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TECHNOLOGICAL PARAMETERS OF DIMERIC IONENE-TYPE IONIC LIQUIDS BASED ON TETRAHYDRO-1,4-OXAZINE

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Experimental studies confirmed that introduction of allyl and hydroxyethyl groups, a benzyl fragment, and alkylaromatic radicals into the cationic center of dimeric ionene-type ionic liquids leads to a high ionic conductivity of 10^{-1} S cm^{-1} at -123^0C. The synthesized dimeric ionic liquids were found to be thermally stable in the range $107-206^0$C. The presence of $-O-CH(CH_3)-CH_2-$ groups in the radical adjacent to the quaternized nitrogen atom was shown to enhance thermal stability. Studies of how the nature of the radical near the ionic center affects melting behavior established that the melting point of the dimeric ionene-type ionic liquids increases with an increasing number of $-O-CH(CH_3)-CH_2-$ units in the radical. In particular, correlations between temperature, chemical structure of the tetrahydro-1,4-oxazine-based dimeric ionic liquids, and their ionic conductivity were established; correlation equations are provided and their chemical basis is discussed. These findings support the proposed mechanism of property formation and demonstrate the practical relevance of the developed compounds.

Keywords: dimeric ionic liquids, ionic conductivity, decomposition temperature, melting temperature, properties.

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Introduction

The technical and economic advantages of implementing the strategic direction of development and effective use of ionic materials, especially their dimeric and polymeric forms [1,2], have led to a significant increase in interest in this new class of materials.

Dimeric ionic liquids are a combination of different types of ionic centers and spacers and are characterized by a more controlled architecture of the spatial distribution of ionic centers and their interaction, respectively, predictable behavior, which allows creating ionic materials with a given set of physicochemical properties [3–5] for predicted applications in a certain field [6,7]. It should be noted that polymeric ionic liquids with unique properties have a more complex hierarchical organization with the formation of specific supramolecular structures [3–5]. Previous studies [8–12] and comparative analysis of polymeric and dimeric ionic liquids have shown that the physicochemical properties demonstrate the advantage of polymeric compounds in thermal stability and mechanical strength, but dimeric compounds have better transport properties and more predictable behavior during phase transitions; solvation properties indicate greater versatility of dimeric compounds in interaction with solvents, while polymeric compounds exhibit specific selectivity and more complex solvation behavior.

From a practical point of view, the use of dimeric ionic liquids shows better process ability (due to high ion mobility and ease of processing), but polymeric ionic liquids are optimal for longer-term stability of use in harsh environments [3,10,13].

The economic feasibility of using dimeric ionic liquids has an advantage in terms of production costs and scaling, but polymeric ionic liquids require more complex technological processes but provide a unique set of properties [14].

Given the fundamental differences and specific features of dimeric and polymeric systems of ionic materials that determine their practical application [14,15], research aimed at further improvement and development of the technology for obtaining ionic liquids of polymeric and dimeric nature with a high operational resource, predictable regulation of their operational properties by using ionic centers and spacers of different nature should be considered relevant. There are reasons [16] to believe that further optimization of the structure of dimeric ionic liquids to increase stability and development of new methods for the synthesis of polymeric ionic liquids to reduce the cost of production are promising areas of necessary research.

Experimental

The research task of this work was to measure the ionic conductivity, decomposition temperature and melting temperature of dimeric ionene-type ionic liquids based on tetrahydro-1,4-oxazine and, based on the established correlations between temperature and their chemical structure, to determine the nature of the cationic part at the functional center of the ionic liquid and its relative influence on the physicochemical properties of ionic monomers with different organic cationic parts. This allows us to predict the influence of the nature of radicals near the Nitrogen atom of the macromolecule of polymeric ionene-type ionic liquids based on tetrahydro-1,4-oxazine on their technological parameters (in particular, ionic conductivity and operating temperature).

The physicochemical properties of ionic monomers based on tetrahydro-1,4-oxazine, water-soluble compounds with organic cations and chloride anions, were studied. The choice of anion was due to the results of previous studies [17]: «a decrease in the radius of the anion leads to an increase in the conductivity of polymeric ionic liquids and ionic liquids of the ionene type – ionic liquids with a chlorine anion have a high level of ionic conductivity». In work [18], new dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine derivatives were obtained, the structure of which was confirmed using infrared spectroscopy and elemental analysis (Table 1).

