиною іонної провідності $\sim 4 \cdot 10^{-4}$ См/см за $T=200$ °С. Зі збільшенням концентрації солі спостерігається зростання температури склування синтезованих композитів, що свідчить про вплив перхлорату літію на їхню молекулярну структуру.

Ключові слова: аліфатичний і ароматичний олігомери, сіль перхлорату літію, структура, йонна провідність.

Ионопроводящие композиты на основе алифатического и ароматического эпоксидных олигомеров и соли перхлората лития

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Исследованы свойства полимерных композитов на основе эпоксидного алифатического олигомера – диглицидилового эфира полиэтиленгликоля ДЭГ-1, ароматической диан-эпоксидной смолы ДЭР-331 и различного содержания соли перхлората лития. Структура и свойства композитов были охарактеризованы с помощью широкоугольной дифракции рентгеновских лучей, термогравиметрического анализа, дифференциальной сканирующей калориметрии и широкополосной диэлектрической спектроскопии. Результаты показали, что синтезированные композиты на основе алифатического и ароматического олигомеров и соли перхлората лития являются аморфными с величиной ионной проводимости $\sim 4 \cdot 10^{-4}$ См/см при $T=200$ °С. С увеличением концентрации соли наблюдается рост температуры стеклования синтезированных композитов, что свидетельствует о влиянии перхлората лития на их молекулярную структуру.

Ключевые слова: алифатический и ароматический олигомеры, соль перхлората лития, структура, ионная проводимость.