According to the results of infrared spectroscopy of the synthesized dimeric ionic liquids of the ionene type, the absorption bands of the $-CH_2-N^+-$ group appear at 1460 cm^{-1} (strong) and 1380 cm^{-1}, which overlap the characteristic scissor vibrations of the $-CH_2-$ group in cyclohexane at 1450 cm^{-1}; between the usual absorption bands, a band indicating the characteristic strain vibrations for C–H groups is determined in the region of 1400–1430 cm^{-1}; the spectra corresponding to the valence vibrations of the $-CH_2N<$ group (characteristic of tertiary diamine functional groups) disappear in the frequency range of 1450–1400 cm^{-1} and the spectra corresponding to the valence vibrations of the C–Cl group (characteristic of a halogenated compound) in the frequency range of 660 cm^{-1}; characteristic absorption bands of the morpholine cycle are present: a group of weak bands on the low-frequency side of the main band ν(CH) in the region of 2800–2700 cm^{-1}, valence vibrations of the C–N bond in cyclic compounds at 1230 cm^{-1}, asymmetric valence vibrations of the C–O–C group at 1150–1070 cm^{-1}; the appearance of characteristic absorption bands corresponding to the bonds of substituents of the initial molecules of the monomers of the halogen-derived compound and the tertiary diamine is determined.

The establishment of correlation dependences of the influence of the structure of substituents at the quaternary nitrogen atom of dimeric ionic liquids of the ionene type in the temperature range from 15 to 50°C on their ionic conductivity was carried out using conductometry. The melting point and decomposition temperature of dimeric ionic liquids of the ionene type were studied using differential scanning calorimetry, differential thermal and differential thermogravimetric analyses.

Results and discussions

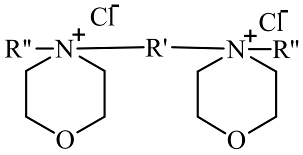
From a practical point of view, the results of the study presented in ref. [17] are of particular interest: dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine melt at a lower temperature in some cases than ionic liquids of the monomeric form. This circumstance is decisive when choosing objects – monomeric models of polymer ionic liquids of the ionene type for studying optimal operating modes, in particular the glass transition temperature.

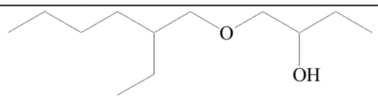
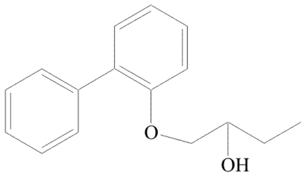
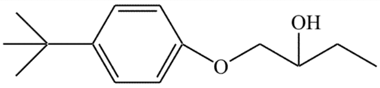
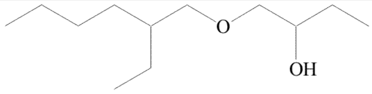
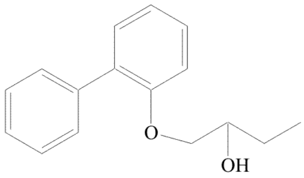
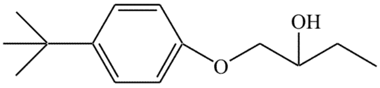
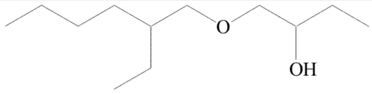
This work also addressed the issue of increasing the thermal stability of dimeric ionic liquids by selecting a certain nature of radicals near the quaternized nitrogen atom.

An indirect assessment of the influence of the nature of the radical, which is a residue of the initial tertiary diamines (both for the synthesis of polymer ionic liquids of the ionene type and for the synthesis of dimeric ionic liquids of the ionene type [18]), on

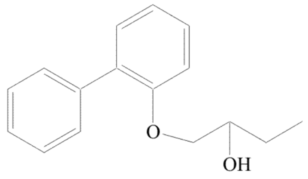
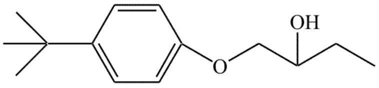
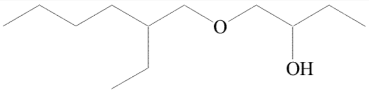
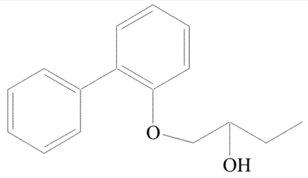
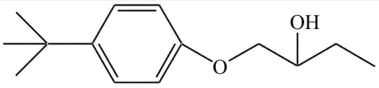
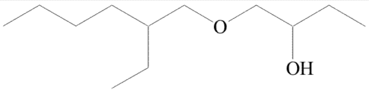
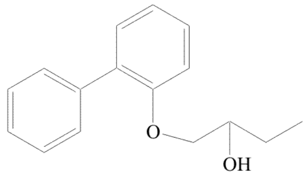
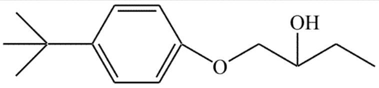
Table 1

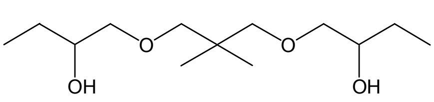
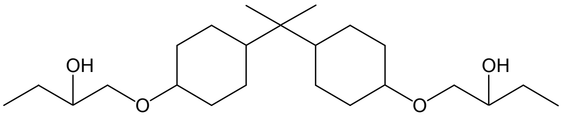
Physicochemical properties of dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine of the general formula

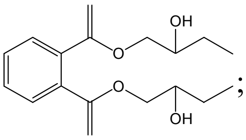
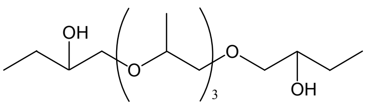
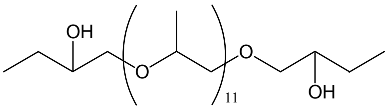


Title	R'	R''	Molecular weight	Chlorine content, %, $\frac{X_{teor.}}{X_{pract.}}$	Density (d_{20}^{20}), kg/m^3	Refractive index (n_D^{20})	Output (ω), %
1	2	3	4	5	6	7	8
C-1-1-1	*		895	$\frac{7.93}{7.95}$	1231.1	1.5368	78.2
C-1-2-2	*		975	$\frac{7.28}{7.30}$	1734.3	1.5675	97.6
C-1-3-3	*		935	$\frac{7.59}{7.60}$	1353.3	1.4852	83.8
C-2-1-1	**		1031	$\frac{6.89}{6.92}$	1239.8	1.5327	77.5
C-2-2-2	**		1111	$\frac{6.39}{6.43}$	1267.5	1.5144	95.9
C-2-3-3	**		1071	$\frac{6.62}{6.63}$	1148.4	1.5653	78.5
C-3-1-1	***		953	$\frac{7.45}{7.48}$	1956.2	1.4730	86.9

Continued Table 1

1	2	3	4	5	6	7	8
C-3-2-2	***		1033	$\frac{6.87}{6.90}$	1486.4	1.5632	95.8
C-3-3-3	***		993	$\frac{7.15}{7.17}$	1562.3	1.5415	86.7
C-4-1-1	****		981	$\frac{7.23}{7.23}$	1002.8	1.5757	96.5
C-4-2-2	****		1061	$\frac{6.69}{6.70}$	1624.5	1.5143	98.9
C-4-3-3	****		1021	$\frac{6.95}{6.97}$	1641.5	1.4745	92.9
C-5-1-1	*****		1445	$\frac{4.91}{4.93}$	1352.6	1.5925	86.5
C-5-2-2	*****		1525	$\frac{4.66}{4.68}$	1187.5	1.4830	94.4
C-5-3-3	*****		1485	$\frac{4.78}{4.81}$	1023.9	1.5547	89.5

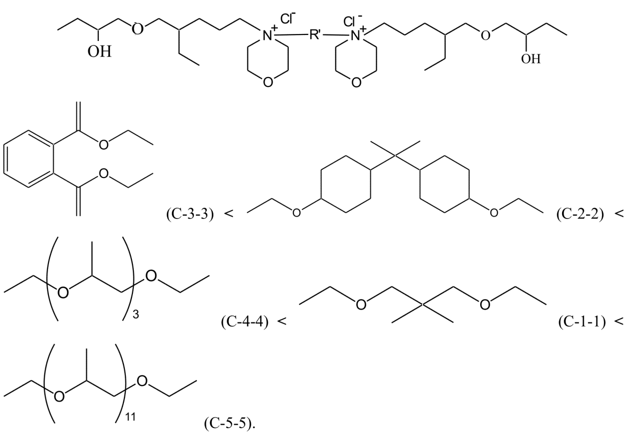
Notes: * –  ; ** –  ;

*** –  ; **** –  ; ***** –  .

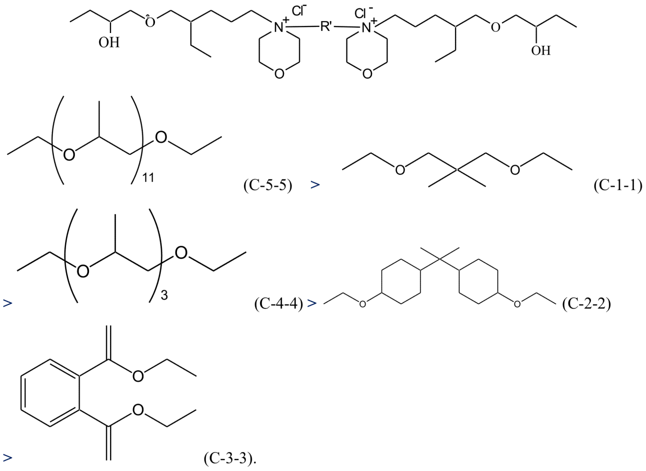
technologically important parameters of polymer ionic liquids of the ionene type was carried out based on the results of determining the thermal stability, glass transition temperature and ionic conductivity of model samples of dimeric ionic liquids of the ionene type. From a theoretical point of view, such results allow us to reasonably approach the choice of the nature of the spacer near the ionic center of polymer ionic liquids of the ionene type and predict its influence on practically important characteristics of ionic compounds of a polymeric nature, which are certain advantages of such a study. Such conclusions are considered appropriate from a practical point of view, because they indicate the possibility of directed regulation of the processes of formation of the defining parameters of polymer ionic liquids of the ionene type directly during the synthesis of these compounds.

According to experimental data on the decomposition temperature of synthesized dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine (Tables 2 and 3), it was found that ionic liquids of the dimeric form are thermally stable up to 107–206°C.

It should be noted that dimeric ionic liquids of the ionene type containing radicals with alkyl and oxyethylene groups are characterized by a more stable state to thermal oxidation degradation. It is worth noting that the introduction of alkylaromatic substituents into the ionic center of dimeric ionic liquids of the ionene type leads to a decrease in their decomposition temperature. Based on the results of the differential thermogravimetric analysis, it can be stated that there is an interesting pattern associated with an increase in the thermal stability of dimeric ionic liquids of the ionic type based on tetrahydro-1,4-oxazine in the series:



The results of determining the influence of the nature of the spacers on the melting point of the synthesized dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine derivatives are given in Tables 2 and 3. It should be noted that the melting point of the dimeric ionic liquids of the ionene type is in the range from −123⁰C to −25⁰C. By the degree of decrease in the melting point of the dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine, the compounds can be arranged in the following series:



The introduction of alkylaromatic substituents to the nitrogen atom of dimeric ionic liquids of the ionene type results in a decrease in the melting point from −98⁰C to −110⁰C (Table 2).

In determining the effectiveness of the influence of the nature of the structural and chemical characteristics of the carriers on the ionic conductivity of polymeric ionic liquids of the ionene type, a detailed analysis of the dependence of the ionic conductivity of dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine on the nature of their spacer at the ionic center was carried out. As follows from the results obtained (Table 2), it is natural that the ionic conductivity of dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine derivatives decreases in the series:

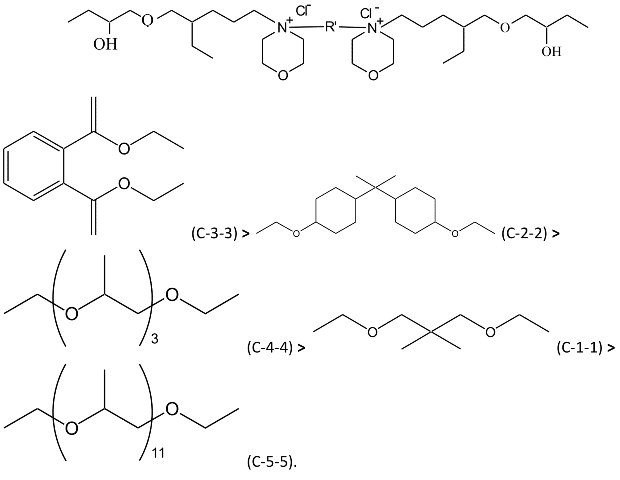
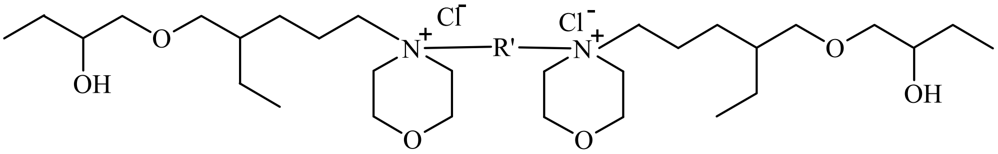
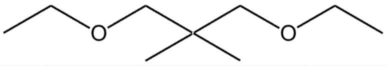
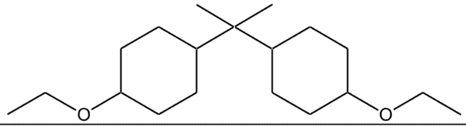
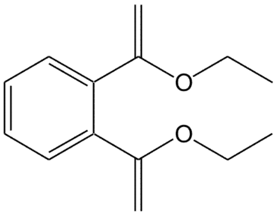
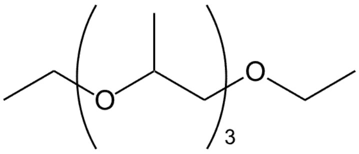
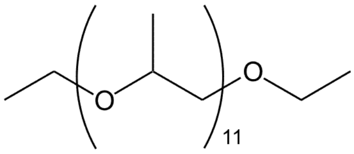


Table 2

Decomposition temperature, melting point, ionic conductivity of dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine of the general formula



Title	R'	Specific ionic conductivity (σ), S·cm^{-1} at 20^{0}C	Decomposition temperature (T_d), ^{0}C	Melting point (T_m), ^{0}C
C-1-1		0.423×10^{-3}	202	–36
C-2-2		0.276×10^{-1}	143	–98
C-3-3		0.362×10^{-1}	123	–110
C-4-4		0.164×10^{-2}	195	–53
C-5-5		0.127×10^{-3}	206	–25

A significant effect on the increase in the ionic conductivity of ionic liquids of the dimeric form is exerted by the transition from dimeric ionic liquids of the ionene type with a radical at the ionic center of an aliphatic structure to dimeric ionic liquids of the ionene type with a radical at the ionic center of an alkylaromatic structure. This occurs due to the reduction of the interaction with protons of methyl or oxyethyl groups of substituents at the nitrogen atom.

Special emphasis should be placed on the fact that the replacement of the group $-O-CHCH_3-CH_2-$ by $-O-CH_2-C(CH_3)_2-CH_2-O-$ leads to a decrease in ionic conductivity. This is probably due to an increase in spatial complications.

The practical feasibility of using radicals near the cation center of a certain nature to ensure a high level of ionic conductivity of ionic liquids of the ionene type while maintaining properties up to very low temperatures is confirmed by the established correlation dependencies between the melting point and ionic conductivity of ionic liquids.

Based on the results of the study (Table 2), it is possible to state the presence of an interesting regularity in a number of dimeric ionic liquids of the ionene type with radicals at the cationic center, which is a residue of the initial tertiary diamines (both for the synthesis of polymeric ionic liquids and for the synthesis of dimeric ionic liquids of the ionene type) of various nature, which is associated with an increase in the ionic conductivity of ionic liquids with a decrease in their melting point. For example, a decrease in the melting point of dimeric ionic liquids of the ionene type C-1-1÷C-1-5 from -28^0C to -110^0C leads to an increase in the specific conductivity from $0.127\cdot10^{-3}$ S·cm^{-1} to $0.362\cdot10^{-1}$ S·cm^{-1}. The generalization of this fact was confirmed by the correlation dependences between the melting point and ionic conductivity of dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine:

C-1-1÷C-5-5 ($\sigma=-0.048\cdot10^{-1}T_m-1.9$, $r=-0.965$).

In this case, the multicomponent nature of the system was taken into account, therefore, the establishment of correlation dependencies between the properties of dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine was carried out by excluding from consideration the influence of the covalent radius of the anion.

The peculiarities of the action of the nature of the radical at the cationic center, which is a residue of the initial dihalides, of dimeric ionic liquids of the ionene type on their melting point and ionic conductivity parameters determine the influence on the change in these characteristics (Table 3) and the dependencies were proven, which is confirmed by the corresponding correlation equations:

C-1-1-1÷C-1-3-3 ($\sigma=-0.038\cdot10^{-1}T_m-3.9$, $r=-0.994$).

The results of determining the temperature dependence of the ionic conductivity of dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine are presented in Figure. The nature of these dependences is the same, therefore the results of the study are given on the example of some compounds. It should be noted that with increasing temperature, the ionic conductivity of dimeric ionic liquids of the ionene type increases. This is due to a decrease in the viscosity of ionic liquids and a decrease in the solvation of ions, which leads to an acceleration of the speed of movement of these ions.

A generalization of the research results can be formulated in the form of the following theses:

– the introduction of allyl and hydroxyethyl groups to the quaternary nitrogen atom, the presence of a benzyl fragment at the cationic center and radicals of alkylaromatic structure will allow to obtain polymeric ionic liquids of the ionene type with a high level of ionic conductivity at a low glass transition temperature;

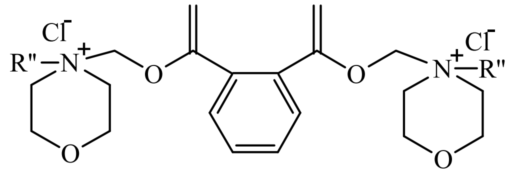
– the ionic conductivity of dimeric ionic liquids of the ionene type increases with decreasing melting point and increasing temperature;

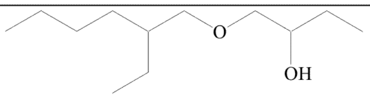
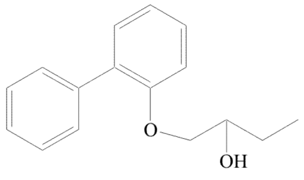
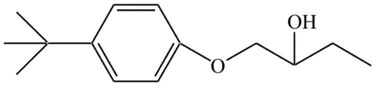
– increasing the stability of dimeric ionic liquids of the Ionene type can be achieved by introducing groups $-O-CHCH_3-CH_2-$ into the radical near the

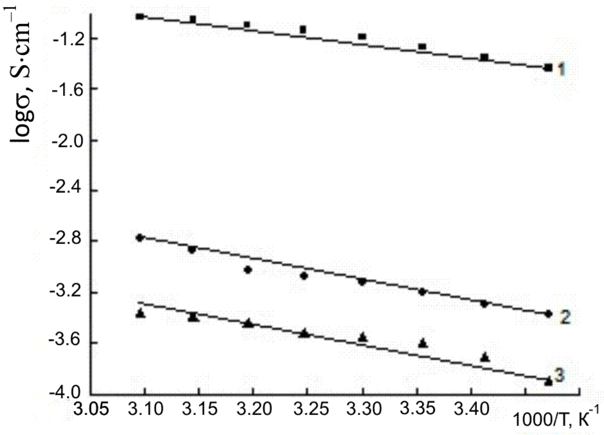
Table 3

Melting point, ionic conductivity of dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine of the

general formula



Title	R''	Specific ionic conductivity (ó), S·cm^{-1} at 20^{0}C	Decomposition temperature (T$_d$), ^{0}C	Melting point (T$_m$), ^{0}C
C-1-1-1		0.362×10^{-1}	123	–110
C-1-2-2		0.541×10^{-1}	115	–116
C-1-3-3		0.857×10^{-1}	107	–123



Temperature (1000/T) dependence of the ionic conductivity (logσ) of dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine: 1 – C-3-3; 2 – C-1-1; 3 – C-5-5

quaternized nitrogen atom.

Such conclusions can be considered expedient from a practical point of view, since they allow a reasonable approach to determining the nature of radicals near the functional center of polymeric ionic liquids of the ionene type and formulate recommendations for the development of new polymeric compounds with high ionic conductivity, which is confirmed by the established similar dependences between temperature, substituents at the quaternary nitrogen atom and the ionic conductivity of polymeric ionic liquids of the ionene type [19].

Conclusions

Summarizing the results of the study in this paper, it is impossible not to note that the synthesized dimeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine derivatives with an ionic conductivity of ~10^{-1}–10^{-3} S·cm^{-1} in the melting temperature range of -123^{0}C÷-25^{0}C have an ionic conductivity that is three to four orders of magnitude higher than the known world analogues based on ionic liquids ($\sigma=10^{-5}-10^{-6}$ S·cm^{-1}; $T_m=-15\div-10^{0}$C) [20]. This discovery makes it possible to supplement a number of existing effective dimeric ionic liquids of the ionene type with compounds with a higher level of ionic conductivity in the range of -123^{0}C to $+206^{0}$C. Due to this, it can be argued that the scientific concepts of predicting the relationship "synthesis–structure–properties" have been further developed, which is the basis for creating a new methodology for the development of polymeric ionic liquids of the ionic type with a high level of properties that retain a liquid aggregate state in a wide temperature range.

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ТЕХНОЛОГІЧНІ ПАРАМЕТРИ ДИМЕРНИХ ІОННИХ РІДИН ІОНЕНОВОГО ТИПУ НА ОСНОВІ ТЕТРАГІДРО-1,4-ОКСАЗИНУ

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Експериментальними дослідженнями підтверджено, що за рахунок введення алільних і гідроксиетильних груп, бензилового фрагменту, радикалів алкілароматичної будови до катіонного центру димерних іонних рідин іоненового типу сприяє отриманню високого рівня іонної провідності 10^{-1} См·см$^{-1}$ при її збереженні до температури -123^{0}C. Встановлено, що синтезовані іонні рідини димерної форми є термостабільними до 107-206^{0}C. Доведено, що наявність груп $-O-CHCH_3-CH_2-$ у радикалі біля кватернізованого атому Нітрогену димерних іонних рідин іоненового типу сприяє підвищенню їх термічної стабільності. Здійсненими дослідженнями впливу природи радикалу біля іонного центру димерних сполук на їх температуру плавлення встановлено, що температура плавлення димерних іонних рідин іоненового типу зростає із збільшенням кількості груп $-O-CHCH_3-CH_2-$ у радикалі іонних сполук. Зокрема встановлено кореляційні залежності між температурою, хімічною будовою димерних іонних рідин іоненового типу на основі похідних тетрагідро-1,4-оксазину та їх іонною провідністю, що підтверджено кореляційними рівняннями і пояснено їх хімічну природу. Це дозволяє стверджувати про відповідність виявленого механізму формування властивостей та практичну привабливість запропонованих рішень.

Ключові слова: димерні іонні рідини, іонна провідність, температура розкладання, температура плавлення, властивості.

TECHNOLOGICAL PARAMETERS OF DIMERIC IONENE-TYPE IONIC LIQUIDS BASED ON TETRAHYDRO-1,4-OXAZINE

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Experimental studies confirmed that introduction of allyl and hydroxyethyl groups, a benzyl fragment, and alkylaromatic radicals into the cationic center of dimeric ionene-type ionic liquids leads to a high ionic conductivity of 10^{-1} S cm^{-1} at -123^{0}C. The synthesized dimeric ionic liquids were found to be thermally stable in the range 107–206^{0}C. The presence of $-O-CH(CH_3)-CH_2-$ groups in the radical adjacent to the quaternized nitrogen atom was shown to enhance thermal stability. Studies of how the nature of the radical near the ionic center affects melting behavior established that the melting point of the dimeric ionene-type ionic liquids increases with an increasing number of $-O-CH(CH_3)-CH_2-$ units in the radical. In particular, correlations between temperature, chemical structure of the tetrahydro-1,4-oxazine-based dimeric ionic liquids, and their ionic conductivity were established; correlation equations are provided and their chemical basis is discussed. These findings support the proposed mechanism of property formation and demonstrate the practical relevance of the developed compounds.

Keywords: dimeric ionic liquids; ionic conductivity; decomposition temperature; melting temperature; properties.

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